

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JAN. 2 and 9, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Mechanical methods of dust collection.** M. A. LISSMAN (Chem. and Met. Eng., 1930, 37, 630—634).—The theory of cyclone dust separators is considered and mathematical expressions are deduced. D. K. MOORE.

**Friction of dry solids in vacuo.** P. E. SHAW and E. W. L. LEAVEY (Phil. Mag., 1930, [vii], 10, 809—822).—Measurements have been made *in vacuo*, by the tilt method, using every possible combination of the following materials: aluminium, silver, copper, nickel, iron, and soda-glass. After repeated heating and cooling in a vacuum, the friction rises to a value which is practically constant between 15° and 350°, except in two or three instances where there is a pronounced positive or negative temperature coefficient. The coefficient of friction,  $\mu$ , is much higher in a vacuum, especially for light loads, than when the materials are treated in the open air, and rises until complete annealing has taken place. The theory of the subject is discussed. M. S. BURR.

**Condensation of vapours.** C. C. MONRAD and W. L. BADGER (Ind. Eng. Chem., 1930, 22, 1103—1112).—The recent increase in liquor velocities in evaporators and condensers has rendered the study of heat transfer through the vapour film of greater importance than it was formerly. The theoretical work of Nusselt (Z. Ver. deut. Ing., 1916) is described. He obtained equations for the heat-transfer coefficient in the cases of (1) vapour condensing on a smooth plane surface making an angle with the horizontal; (2) vapour condensing on the outside of a horizontal tube; (3) as in the first case, but the vapour having appreciable velocity. He also investigated the effect of superheat and the presence of non-condensable gases with less completely satisfactory results. Experimental data obtained by various investigators are compared with Nusselt's equations, and the limitations of the latter are discussed. Whilst good agreement is usually obtained with horizontal tubes, discrepancies occur with vertical tubes owing to the effect of turbulence and the formation of drops; this will usually occur under industrial conditions. Nusselt's calculations on the effect of vapour velocity and superheat, again, are sound when turbulence is absent in the condensate film. C. IRWIN.

**Automatic gas-analysis apparatus dependent on the thermal conductivity of gases, and its industrial use.** P. JARRIER (Chaleur et Ind., 1930, 181; Fuel, 1930, 9, 458—462).—Gas-analysis apparatus based on the thermal conductivity of the gas, *e.g.*, the Siemens-Halske CO<sub>2</sub> recorder, offers certain facilities for the transmission of the readings to a distance or for

automatic regulation. These instruments are applicable to certain mixtures of two or more clean, dry gases, but are less generally applicable to the flue gases from industrial furnaces. The possible sources of error when they are so used, *e.g.*, contamination of the wires, the presence of water vapour or combustible gases, etc., are discussed. To obtain reliable readings constant attention to the filters and connexions is necessary, and the electric current and water current must be exactly regulated. A. B. MANNING.

**"Dry-ice."** KILLEFFER.—See VII. **Firing of boiler furnaces.** FREISE.—See VIII. **Measurement of liquid consistency.** FAWKES. **Photo-electric process control.** STYER and VEDDER; McMASTER.—See XI. **Universal thermostat.** FINK.—See XVIII. **Protection against toxics.** TURNER.—See XXIII.

### PATENTS.

**Heat-exchanging apparatus for fluids.** C. A. HUBERT (B.P. 337,236, 22.11.29).—The apparatus comprises a number of ribbed tubes, so stacked that the ribs come in exact vertical rows in order that cleaning jets may be effective when required.

B. M. VENABLES.

**Tubular heat exchangers.** J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 336,755, 21.9.29).—An exchanger comprising a number of tubes arranged in diagonal lines is provided with metallic filling between the tubes in one diagonal direction, so that the outer fluid flows in parallel sinuous paths and makes contact with a larger surface of metal. B. M. VENABLES.

**Reduction of temperature [of air] by dehydration.** W. L. FLEISHER, Assr. to COOLING & AIR CONDITIONING CORP. (U.S.P. 1,749,763, 11.3.30. Appl., 8.8.28).—The apparatus comprises an inlet mixing chamber, a silica gel dehydrator, a cooling chamber, a spray chamber, an outlet mixing chamber, and a fan which delivers the air to the enclosure to be ventilated. The entering air is dehydrated to a very low dew point by the gel, and the latent heat of absorption is removed by coils cooled by an abundance of water, so that in the cooling chamber the air attains a moderately low dry-bulb and very low wet-bulb temperature. In the spray chamber a limited quantity of water is used over and over again, except for a little make-up, so that the temperature of the water and the actual or dry-bulb temperature of the air both become reduced to the wet-bulb temperature. In cases where it is desirable to recirculate air from the enclosure, part will be returned direct to the outlet mixer and the other part with some fresh air will enter the inlet mixer for treatment.

\* The remainder of this set of Abstracts will appear in next week's issue.

The proportion treated will be under the control of a dry-bulb thermostat placed in the return conduit; another thermostat with a wet bulb regulates the cooling water in the coil. B. M. VENABLES.

**Thermostats.** R. MACLAREN (B.P. 337,093, 10.8.29. Addn. to B.P. 307,135; B., 1929, 496).—A bimetallic thermostat as described in the prior patent is provided with a modified switch movement incorporating a magnetic blow-out. B. M. VENABLES.

**Drying of material.** L. N. LLOYD, and PHILLIPS ENG. CO., LTD. (B.P. 336,602, 9.5. and 3.8.29).—The material is spread in a thin layer upon the uppermost storey of a series of supports each comprising a number of tray-like devices adapted to discharge, at intervals, the whole of the load on one storey on to the next below. The trays are heated by internal or closely adjacent means, and the feed and discharge devices operate through air-locks so that drying may be effected in a vacuum. B. M. VENABLES.

**Rotary drying apparatus.** J. B. VERNAY (B.P. 336,874, 19.12.29. Fr., 21.12.28).—The material is passed in a zig-zag manner through a number of co-axial shells, the innermost of which is stationary and is provided with a heating jacket, whilst the others are rotated. A certain amount of grinding is effected during the drying, and air-locks are provided so that a vacuum may be applied. B. M. VENABLES.

**Spray dryer.** W. F. RICHARDS (B.P. 337,080, 2.8.29).—The spraying chamber is surrounded by a number of compartments containing bag filters; the inlet sprays of liquid are situated in the upper circumference, the inlet of drying gases is upwards from the bottom centre, and the passages to the filters are along the floor under the circumference of the inner chamber, which has a roof adjustable in height to vary the drying space. B. M. VENABLES.

**Crushing and grinding of solids.** M. P. APPLEBEY, F. POSTLETHWAITE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,730, 29.8.29).—Crushed material, *e.g.*, coal, is removed from a jaw crusher by means of upward currents of air or other gas as fast as it is ground. B. M. VENABLES.

**Reducing friable materials.** G. C. E. KEET (B.P. 336,687, 24.7.29).—The material is passed between rolls which are convex and concave or V-shaped, and the distance between their axes is continuously varied by means of a toggle mechanism. B. M. VENABLES.

**Disintegrating machines particularly applicable to rubber.** INTERNAT. PULVERISING & GRINDING MACHINES (PARENT) CORP., LTD., and P. LEGRAND (B.P. 336,828, 7.11.29).—Forms of disc grinders suitable for rubber are described, the discs of which are provided with rasp teeth. B. M. VENABLES.

**Pulverising mills.** BABCOCK & WILCOX, LTD. FROM FULLER LEHIGH CO. (B.P. 336,787, 14.10.29).—The machine is of the ball-mill type with hollow trunnions, the feed being through one trunnion and the discharge air-borne through the other trunnion; adjacent to the latter is a classifier for separation of oversize, which is returned to the mill through the same trunnion by means of a screw or other type of conveyor. B. M. VENABLES.

**Grinding, crushing, and mixing mills.** J. WASS (B.P. 336,212, 6.7.29).—An edge-runner mill, preferably with a conical bottom and correspondingly shaped roller, is provided with means by which the roller can be lifted out of action so that mixing can continue by means of stirring blades alone. B. M. VENABLES.

**Centrifugal machine for clarifying and purifying liquids.** RAMESOHL & SCHMIDT A.-G. (B.P. 336,865, 10.12.29. Ger., 10.12.28).—A machine suitable for separating two liquids (also solids), when the heavier liquid is desired of maximum purity, is described. There are three concentric separating zones, and the heavier liquid finally leaves inwards through a filter. Application to cleaners' solvent liquid is indicated. B. M. VENABLES.

**Centrifugal machine.** C. and F. GREAVES (B.P. 336,743, 12.9.29).—A method of feeding the material to, and supporting the inclined axes of, the planetary baskets of a centrifugal machine is described. B. M. VENABLES.

**Sterilisation of liquids by heating.** N. J. NIELSEN (B.P. 337,027, 27.6.29).—The liquid to be treated, *e.g.*, milk, is heated while under pressure by heat exchange with another liquid, *e.g.*, water, which is also under a pressure sufficient to prevent the formation of steam or vapour. The water flows in a continuous circuit: from a reheater to an exchanger, where it flows countercurrent to the arriving milk, then through another exchanger countercurrent to the departing milk (which is thereby cooled), back to the reheater. B. M. VENABLES.

**Apparatus for the hydrogenation of liquids, solids in suspension, or gases.** TECHNICAL RES. WORKS, LTD., and E. J. LUSH (B.P. 336,569, 11.7.29).—Forms of apparatus are described in which the catalyst, consisting of nickel wire or turnings activated by anodic oxidation, is supported in masses not materially exceeding 6 in. thick. The cages and container may be formed of copper or a copper-nickel alloy. B. M. VENABLES.

**Dust filters.** W. M. ODDIE (B.P. 337,304, 30.1.30).—Filter bags, supported at the top by individual chains, have inlets at the bottom in the form of inverted truncated cones within which are heavy correspondingly shaped plugs supported by a continuation of the chains. When a bag is in the operative stretched position the plug valve will be open, but when the suspending chain is slackened the inlet will be closed and the bag simultaneously given a jolt. On restretching the bag the dislodged dust will slide out through the inlet cone. B. M. VENABLES.

**Deposition of dust or the like from an atmosphere in which it is suspended.** WOODALL-DUCKHAM (1920), LTD., and A. MCD. DUCKHAM (B.P. 336,201, 6.6.29).—Air containing dust, *e.g.*, coal from a pneumatic separator, is repeatedly circulated round a depositing vessel, and sprays of steam or other suitable liquid, gas, or vapour are admitted countercurrent to the air so that the density of the solid particles is changed relatively to the air, both by coagulation and by admission of a gas (steam) of lower density than the air. B. M. VENABLES.

**Separation of dust from air or gases.** G. RAW (B.P. 336,221, 4.7.29).—Dust, *e.g.*, coal dust from a



pneumatic separator, is coagulated by admission of a mist of liquid to the air while in a substantially stagnant state; the mist should be too fine to settle except in combination with the dust, and the quantity of liquid should be so small that the moisture is practically imperceptible. B. M. VENABLES.

**Extraction of dust and grit from air, flue gas, or other medium.** PNEUMATIC CONVEYANCE & EXTRACTION (1929), LTD., and W. A. SMITH (B.P. 336,620, 17.6.29. Addn. to B.P. 256,063).—The air to be cleaned is passed through an involute passage, entering at the point of largest radius, which has a V-shaped outer wall and a slit along the apex of the V for the outlet of dust concentrated in a small quantity of air to a surrounding casing acting as a settling chamber. A pair of dust extractors may be attached to the twin inlets of an extraction fan having by-passes to transmit the reduction of pressure to the settling chambers also. B. M. VENABLES.

**Liquefaction of air and other gases.** M. HAZARD-FLAMAND (B.P. 336,798, 17.10.29. Fr., 18.10.28).—The air after compression to 200 atm. is passed through one side, say the inner, of three heat exchangers in series, and a part of it also through a fourth, after which the same portion is expanded through a constriction without doing work, and thus is partially liquefied. The unliquefied gas is passed backwards through the outer sides of the fourth and second exchangers, together with the other portion of gas that was tapped off between the third and fourth exchangers, which portion has been expanded while doing external work. The first exchanger is cooled by water, and the third by the vaporisation of liquefied ammonia. A purifier may be placed between the first and second exchangers. B. M. VENABLES.

**Gas-liquefying plant.** A.-G. BROWN, BOVERI & Co. (B.P. 337,325, 22.2.30. Ger., 18.3.29).—A centrifugal compressor has its bearings lubricated by the liquefied gas. B. M. VENABLES.

**High-vacuum vapour pumps.** W. GAEDE (B.P. 337,214, 11.11.29. Ger., 13.11.28).—A pump of the diffusion type is described. The diffusion passage is cooled externally by a jacket and internally by a coil, and any mercury condensed on the latter is prevented from dropping into the hot mercury jets by a hood. B. M. VENABLES.

**Manufacture of vessels and other structures that are proof against chemical and atmospheric influences.** P. PICK (B.P. 316,134, 4.7.29. Ger., 23.7.28).—Constructional elements are formed from perforated metallic plates covered with one or more layers of plastic material which is unvulcanised (but capable of being afterwards vulcanised), the sandwich produced being hydraulically pressed while subjected to a vacuum. The metal plate may have stiffening ribs pressed out of the plate, but they should not extend to the edges, and the plastic material may be in layers of different hardness indissolubly welded and having the air removed by the pressure and vacuum. B. M. VENABLES.

**Apparatus for carrying out chemical reactions.** BRIT. CELANESE, LTD. (B.P. 310,415, 15.4.29. U.S., 25.4.28).—A mixing vessel with fixed and moving vertical blades is described. B. M. VENABLES.

**Photometers.** HOLOPHANE, LTD., and H. S. ALLPRESS (B.P. 336,897, 20.1.30).—The instrument is suitable for expressing the illumination inside a room as a proportion of that of the sky. B. M. VENABLES.

**Measuring the temperatures of incandescent bodies.** KAISER-WILHELM INST. F. EISENFORSCHUNG (B.P. 336,296, 12.7.29. Ger., 31.10.28).—An optical pyrometer is provided with a parallel-faced filter which cuts off all light except red and green (or another pair of selected colours), and with a wedge-filter which permits one of those colours, e.g., red, to pass through freely, but cuts off the other, green, in proportion to the thickness of the wedge. To measure the temperature, the wedge is adjusted so that a whitish mixed colour is transmitted; then should the observed body become hotter the light will become green, or if cooler red. This method is stated to be less affected by the nature of the emitting surface than are those which depend on the total radiation. B. M. VENABLES.

**Measurement of humidity.** CAMBRIDGE INSTRUMENT Co., LTD., J. L. ORCHARD, and H. T. GLOVER (B.P. 336,855, 2.12.29).—A pair of equal coils of wire forming resistance thermometers are placed close together in a gas stream, one being kept dry and wholly exposed to the gas, the other being wholly covered with fabric which is kept wet. They form two arms of a Wheatstone bridge, and electrical devices are combined with the other two arms of the bridge to compensate for variations from standard of the dry-bulb temperature, so that the galvanometer may be calibrated to read directly in percentage of relative humidity. B. M. VENABLES.

**Apparatus for heat treatment of liquids.** W. WINSHIP (B.P. 337,762, 3.5.29).—See U.S.P. 1,712,372; B., 1929, 579.

**Testing [the physical properties of] materials.** R. ESNAULT-PELTERIE (U.S.P. 1,781,002, 11.11.30. Appl., 4.4.27. U.K., 4.2.27).—See B.P. 289,960; B., 1928, 553.

**Conservation of heat in a steam power plant.** STEAM HEAT CONSERVATION Co., LTD., H. P. H. ANDERSON, and J. McCULLUM (B.P. 336,599, 13.4.29).

**Absorption refrigerating apparatus.** G. BEAUMONT (B.P. 337,912, 30.9.29).

**Refrigerating apparatus of the absorption type.** A. SILBERNAGEL (B.P. 337,964, 1.11.29. Switz., 12.11.28).

**Absorption machines for refrigerating or heating.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 317,052, 6.8.29. Ger., 9.8.28).

**Safety devices for hydro-extractors.** D. K. and A. F. TULLIS (B.P. 337,670, 20.12.29).

**Apparatus for automatically discharging the centrifuge drum of a continuously operating high-speed centrifuge.** K. Egg (B.P. 337,970, 5.11.29).

**[Manufacture of segmental] grinding wheels.** CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 336,631, 17.7.29).

Appliances for viewing fluorescent and phosphorescent substances [for advertising purposes]. L. A. LEVY (B.P. 336,317, 19.7. and 4.12.29).

Manufacture of sheets, films, etc. (B.P. 315,840 and 318,250).—See V. Tunnel kilns (B.P. 336,171).—See VIII. Zinc alloys for bearings (B.P. 336,881).—See X.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Transformation of fatty acids during geological periods. IV. G. STADNIKOV and Z. VOSSCHINSKAJA (Brennstoff-Chem., 1930, 11, 414—416. Cf. Stadnikov and Weizmann, B., 1929, 1002).—"Balkashite," a sapropelite from Lake Balkash (cf. Zelinski, B., 1926, 226), has been formed by the oxidation, polymerisation, and dehydration of the fats and fatty acids of a green alga, *Botryococcus Braunii*, K., which grows in enormous quantities in the lake. Chemical and microscopical examination shows it to be closely related to the Siberian and Moscow boghead coals, and to resemble the saponifiable fraction of coorongite. A. B. MANNING.

Significance of spores in the correlation of coal seams. I. The Parkgate seam—South Yorkshire area. L. SLATER, M. M. EVANS, and G. E. EDDY (Dept. Sci. Ind. Res., Fuel Res., Phys. and Chem. Survey of Nat. Coal Resources No. 17, 1930, 28 pp.).—The object of the investigation was to determine whether a single spore type, by its presence in any part of a seam, or by its concentration in certain parts, or the distribution of the spores as a whole, is characteristic and would therefore serve as a basis for the correct identification and correlation of different coal seams. The major portion of the Parkgate seam is composed of three bands, viz., "tops," "middle coal" or "hards," and "bottoms"; the tops and bottoms are both bright in appearance, consisting mainly of clarain, whilst the middle coal consists essentially of durain. Three pillar samples (i.e., seam sections) of Parkgate coal were taken from different districts, and a series of thin sections, cut at right angles to the bedding plans, were prepared; the whole area of each thin section was examined carefully and the spore exines were classified. It was found that (a) spore remains were more numerous in the hards than in the tops or bottoms; (b) in certain thin sections, principally in the hards, they were more numerous than in others; (c) most of the megaspore types recorded were present in all three pillar samples; (d) only certain types of megaspores showed a similarity of distribution in each pillar sample; (e) certain of the remaining types were concentrated in a similar manner in two of the pillar samples, but in the third showed considerable variation either in a part or throughout the whole of the sample. It is suggested that despite such irregularity of distribution, the presence of these spores may be used for correlation purposes.

C. B. MARSON.

Some aspects of the problem of cleaning Witbank coal. I. J. BLOM and T. J. W. JORDEN (Fuel, 1930, 9, 464—476).—Five samples of coal from the Witbank deposits were crushed and screened, and each of the sized fractions (0.4 mm., 0.4—1.0 mm., 1—2 mm., 2—3 mm., 3 mm.) was submitted to a float-and-sink

analysis. No sharp bends were observed in the washability curves, which were examined from the viewpoint of dividing the coals into three fractions: (1) coking coal of normal ash content, e.g., 7%; (2) steam coal of high ash content, e.g., 13—14%; and (3) waste. The ash content of the raw coals varied from 9.7 to 11.8%. An examination of the float-and-sink data by Bird's method (2nd Internat. Conf. on Bit. Coal, 1928, 2, 82) indicates that the separation at 7% ash content would be very difficult in an ordinary washing plant, but could probably be effected by means of the Lessing Rational Coal-Cleaning Plant (*ibid.*, 1928, 2), after removal of the dust which would be added to the steam coal. The relation between the ash content and the sp. gr. of the fractions of each sample of coal is roughly linear. Caution is necessary, however, in applying Lategan's method of determining the ash content (B., 1927, 833) to coal-cleaning operations; it may become accurate enough for practical purposes if a "proportionality curve" is determined for each separate colliery and is used only between the limits within which it has been shown to be valid. The variation in the sulphur and nitrogen contents of one of the coals (T. & D.B.) has been studied. The sulphur content at first falls rapidly with decreasing sp. gr. of the separated fractions up to a point corresponding with 9.5% of ash, due probably to the removal of pyrites, and thereafter remains more or less constant at 0.7—0.8%. The nitrogen content increases in steps as the ash content falls, corresponding probably with the successive removal of the fusain and durain as the washing proceeds. An examination of the ash composition curves shows a change in the nature of the inorganic constituents with increasing ash content of the floated material at the point corresponding with 8% of ash for Oogies coal and 9—10% for Douglas coal. It is concluded that these values represent the "inherent ash" of the coals (cf. Mott and Wheeler, B., 1927, 802). A. B. MANNING.

Oxidation of mineral coals in an atmosphere of oxygen. Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 199—207).—Curves showing the loss in weight of coals as a function of the time of exposure to oxygen are characteristic for the particular coals. The reduction of cokability due to treatment with oxygen is a qualitative measure of the stability of the coal. CHEMICAL ABSTRACTS.

Determination of cokability of U.S.S.R. coals by Meurice's method. Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 177—198).—Concordant results were obtained with Russian coals. CHEMICAL ABSTRACTS.

Coking of Kuznetz coals (Kemerovo, Siberia). P. G. RUBIN and Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 33—79).—Unlike Donetz and most European coals, Kuznetz coals exhibit a cokability which increases with an increase in the amount of volatile matter. CHEMICAL ABSTRACTS.

Cokability and stability of Donetz coal. P. G. RUBIN and Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 81—124).—Good coking coals gave a Meurice value of at least 120, medium 70—100, and poor less than 70. The stability ratios (of Meurice

value of original coal to that after exposure to oxygen for 6–7 days) were, respectively, 100, 80–90, and 50–60%.

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**Dehydrating action of coal ash.** M. TANAKA (Rep. Lab. S. Manchuria Rly., 1929, 36–37).—The composition of the ash of coal and oil shale has been found to resemble that of acid clay. When applied to the synthesis of anthraquinone derivatives the dehydrating powers of Fushun coal ash and Japanese acid earth were very similar, being fairly active at 260°, although slightly inferior to that of anhydrous aluminium chloride.

H. J. DOWDEN.

**Determination of fusain.** F. HEATHCOAT (Fuel, 1930, 9, 452–458).—Fusain is determined by a method depending on its inertness towards oxidising agents. The dry and powdered coal sample is refluxed with Schultze's solution (3 g. of potassium chlorate in 100 c.c. of 2*N*–4*N*-nitric acid), filtered through a fritted glass crucible, washed, transferred to a beaker, boiled for 1 hr. with *N*/6-sodium hydroxide, filtered, washed, dried, and weighed. The ulmin material and the organised plant remains are completely oxidised and removed by this treatment. The result is low owing to the partial conversion of the fusain into alkali-soluble products, but from an examination of a series of coal dusts and slurries it appears that, in general, the errors arising from the oxidation of fusain are not serious. The method possesses decided advantages over that based on the microscopical examination of the particles. The examination of the sieved fractions of a hand-picked "fusain" shows that the estimated fusain content is higher in the finer sizes than in the coarser.

A. B. MANNING.

**Determination of total acid in peat.** E. SAUER and H. J. STEINMETZ (Kolloid-Z., 1930, 53, 218–224).—An excess of calcium bicarbonate solution is added to the peat and the excess is titrated after reaction is complete. The method gives more reliable results than are obtained by adding calcium carbonate and determining the liberated carbon dioxide. The accuracy of the method increases with the degree of dispersion of the peat, and the material is best dried and ground in a porcelain ball-mill. The presence of neutral salts does not affect the results. Several kinds of peat examined showed a remarkable agreement in total acidity.

E. S. HEDGES.

**Comparison of recent methods of determining the agglutinating power of bituminous coals.** G. AGDE and A. WINTER (Brennstoff-Chem., 1930, 11, 394–396).—The agglutinating values of mixtures of a caking coal and anthracite have been determined by the following methods: Meurice-Kattwinkel (B., 1926, 207, 905), Meurice-Gabinsky (Mitt. Lehrst. Eisenhütten- u. Brennstoffkunde Dnepropetrovsk, 1929, 1, 177), and Marshall-Bird (cf. Johnson and Yancey, B., 1930, 1052). Each method gave results for the different mixtures which were proportional to those of the other methods. This proportionality failed, however, when coals of different type were compared. The methods of Kattwinkel and of Gabinsky may fail with some coals, due to segregation of the sand during the heating. The Marshall-Bird method is the most reliable, giving less

variation from the mean value with repeated determinations.

A. B. MANNING.

**Plastic range of coking coals.** W. DAVIDSON (Fuel, 1930, 9, 489–492).—The plastic ranges of 12 coking coals have been determined by a modification of Foxwell's method (B., 1924, 4 *seq.*). Whilst the beginning of the plastic range did not vary with the rate of heating (from 1–5°/min.), the extent of the range increased with increased rate of heating. The volatile matter evolved before the beginning of the plastic range and that evolved during the plastic range were determined separately. There is a distinct relationship between the nature of the coke obtained and the percentage of volatile matter evolved subsequent to the plastic range, and it is considered probable that if the volatile matter retained by a coal at the end of the plastic range could be diminished, *e.g.*, by adding a material of low volatile matter content, or a coal having a higher temperature and lower volatile content at the end of the plastic range, a coke having less fissures would be obtained. This supposition was confirmed by blending a Scottish coal with finely-ground high-temperature coke, whereby a coke of increased size and resistance to shatter was obtained.

A. B. MANNING.

**Production of strong coherent coke by the low-temperature carbonisation of brown coal mixed with tar.** C. STAEMMLER (Brennstoff-Chem., 1930, 11, 413–414).—Mixtures of brown coal with a brown-coal generator tar in the ratio 1:1 were carbonised in a small aluminium retort, but failed to give a coherent coke. The character of the coke was greatly improved by preoxidising the tar with air, preferably in the presence of a catalyst, *e.g.*, manganese or lead dioxide, but even under these conditions the process was uneconomical.

A. B. MANNING.

**Possibilities of the utilisation of coke fines and its value in Upper Silesia.** P. DAMM and F. WESE-MANN (Stahl u. Eisen, 1930, 50, 1495–1500).—Under the economic conditions in Upper Silesia, the value of coke fines for use in gas producers or as an addition to coking coals is shown to be small, but the fines are of considerable value for firing boilers, for use as a reducing agent in zinc smelters, and, above all, for briquetting. The economics of the use of coke dust for these purposes is discussed in considerable detail.

A. R. POWELL.

**Utilisation of coke breeze by the Trefois producer.** A. L. HOLTON and H. C. APPLEBEE (Gas J., 1930, 192, 151–158).—The producer is fitted with rotary grate and water jackets, and has a daily capacity of 10–12 tons of fuel, making about 1½ million cub. ft. of gas at 115–130 B.Th.U./cub. ft. In its original form clinker troubles were severe, and necessitated allowance for a lower repose angle of the ashes and better arrangements for breaking clinker. All types of coke breeze, except that entirely passing a ½-in. screen, have been used; no difficulties were experienced with fuels having high moisture contents, but dust and tar deposits were sometimes found. Full details are given for a number of tests, in England and abroad, showing a gas-making efficiency as high as 88.5%. Control of the machine is easy and its flexibility considerable. The application of the producer to the heating of horizontal retorts has been

achieved, and the advantages arising from its use are discussed.

R. H. GRIFFITH.

**Friability of mid-temperature cokes.** S. W. PARR and D. R. MITCHELL (Ind. Eng. Chem., 1930, 22, 1134—1135).—One-ton charges of Illinois, Iowa, and West Virginia coals were prepared in an experimental plant, using a standard 12-in. oven operated according to the Parr process, the special characteristics being quick carbonisation and use of the mid-temperature range; all the coals were preheated at about 300° and then charged hot into the coke oven, the coking temperatures ranging from 750° to 900°. Shatter tests on the resulting cokes showed that the shatter index (2 in.) varied from 57 to 79, that shortening of the preheating period slightly increased the strength of the coke, and that an increase in coking time effected an improvement in the quality of the coke. It is concluded that mid-temperature cokes are somewhat tougher than cokes made by high-temperature processes; cokes made at 750–900° contain 4–5% of volatile matter and are very desirable as domestic fuel, being smokeless, of low ignition temperature, and of high physical strength.

C. B. MARSON.

**Reactivity of metallurgical coke.** M. DUFRANKE (Rev. Mét., 1930, 27, 509–511).—Reactivity of fuel is defined by the extent to which the reaction  $C + CO_2 = 2CO$  proceeds in the forward (endothermic) direction. High reactivity of blast-furnace coke is a cause of irregularities in working the furnace. Samples of coke from three sources have been studied at 800°, 900°, 1000°, and 1100°. The reactivity increases rapidly with rise in temperature. Treatment of a very reactive coke in a slurry of lime or cement is found to reduce the reactivity at all temperatures to a value below that of the other (untreated) materials; the compressive strength is raised in consequence.

E. H. BUCKNALL.

**Gas-, coke-, and by-product-making properties of American coals and their determination.** A. C. FIELDNER, J. D. DAVIS, and D. A. REYNOLDS (Ind. Eng. Chem., 1930, 22, 1113–1123).—An experimental carbonising apparatus, taking a charge of 75–100 lb. of coal, is described and the results obtained with two gas coals which had previously been tested under full-scale conditions are discussed. Tests were carried out at temperatures between 500° and 1100° and the yields and quality of coke, gas, tar, light oil, and liquor determined. Close agreement between the experimental and full-scale methods was obtained in the case of gas, coke, and tar yields, but the experimental method gave lower ammonium sulphate yields, which is attributed to the catalytic effect of the iron retort in decomposing ammonia. The experimental coke had approximately the same volatile matter, cell space, apparent  $d$ , and resistance to abrasion as full-scale coke made from the same coal at the same temperature of carbonisation, and was characterised by a lower shatter index; reasons are advanced for this difference. It is believed that shatter and tumbler tests will give relative values for comparing different coals and coal blends in the experimental apparatus. The first coal examined gave the strongest coke, as judged by shatter and tumbler tests, at carbonising temperatures of 800–900°; the second coal

gave the strongest coke at 600°, and it is suggested that mid- and low-temperature processes, respectively, are therefore suitable for these two coals. Using this method of carbonisation, the carbonising properties of a coal may be correlated with its constitution etc., and results of the following tests on the two coals examined are given: softening and decomposition temperatures, agglutinating value, plastic range, low-temperature assay, oily and solid bitumen contents.

C. B. MARSON.

**Comparison of acetylene black with gas black and lampblack.** T. R. DAWSON and, in part, N. H. HARTSHORNE (Trans. Inst. Rubber Ind., 1930, 5, 48–70).—Analyses of samples of the three blacks are given and comparisons made as to their colour, pigmenting power, bulkiness, sp. gr., and particle size. The acetylene black is chemically the purest and the lampblack the least pure. All three types of black retard the vulcanisation of a rubber-sulphur mixture. In tensile strength and reinforcing power, although possibly not in permanent set and resilience, gas and acetylene blacks approach one another more closely than they do lampblack, this resemblance extending even to the abnormal discontinuous course of vulcanisation observable with mixtures containing either of the former blacks, together with hexamethylenetetramine. The degree of variability in acetylene black is of the same order as the difference between acetylene black and gas black, but is markedly smaller than the difference between acetylene black and lampblack.

D. F. TWISS.

**Composition of carbonised molasses waste.** W. KILP (Z. Spiritusind., 1930, 53, 305).—Carbonised molasses waste consists essentially of potassium carbonate, chloride, and sulphate, sodium carbonate, and insoluble matter, with a small proportion of moisture. The composition varies according to the stage to which carbonisation has been taken, and black-coloured char may contain over 60% of insoluble matter and approx. 25% of soluble alkali salts, whereas one incinerated more thoroughly may yield over 80% of soluble alkali salts of which 90% consists of potassium salts. The char from beet molasses has a higher content of potassium salts than that from sugar cane, but the latter contains a higher percentage of insoluble matter, owing to its high content of phosphate, silicate, and lime.

C. RANKEN.

**Avoidance of naphthalene troubles [in coal gas].** F. SCHUSTER (Gas- u. Wasserfach, 1930, 73, 1009–1015).—The development of the use of tetralin for removal of naphthalene from coal gas is reviewed, and a system for introduction of tetralin vapour into the gas is described. This is achieved either by supply from a boiler or by atomisation of the liquid. In both cases sufficient tetralin is carried in the gas to prevent deposition of solid; liquid condensate is removed at intervals. The process costs 1–2 pf. for 100 m.<sup>3</sup> of gas, and completely eliminates blockage of services.

R. H. GRIFFITH.

**Formation of copper carbide in acetylene cylinders.** G. F. KOMOVSKI (J. Appl. Chem., Russia, 1930, 3, 401–411).—Pure, dry acetylene does not combine with copper or its alloys. Appreciable quantities of cuprous acetylide are formed only on prolonged contact of

compressed technical acetylene with pure copper; it decomposes in air at 120—123°, in acetylene at 130—150°, or when heated in air at 50° and then introduced into acetylene. In acetylene at 4—5 atm. decomposition occurs at 200—251°. CHEMICAL ABSTRACTS.

**Causes of explosion [in gas holders].** H. WOLF (Chem.-Ztg., 1930, 54, 796).—Deposits of iron sulphide produced by the action of the sulphur compounds in the gas on the metal of the gas holders may ignite spontaneously on contact with air, and constitute a source of danger if the holder contains an explosive mixture. Thin aluminium foil or aluminium bronze powder in contact with air or oxygen may be ignited by the action of a bright light.

A. B. MANNING.

**Problems relating to coal tar.** W. J. CHADDER and H. M. SPIERS (Gas J., 1930, 192, 161—166).—Tars produced in a wide range of coal-carbonising systems have very different properties for road-making purposes. By blending suitable light and heavy fractions from various tars, it is possible to prepare mixtures which conform to the specification, and thus to utilise material which would otherwise be rejected. Calculations can be made of the proper proportions of tars or tar fractions which are required to give a product of stated consistency, and nomograms are shown which make rapid determinations possible.

R. H. GRIFFITH.

**Free carbon of bituminous coal tar.** J. MARCUSON (Chem.-Ztg., 1930, 54, 795—796).—The benzene-insoluble constituents of vertical- and horizontal-retort tars, amounting to 7% and 24%, respectively, consisted of oxy-acids 8.6%, 0.5%; pyridine-soluble resins 73%, 16.3%; pyridine-insoluble resins 18.4%, 32%; and partly-coked material 0%, 51.2%. No actual free carbon was present. The resins could be readily nitrated or sulphonated. Neither they nor the oxy-acids can be regarded as deleterious constituents of the tars, and it appears preferable therefore to consider the "free carbon" as including only the partly-coked material. This may be determined as follows: the tar (5 g.) is treated with 15 c.c. of fuming sulphuric acid (20% SO<sub>3</sub>), and the mixture, with thorough stirring from time to time, is set aside for 2 hrs. It is then diluted with 100—150 c.c. of water, warmed for 15 min. on the water-bath, and the precipitate separated by centrifuging.

A. B. MANNING.

**Resin formation in benzols.** III. Continuation of tests on production of stabilised benzols on the technical scale—storage and road trials. W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 476—481; cf. B., 1930, 1137).—The general running of the engine in road trials on a coke-oven distillate stabilised by the addition of 0.03% of tricresol was as good as, if not better than, with National benzol mixture. Even after six months' storage this distillate gave no weighable amounts of deposit in the induction system and only a trace of soft carbon on the inlet valves. A similar distillate, which, however, contained some free sulphur, gave considerable deposits in the induction system. There is evidence that the lack of agreement between the results of the oxidation and storage tests with some benzols is due to the presence of free sulphur, which

forms non-volatile material from other constituents of the fuel during refluxing. With such fuels the measurement of the rate of oxygen absorption, instead of weight of non-volatile gums formed, might be a better guide to its behaviour on storage. A further cause of discrepancy between the results of the oxidation and storage tests is that in the former the residue corresponds with the non-volatile products which would be formed after a definite period of storage, e.g., 6 months, but gives no indication of the behaviour of the oil for intermediate periods. Tests of the benzols in a variable-compression engine indicate that the anti-knock value of the unsaturated hydrocarbons in the stabilised benzols does not differ appreciably from that of the aromatics. Fleet trials carried out by the Gas Light & Coke Co. on a stabilised benzol-petrol mixture gave satisfactory results.

A. B. MANNING.

**Phenols of petroleum.** E. HOLZMANN and S. VON PILAT (Brennstoff-Chem., 1930, 11, 409—413).—The phenols isolated from the crude naphthenic acids obtained from the distillates of two Polish petroleums (Borislav and Bitkow) have been examined; the phenol content of the crude acids amounted to 30% and 3.1%, respectively. The absence of phenol itself from the Borislav naphthenic acids has been established; all three cresols, however, together with *m*-5- and *m*-4-xenols and  $\beta$ -naphthol were isolated therefrom, and the presence of *o*-4-xenol and trimethylphenols was shown to be probable. No phenols were found having long side-chains or containing methoxy-groups. The phenols, but not the naphthenic acids, could be detected in the original crude petroleum (cf. von Kozicki and von Pilat, B., 1916, 526).

A. B. MANNING.

**Oil emulsions.** A. C. BROWNE (Cal. Dep. Agric., Mon. Bull., 1930, 19, 389—405).—Analytical data of numerous oil types are recorded. No correlation is possible between viscosity, sulphonation tests, distillation ratios, or rates of evaporation.

A. G. POLLARD.

**Breaking crude oil emulsions.** S. VUISHETRAVSKI (Azerbaid. Neft. Choz., 1930, No. 6, 83—98).—Water should be added at the moment when the emulsion leaves the well; demulsifying agents may be added at the same time. Factors affecting the formation of the emulsions were also investigated.

CHEMICAL ABSTRACTS.

**Motor spirit containing alcohol.** III—VI. W. SWIENTOSLAWSKI (Przemysl Chem., 1930, 14, 385—391, 409—412, 433—436, 457—461; cf. B., 1930, 976).—III. [With J. PFANHAUSER.] The fraction of azeotropic mixtures of gasoline, alcohol, and benzene, used as motor fuel, b.p. below 78.3°, is greater the higher is the benzene content; thus 92% of a mixture containing 30 vol.-% of benzene distils over below 78.3°, the b.p. of the residue then rising sharply to 140—180°. In general, this sharp rise in b.p. is indicative of the phenomenon of azeotropism, and corresponds with the exhaustion of the azeotropic mixture.

IV. [With J. PFANHAUSER and B. KARPIŃSKI.] The vapour pressure-temperature curves of a number of motor fuels containing alcohol show that at temperatures above 25° the vapour pressures of these fuels and of ordinary motor spirit are approximately equal, whilst

below 25° the mixtures examined are less volatile, showing that they are not well adapted to use in a cold motor.

V. [With H. STARCZEWSKA.] The b.p., condensation points, and other tonometric factors of a number of motor-fuel mixtures, containing (apart from petrol) alcohol, ether, benzene, solvent naphtha, or oleic acid, are given.

VI. [With H. STARCZEWSKA and J. KRZYKIEWICZ.] The heats of combustion of a number of motor mixtures containing alcohols have been determined in a Junker's calorimeter, and using Starczewska's bomb calorimeter. The former method gives a constant relative error, the results obtained by the latter method being 1.02 times as great. R. TRUSZKOWSKI.

**Analysis of the gradual oxidation prior to ignition of fuels in internal-combustion engines and the relation of such oxidation to detonation.**

W. M. ZAIKOWSKY, H. B. HOLROYD, and V. M. SOKOLOV (Physical Rev., 1929, [ii], 33, 264).—The change in oxidation of gasoline-air mixtures prior to ignition produced by the admixture of 0.25% of lead tetraethyl has been studied. The untreated mixture undergoes greater oxidation, but no corresponding increase of the compression pressure was observed, indicating that oxidation is not a homogeneous reaction. Lead tetraethyl and other anti-knock compounds reduce the rate of oxidation of the explosive mixture in the gaseous phase for a short time only, whilst oxides deposited on the tube walls remain as effective anti-oxidising agents. Lead oxide acts as a positive surface catalyst for carbon. The effectiveness of anti-knock compounds is attributed to their temporary elimination of oxygen molecules activated sufficiently to react directly with hydrocarbons. L. S. THEOBALD.

**Determination of lead tetraethyl in gasoline.**

L. J. CATLIN and J. E. STARRETT (Refiner Nat. Gasol. Mfr., 1930, 9, No. 7, 155).—To 100 c.c. is added a 10% solution of bromine in carbon tetrachloride 5 c.c. in excess of the volume required to produce a precipitate, decanting after 5 min. After washing with carbon disulphide and removing the solvent, the precipitate is boiled with 15% nitric acid, filtered, and the filtrate evaporated to fuming with sulphuric acid. After dilution the lead sulphate is allowed to deposit, and is then dissolved in ammonium acetate solution and titrated with ammonium molybdate, using tannin as external indicator. CHEMICAL ABSTRACTS.

**Gas-analysis apparatus.** JARRIER.—See I. **Adsorptive power of carbon for rubber.** DITMAR and PREUSZE.—See XIV. **Protection against toxics.** TURNER.—See XXIII.

**PATENTS.**

**Coal-washing apparatus.** A. WITZAL, A. G. RABEL, and H. F. STEVENS (U.S.P. 1,760,293, 27.5.30. Appl., 12.1.27).—The apparatus consists of a tank with two compartments, in the first of which the coal is separated from the slate and other refuse by means of an upward current of water flowing through a screen upon which the refuse collects, and whence it is discharged through an opening connecting the two compartments; it is finally removed from the tank by an endless conveyor. The water in the second compart-

ment is maintained in a more or less quiescent state; the height of the water level therein varies with the resistance which the material on the screen offers to the flow of water, and therefore with the amount of refuse which collects on the screen. The rate of discharge of the refuse from the screen can be varied by altering the angle of slope of a hinged plate at the discharge outlet, and the angle of the plate is controlled automatically by the height of the water in the second compartment in such a manner that the rate of discharge adjusts itself in accordance with the amount of refuse in the coal being washed. A. B. MANNING.

**Coke oven.** C. OTTO (U.S.P. 1,760,770, 27.5.30. Appl., 20.1.28. Ger., 16.11.26).—Each of the heating walls of a coke oven comprises a pair of runner walls and a series of parallel transverse binder walls extending between the runner walls and forming a series of vertical hairpin heating flues. The alternate binding walls, which extend vertically to the oven cover, constitute supporting columns and are made of materially greater thickness than the maximum thickness of the runner walls, in order to support the major part of the weight of the cover and loads resting thereon, and so enable the thickness of the runner walls to be decreased to permit quicker transmission of heat to the charge. A. B. MANNING.

**Coking retort ovens.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,764,065 and 1,764,496—7, 17.6.30. Appl., [A] 22.5.22, [B] 18.11.20, [C] 17.11.20. Renewed [A] 18.6.26).—(A) A coking retort oven has the horizontal or bus flue, which connects the tops of the vertical flame flues, so placed as to lie in the top structure above the coking chambers and heating walls. The ducts connecting the vertical flues to the horizontal flue are insulated from the heating walls, and the horizontal flue is insulated from those parts of the top of the battery which lie directly over the coking chambers. Overheating of the upper part of the coking chamber and undue cracking of the volatile distillation products are thereby avoided. (B) A coke-oven battery which can be heated either by producer gas or coke-oven gas is provided with a main at one side alternatively operable for feeding producer gas or waste gas, and having reversing valve connexions to alternate pairs of regenerators. Another main on the same side of the battery supplies either coke-oven gas to the heating walls or air to the pairs of regenerators between the alternate pairs referred to above. The coke-oven gas can be fed alternately to the flues on opposite sides of the battery. Reversing valves are provided between the second main and the regenerators with which it is connected, and means are provided also for regulating the air supply through the main when producer gas is used as the heating medium. When coke-oven gas is used it is burned in an atmosphere containing both air and a neutral diluent, e.g., waste gas; the latter is supplied through the first main, and the air through flow pipes connected directly to the atmosphere. (C) In a further modification a pair of air mains is provided on the side of the battery opposed to that having the producer (or waste) gas and coke-oven gas mains, the air mains being connected to alternate pairs of regenerators. Means are provided for permitting air to flow

alternately into each of the two air mains, and for regulating the volume of flow therein in accordance with the gas requirements.

A. B. MANNING.

**Retort [or combustion device].** W. F. RIEGER, Assr. to STOKER MATIC CORP. (U.S.P. 1,763,442, 10.6.30. Appl., 27.3.28).—A combustion device or burner for an underfed furnace comprises a mouthpiece to which the fuel is conveyed by means of a screw, and an air chamber below the mouthpiece and communicating therewith through a number of tapered air pipes. The edges of the mouthpiece rest on tubular metal pipes through which water is circulated.

A. B. MANNING.

**Manufacture of [activated] carbonaceous material.** A. OBERLE (U.S.P. 1,763,101—2, 10.6.30. Appl., [A] 3.11.23, [B] 31.1.25).—(A) Petroleum coke, or similar material, is heated in the presence of water and lime, or other calcareous material, to a temperature, preferably 480—510°, at which the water and volatile impurities are vaporised. The evolved vapours are distilled off under increased pressure, *e.g.*, 300 lb./in.<sup>2</sup>, which is subsequently reduced until the material, while still hot, is under a vacuum. Finally a current of air, carbon dioxide, or flue gas, etc. is passed through the material to remove any remaining volatile impurities. If desired, the activated carbon so produced may be further purified by treatment with an acid or with an organic solvent. (B) Petroleum coke is mixed with sawdust and acid sludge, and the mixture is set aside to permit the acid to disintegrate the sawdust. The mass is then treated with highly superheated steam, and, after being moulded if desired, may be carbonised further.

A. B. MANNING.

**Manufacture of fuels.** GEN. CARBONALPHA Co. (B.P. 313,889, 18.6.29. Holl., 18.6.28).—Gaseous, liquid, or solid fuels for use in internal-combustion engines are improved by the addition of carbon produced by the autocatalytic decomposition of carbon monoxide. This carbon has a low ignition point, *e.g.*, about 200°, and, in contradistinction to hydrogen, burns smoothly in the cylinder owing to its relatively lower speed of combustion. The speed of combustion of the mixed fuel may be controlled by the proportion of carbon added.

A. B. MANNING.

**Manufacture of liquid products from coal or other carbonaceous material by heating under pressure in the presence of hydrogen or gases or vapours containing hydrogen.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,386 and 335,513, [A] 3.10.29, [B] 31.5.29. [A] Holl., 7.11.28).—(A) The process is carried out in the presence of a catalyst consisting of a mixture of "Luxmass," pretreated as described in B.P. 314,859 (B., 1930, 1012) or in (B), and molybdenum or its compounds, *e.g.*, ferric molybdate or molybdenum sulphide. (B) Another suitable catalyst is prepared by dissolving "Luxmass" as far as possible in nitric acid, precipitating the solution with ammonia, and drying.

A. B. MANNING.

**Obtaining light hydrocarbons (benzines and petrols) during the low-temperature carbonisation of solid fuels.** A. MEIRO (B.P. 317,857, 22.8.29. Belg., 24.8.28).—The finely-crushed fuel is conveyed through a series of retorts in which it is subjected to

successively higher temperatures. The volatile products of distillation are fractionated, and the heavier hydrocarbons and part of the uncondensed gases are returned continuously to the retorts in order to undergo further decomposition into light hydrocarbons. The retorts are of metal, and are arranged horizontally one above the other in a setting heated by the gases from a furnace in which a part of the distillation gases is burned. Each retort is provided with a worm conveyor, and the material passes from one retort to the next lower one by gravity. Each retort has a gas outlet which is connected to the condenser in which the oils are fractionated, and is arranged immediately above the setting. Below the distillation retorts is an unheated inclined retort up which the semi-coke is conveyed to a closed storage bin, its lower end communicating through a siphon with a tar receiver.

A. B. MANNING.

**Gas generator.** W. G. PAQUETTE (U.S.P. 1,764,974, 17.6.30. Appl., 5.3.29).—A generator for the production of gas by the vaporisation of a liquid fuel comprises a closed compartment, another compartment open at the top and containing a movable bell, and a carburettor arranged below both compartments. A constant water level is maintained in the two compartments. A jet of water carries a current of air into the closed compartment, and thence to the carburettor, into which the liquid fuel is fed simultaneously. The carburetted air collects in the bell, the movement of which automatically shuts off the supplies of water (and therefore air) and liquid fuel when the bell has risen to a predetermined height, and re-establishes the connexions when the bell has again fallen.

A. B. MANNING.

**Gas generator.** F. W. STEERE, Assr. to SEMET-SOLVAY ENG. CORP. (U.S.P. 1,764,992, 17.6.30. Appl., 7.4.23).—A water-gas generator has a closed ash-pit below the grate through which alternate blasts of air and steam are passed. A number of pokers attached to a spider, which can be raised and lowered by hydraulic means, serve to break up and remove clinker and ash from the fuel bed. The grate may consist of a series of alternately fixed and movable rings, the latter being given a limited up-and-down movement by the motion of the pokers. In an alternative arrangement a single centrally located poker of relatively large diameter is used; the poker is hollow and is provided with perforations through which the air and steam may pass to the fuel bed. The grate may consist of a series of rotatable bars so designed as to crush any clinker caught between them and discharge it into the ash-pit.

A. B. MANNING.

**Producer gas generators, and apparatus for cleansing and cooling producer gas for automobiles etc.** A. E. HODGSON, N. C. JONES, and MOTOR OWNERS' PETROL COMBINE, LTD. (B.P. 334,888—9, 10.6.29).—(A) A generator for supplying gas to internal-combustion engines, particularly for automobiles, trucks, etc., and (B) a cooling and scrubbing apparatus for such gas, are described.

A. B. MANNING.

**Manufacture of mixed oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., Asses. of H. G. TERZIAN (B.P. 335,495, 29.1.30. U.S., 11.7.29).—The blast gases from a generator are burned with secondary air in a



carburettor and superheater. Oil and steam are then introduced into the carburettor and the lightly cracked vapours and oil gas are passed through the superheater and thence through a wash-box, wherein the hot gases take up more water vapour, and a condenser. The oil gas and remaining water vapour are then passed through the fuel bed of the generator, whereby the oil gas is cracked to re-form gas of lower calorific value and sp. gr., and the excess carbon released, which is deposited on the fuel, reacts with the water vapour to form water-gas.

A. B. MANNING.

**Production of combustible gas.** W. BERTELSMANN and A. C. BECKER (B.P. 335,228, 18.3.29).—A non-poisonous combustible gas is produced by freeing ordinary coal gas from carbon monoxide, *e.g.*, by conducting it over catalysts in the presence of hydrogen ( $3\text{H}_2 + \text{CO} = \text{CH}_4 + \text{H}_2\text{O}$ ), or by separation by compression and cooling, etc., and the gas is then restored to its original sp. gr., calorific value, and speed of ignition by the suitable addition of hydrogen, carbon dioxide, and/or nitrogen. The hydrogen and carbon dioxide may be derived from the carbon monoxide by passing it with steam over a heated catalyst. Any excess of carbon dioxide may be removed by washing. The hydrogen required may be obtained by electrolysis, utilising current produced during the periods when the external demand is small. The oxygen produced at the same time may be used for the continuous production of water-gas.

A. B. MANNING.

**Desulphurisation of gases and vapours.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,394, 8.10.29. Holl., 23.10.28).—The gases are passed over a contact mass containing thorium, preferably as oxide, at a raised temperature, *e.g.*, 650°. Small quantities of promoters, *e.g.*, copper, nickel, magnesium oxide, or lead oxide, may be added to the thorium oxide.

A. B. MANNING.

**Storage of acetylene.** F. ROFFEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,820, 28.11.29).—Dry acetylene is stored under pressure in a cylinder containing absorbent solid (kieselguhr, kapok, etc.) impregnated with a solvent for acetylene, preferably of b.p. above 150°. Suitable solvents are  $\beta$ -ethoxyethyl acetate, glycerol diacetate,  $\beta$ -diethoxy-*n*-propyl acetate, glycol diacetate, glycerol diethyl ether, etc.

C. HOLLINS.

**Tar-separating apparatus.** H. HENNEBUTTE and E. GOUTAL, ASSRS. to SOC. ANON. "LA CARBONITE" (U.S.P. 1,760,058, 27.5.30. Appl., 28.11.27. Fr., 2.12.26).—The hot gases and vapours obtained from the carbonisation of wood, peat, lignite, etc. are led into a receptacle through a central, stationary, conical conduit arranged therein. A rapidly rotating truncated cone to which blades or stirrers are attached is mounted within the lower part of the conical conduit, and serves to bring the incoming gases and vapours into intimate contact with the liquid in the receptacle. Curved tubes mounted on the walls of the stationary conduit, and having their ends immersed in the liquid, bring about a constant circulation of the liquid within the receptacle. The fixed gases, together with the water vapour if the apparatus is operated at a suitably high temperature, are

withdrawn from the top of the receptacle, while the condensed liquid is withdrawn through an overflow pipe which maintains a constant level within the vessel.

A. B. MANNING.

**Distillation of tar.** A. A. MACCUBBIN and J. ZAVERNIK, ASSRS. to BARRETT CO. (U.S.P. 1,759,816, 20.5.30. Appl., 11.9.28).—The tar is pumped continuously through a pipe coil wherein it is heated under pressure, but without objectionable decomposition, and passes thence to a vapour box wherein the oil vapours are separated from the pitch residue, and are withdrawn to a condenser. The pitch residue is passed continuously to a vacuum box wherein it undergoes further distillation with or without additional heating. Increased oil yields and pitches of high m.p. are obtained.

A. B. MANNING.

**Conversion of olefines into other hydrocarbons.** J. Y. JOHNSON. From I. G. FARRENIND. A.-G. (B.P. 336,234, 4.5.29).—Olefine-containing gases, not necessarily freed from hydrogen sulphide, are passed at atmospheric or higher pressures over a silicon catalyst contained in a tinned chromium-nickel (V2A) steel reaction vessel at 400–600°. Higher olefines or aromatic substances are produced, according to the velocity of the gas over the catalyst. At lower pressures higher temperatures are required. *E.g.*, ethylene, present in a gas mixture, was converted into methane, ethane, butylene, benzene, and toluene. T. A. SMITH.

**Conversion of hydrocarbon oils.** PETROLEUM CONVERSION CORP., ASSSES. of A. P. SACHS (B.P. 336,380, 19.9.29. U.S., 6.10.28).—When hot inert gas is used as a direct heating medium in the cracking of oils or similar processes, it removes sulphur compounds in the form of hydrogen sulphide, and has to be freed from this before recycling. This can be effected by extraction with water at a point in the system where the gas is sufficiently cool.

T. A. SMITH.

**Cracking of liquid hydrocarbons.** O. D. LUCAS (B.P. 336,269, 10.4.29).—Oil is heated to cracking temperature in heat exchangers by means of hot diphenyl or diphenyl oxide. The heating fluid is circulated through a pipe heater, and does not undergo any decomposition. Those parts of the plant in which the cracking stock is heated to decomposition temperature are arranged so that they can be readily freed from carbon.

T. A. SMITH.

**Purification of benzol.** C. F. HAGEN (U.S.P. 1,758,661, 13.5.30. Appl., 13.8.28).—A blended distillate suitable for motor fuel is obtained by mixing discoloured commercial benzol with a heavier hydrocarbon oil and distilling the mixture until a clear distillate is obtained, rejecting the first runnings and stopping the distillation when tar oils begin to come over.

T. A. SMITH.

**Purification of light oils extracted from coal gas.** KOPPERS CO., ASSSES. of A. R. ALBRIGHT (B.P. 335,155, 15.4.29. U.S., 10.10.28).—The crude light oils are washed first with acid and then with a solution of sodium phenoxide in order to free them from sulphonic acids. The phenoxide solution used is that obtained in the process of removing the phenols from

the gas liquor, the latter being washed with a suitable transfer medium which is subsequently extracted with an alkali.

A. B. MANNING.

**Absorption process [for removal of light hydrocarbons from natural gas].** C. D. COULTER, Assr. to SOUTH WESTERN ENG. CORP. (U.S.P. 1,759,346, 20.5.30. Appl., 16.8.26).—Natural gas is commingled and washed with a heavy absorbing oil at a predetermined temperature, and under pressure such that only hydrocarbons condensable at that temperature are absorbed. The pressure on the oil is then released without appreciable change in temperature, liberating thereby some of the lighter hydrocarbons. The remainder of the laden absorbing oil is heated and the absorbed hydrocarbons and a portion of the oil are vaporised, cooled, and fractionally condensed. The condensed absorbing oil is returned to the main portion of oil in the system after it has left the absorption operation and before it reaches the heating stage. The vapours are cooled and fractionally condensed again, leaving the still lighter hydrocarbon vapours in vaporous form, the condensing action being carried on so that the gasoline condensate is not forcibly mixed with the non-condensed vapour.

H. S. GARLICK.

**Motor fuel. Liquid fuel.** C. O. JOHNS, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,757,837—8, 6.5.30. Appl., 21.5.24).—In the production of anti-knock fuels, the addition of a first small portion of lead tetraethyl has more effect than have the successive quantities added. Additional anti-knock effect is obtained by the addition of benzol and/or alcohols, instead of lead tetraethyl. Examples of suitable mixtures are: gasoline 80%, benzol 20%, lead tetraethyl 1.5 c.c./gal. of mixture; gasoline 95%, ethyl alcohol 5%, lead tetraethyl 1.5 c.c./gal. of mixture.

T. A. SMITH.

**Preparation of [anti-knock] motor fuels.** F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,758,898, 13.5.30. Appl., 21.7.22).—The heavier fractions of gasoline are vaporised and mixed with a limited amount of air. The mixed gases, containing not more than 4 mols. of oxygen per mol. of hydrocarbon, are passed through a tube heated at 230–370°. The products are condensed, treated to remove gummy matter by steam-distillation or dilute soda washing, and remixed with the gasoline.

T. A. SMITH.

**Rectification of lubricating oil.** M. CZARNY and F. C. FROLANDER (B.P. 336,202, 6.6.29).—Lubricating oil from an internal-combustion engine is pumped through a cloth filter to remove solid matter and then through a small vessel containing a wick through the upper end of which a current of air is drawn. Any light constituents in the oil are drawn up the wick and evaporated in the current of air. Oil not drawn up the wick is returned to the engine.

T. A. SMITH.

**Manufacture of lubricants.** ALOX CHEM. CORP. (B.P. 335,152, 10.4.29. U.S., 18.12.28).—The saponifiable products of controlled oxidation of paraffins obtainable by the processes of B.P. 303,560 and 309,382 (B., 1929, 219, 467) are added, with or without "mould oils," to lubricating oils in order to decrease internal

friction and enhance the lubricating power. The amount added is preferably 0.25–0.75%. The crude oxidation products may be extracted with 95% alcohol; preferably they are saponified with caustic alkali and the soap solution, after dilution and clearing, is acidified, the acid precipitate being finally steamed to remove volatile products.

C. HOLLINS.

**Apparatus for pulverising coal and like substances.** W. J. DRUMMOND, Assr. to ASHINGTON COAL CO., LTD. (U.S.P. 1,781,572, 11.11.30. Appl., 4.1.30. U.K., 24.4.29).—See B.P. 328,437; B., 1930, 750.

**Carbonising process.** W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,781,613, 11.11.30. Appl., 27.2.25).—See B.P. 261,954; B., 1927, 99.

**Gas-reversing device for regenerative coke ovens.** COLLIN & Co. (B.P. 338,040, 20.12.29. Ger., 27.12.28).

**Burner for heavy oil.** D. PIZZI (B.P. 337,983, 11.11.29).

**Liquid-fuel burners.** S. M. PYANKOW (B.P. 337,787, 6.8.29).

**Filters or strainers for petrol or other liquids [in pumps].** A. G. FENN, and ANGLO-AMERICAN OIL CO., LTD. (B.P. 337,539, 27.9.29).

**Crushing of solids** (B.P. 336,730). **Hydrogenation** (B.P. 336,569). **Deposition of dust** (B.P. 336,201). **Separation of dust from air etc.** (B.P. 336,221).—See I. **Gases containing hydrogen** (B.P. 336,944).—See VII. **Lubricating grease** (U.S.P. 1,749,251).—See XII. **Synthetic resins** (B.P. 314,810).—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Thermal requirements and operating characteristics of the urea autoclave.** K. G. CLARK and U. L. GADDY (Ind. Eng. Chem., 1930, 22, 1084–1087).—Investigations were made on the continuous synthesis of urea from liquid ammonia and liquid carbon dioxide, using an autoclave working at 100 atm. and 183° with a conversion of 35–40%. In the experimental plant used, the urea was separated from unconverted ammonia-carbon dioxide compounds by steam-distillation of the latter. By the use of a series of thermocouples and blank experiments it was established that the reaction is exothermic, the heat of reaction being about 120 kg.-cal./kg. of urea. A heat balance is given and it is considered that with well-designed large-scale equipment the steam consumption in operating this process should be negligible. A series of corrosion tests on samples placed within the autoclave are given in the form of loss in weight/unit surface/24 hrs. They indicate the suitability of high-silicon chromium-nickel steel as a structural material.

C. IRWIN.

**Phenols from petroleum.** HOLZMANN and VON PILAT.—See II. **By-products of saccharin manufacture.** HERZOG.—See XII. **Detection and determination of *p*-hydroxybenzoic acid.** WEISS.—See XIX.

PATENTS.

**Manufacture of condensation products from diolefines and hydrocarbons.** I. G. FARREND.

A.-G. (B.P. 335,512, 5.7.29. Addn. to B.P. 315,312; B., 1930, 1103).—The following additional examples are given. Isoprene in toluene, heated with sodium at 160° for 1 hr., gives  $\epsilon$ (or  $\gamma$ )-phenyl- $\gamma$ (or  $\epsilon$ )-methyl- $\Delta^{\beta}$ -amylene, b.p. 218—222°.  $\beta\gamma$ -Dimethylbutadiene similarly yields  $\epsilon$ -phenyl- $\beta\gamma$ -dimethyl- $\Delta^{\beta}$ -amylene, b.p. 235—238°, whilst from butadiene in tetrahydronaphthalene a butenyltetrahydronaphthalene, b.p. 270—272°, is obtained.

C. HOLLINS.

**Manufacture of alcohols [from olefines].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,551, 23.5. and 4.6.29).—Ethylene (or other olefine, diolefine, etc.) is led with steam over the following catalysts: platinum, gold, silver, copper, iron, nickel, cobalt, chromium, tantalum, vanadium, tungsten, molybdenum, manganese, or compounds of these, especially oxalates, carbonyls, etc. which yield the metal under the conditions of reaction; or bismuth vanadate, copper vanadate, or copper phosphate, or mixtures of these. Examples are platinised pumice at 150°, copper phosphate on pumice at 155°, tungsten trioxide and ferric oxide on silica gel at 300°, copper oxide and tungsten trioxide on active carbon at 300°.

C. HOLLINS.

**Manufacture of methanol [methyl alcohol, from carbon monoxide].** H. DREYFUS (B.P. 335,962, 5.6.29).—Carbon monoxide and hydrogen at 50—100 atm. are passed over zinc oxide or mixed zinc-chromium oxides at 300—450° and then, after removal of methyl alcohol, over a copper or manganese catalyst or other catalyst sensitive to sulphur-poisoning. Several vessels of each catalyst may be used in series.

C. HOLLINS.

**Manufacture of oxygenated organic compounds [pyrolysis of methyl alcohol].** H. DREYFUS (B.P. 335,631, 2.7.29).—Methyl alcohol vapour is converted into ethyl alcohol and other products when passed, preferably with 1—2 vols. of nitrogen, at 500—600° over oxides, hydroxides, or alkoxides of sodium, potassium, barium, or calcium in a copper tube.

C. HOLLINS.

**Purification of synthetic *n*-butyl alcohol.** R. RILEY, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,683, 13.8.29).—The material is purified by electrolytic reduction; *e.g.*, it is stirred vigorously with dilute sulphuric acid or bisulphite solution in the cathode cell with a mercury cathode, the current density being 0.05 amp./cm.<sup>2</sup>, and the alcohol layer is subsequently distilled.

C. HOLLINS.

**Manufacture of urea.** W. W. TRIGGS. From A. B. LAMB (B.P. 335,913, 27.6.29).—Liquid ammonia and liquid carbon dioxide are separately fed into an autoclave at 150° and the equilibrium mixture (40% of urea) is withdrawn from the top of the autoclave and transferred at atmospheric pressure to a still, where ammonia and carbon dioxide are removed by heat, dried, and returned separately to the liquefiers; the still liquid is an aqueous solution of urea.

C. HOLLINS.

**Manufacture of thiourea [thiocarbamide].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,111, 19.10.29).—Calcium cyanamide is treated with hydrogen sulphide in absence of liquids or in presence of only sufficient liquid to form pastes with the solid. The product may be extracted with organic solvents, *e.g.*,

alcohol-ether, alcohol-carbon tetrachloride, benzene, with or without previous removal of water.

C. HOLLINS.

**Production of substances which may be used as treatment agents in the textile, leather, and allied industries.** H. T. BÖHME A.-G. (B.P. 317,039, 26.6.29. Ger., 9.8.28).—Wetting, cleaning, foaming, and dispersing agents are produced by cold sulphonation, in presence of acetic anhydride or acetyl chloride, of the alcohols derived from fatty acids by replacement of CO<sub>2</sub>H by CH<sub>2</sub>·OH (cf. B.P. 308,824; B., 1930, 809). The alcohol from oleic acid, *e.g.*, is sulphonated with concentrated sulphuric acid in acetic anhydride.

C. HOLLINS.

**Production of emulsions.** G. SCHICHT A.-G., and E. ULBRICH (B.P. 314,072, 17.6.29. Czechoslov., 22.6.28).—Esters of aminoalcohols are used as emulsifying agents for oil-in-water emulsions, *e.g.*, the dihydroxypropylaniline ester of castor oil acids, or *N*- $\beta$ -hydroxyethylpiperidine ester of phthalic acid. The agents described in B.P. 309,842, 295,024, and 306,116 (B., 1929, 590; B., 1930, 467, 809) are disclaimed.

C. HOLLINS.

**Manufacture of polymerisation products of unsaturated ketones.** I. G. FARBERIND. A.-G. (B.P. 335,185, 15.3.29. Ger., 16.3.28. Cf. B.P. 307,936; B., 1930, 1016).—Ketones containing an aliphatic chain having only one double linking are polymerised in the form of aqueous emulsions; *e.g.*, the methylene derivative of methyl ethyl ketone is agitated for 8 days at 60° with 1% aqueous sodium oleate.

C. HOLLINS.

**Catalytic [reduction] process.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,043, 18.3.29. U.S., 18.5.28).—In the hydrogenation of ketones or aldehydes to alcohols in presence of an oxide catalyst (oxides of zinc, magnesium, aluminium, silicon, chromium, vanadium, iron, etc.) having a dehydrating as well as a hydrogenating activity, the dehydrating effect is suppressed by addition of an oxide, hydroxide, carbonate, or other weak-acid salt of an alkali or alkaline-earth metal, *e.g.*, potassium or sodium carbonate. The reduction of acetone to isopropyl alcohol in presence of zinc chromite and potassium carbonate, and of aldol to  $\alpha\gamma$ -butylene glycol, is described.

C. HOLLINS.

**Manufacture of (A, B) esters [vinyl acetate] and (c) di-esters of carboxylic acids [ethylidene diacetate].** CANADIAN ELECTRO PRODUCTS Co., LTD., Assecs. of F. W. SKIRROW and G. O. MORRISON (B.P. 308,169, 308,170, and Addn. B.P. 335,223, 18.3.29. U.S., [A, B] 17.3.28).—Acetylene is passed into acetic acid to which has been added a catalyst precipitated by adding below 45° acetylsulphuric acid or a solution of sulphur trioxide in acetic acid to a solution of mercuric oxide in acetic acid. (A, B) For vinyl acetate the temperature of reaction with the acetylene should be 30—35°, and vinyl acetate should be removed as formed by means of large excess of acetylene. For ethylidene diacetate the optimum temperature is 80°. (c) The yield of diacetate is increased by condensing the vinyl acetate in the exit gases and returning it to the reaction.

C. HOLLINS.

**Manufacture of glycidic acids.** A. A. KAUFMANN

(B.P. 335,391, 8.10.29).— $\alpha$ -Unsaturated aldehydes are treated with alkali and either alkali hypobromite or hydrogen peroxide. Cinnamaldehyde, aqueous sodium hydroxide, and sodium hypobromite or hydrogen peroxide give phenylglycidic acid; methylglycidic acid, m.p.  $84^{\circ}$ , is obtained from crotonaldehyde.

C. HOLLINS.

#### Sulphonation of pyridine or its homologues.

I. G. FARBENIND. A.-G. (B.P. 335,817, 26.11.29. Ger., 26.11.28).—Pyridines are sulphonated with oleum in presence of mercuric sulphate.  $\alpha$ -Picoline and pyridine at  $225^{\circ}$  yield 3-sulphonic acids.

C. HOLLINS.

**Manufacture of 3-hydroxy- $\alpha$ -picoline.** I. G. FARBENIND. A.-G. (B.P. 335,818, 26.11.29. Ger., 26.11.28).— $\alpha$ -Picoline-3-sulphonic acid is fused with caustic alkali at  $210$ – $240^{\circ}$  to give 3-hydroxy- $\alpha$ -picoline, m.p.  $164$ – $166^{\circ}$ .

C. HOLLINS.

#### Manufacture of condensation products [mothproofing agents] from aldehydes and phenols.

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,547, 19.4.29).—Formaldehyde is condensed in acid medium with 2 mols. of a mixture of phenols comprising at least 1 mol. of a *p*-halogenophenol; or a non-hydroxylated aromatic aldehyde is similarly condensed with 2 mols. of a mixture of phenols comprising at least 1 mol. of a *p*-alkylated or *p*-halogenated phenol. The phenols must contain a free *o*-position and no salt-forming groups as substituents. Sulphuric acid, sulphuric and acetic acids, aluminium chloride, or ferric chloride may be used as condensing agents. The products are hydroxylated di- and tri-arylmethanes and are useful for mothproofing. Examples are: *o*-sulphobenzaldehyde with 2:4-dichlorophenol containing 2% of *p*-chlorophenol and 5% of 2:6-dichlorophenol, or with chlorinated crude *m*- and *p*-cresol mixture; formaldehyde with *p*-fluorophenol containing phenol.

C. HOLLINS.

**Manufacture of optically active 1-phenyl-2-methylaralkylaminopropanols-1 and 1-phenyl-2-methylaminopropanol-1 [ $\beta$ -aralkylmethylamino- and  $\beta$ -methylamino- $\alpha$ -hydroxy-*n*-propylbenzenes].** I. G. FARBENIND. A.-G. (B.P. 318,488, 29.8.29. Ger., 3.9.28).—*dl*- $\beta$ -Benzylmethylamino- $\alpha$ -hydroxy-*n*-propylbenzene, m.p.  $72$ – $73^{\circ}$ , prepared by reduction of  $\beta$ -benzylmethylaminopropiophenone (cf. Can. P. 291,001), is resolved by means of *d*-tartaric acid. The less soluble *d*-tartrate of the *d*-base, m.p.  $101$ – $102^{\circ}$ , and the *l*-base, m.p.  $49$ – $50^{\circ}$ , are described. Catalytic hydrogenation of the *l*-base or its hydrochloride removes the benzyl group as toluene and gives *l*- $\beta$ -methylamino- $\alpha$ -hydroxy-*n*-propylbenzene, m.p.  $39$ – $40^{\circ}$  (hydrochloride, m.p.  $215$ – $216^{\circ}$ ,  $\alpha_D$   $-35^{\circ}$ ); the *d*-base similarly gives the *d*-compound. Resolution of the *dl*-benzyl derivative with *l*-tartaric acid gives first the *l*-tartrate of the *l*-base.

C. HOLLINS.

**Manufacture of 2:4-di-(3'-nitrophenyl)-6-hydroxytriazine-1:3:5 [6-hydroxy-2:4-di-m-nitrophenyl-1:3:5-triazine].** I. G. FARBENIND. A.-G. (B.P. 335,783, 30.10.29. Ger., 31.10.28).—6-Hydroxy-2:4-diphenyl-1:3:5-triazine, m.p.  $283^{\circ}$ , is dinitrated in *m*-positions by mixed acid at  $10$ – $15^{\circ}$ ; the product has m.p.  $280$ – $281^{\circ}$ .

C. HOLLINS.

**Manufacture of mercapto[thiol]benzthiazole.**

IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 335,567, 26.3.29).—A solution of sulphur in carbon disulphide mixed with aniline is caused to flow upwards through a cylindrical pressure vessel at  $200$ – $275^{\circ}$ , reaction being completed during the time of passage, and overflows into an inner cylinder from which the product is drawn off. Hydrogen sulphide is blown off through a valve at the top of the vessel, and reduces the pressure to any desired degree. The process is continuous, and the apparatus may be used for other liquid-phase reactions under pressure.

C. HOLLINS.

**Manufacture of secondary aromatic amines [dinaphthylamines].** GOODYEAR TIRE & RUBBER CO., ASSECS. of A. M. CLIFFORD (B.P. 310,871, 20.3.29. U.S. 2.5.28).— $\alpha$ - or  $\beta$ -Naphthol, or an equimolecular mixture, is heated with anhydrous ammonia (1.5 mols.) in a closed vessel at  $300$ – $310^{\circ}$ . The pressure initially is about 43 atm.

C. HOLLINS.

**Production of aromatic amino-derivatives from monoazo compounds, and of aminoazo compounds containing a  $\beta$ -naphthol component simultaneously therewith.** BRIT. RES. ASSOC. FOR THE WOOLLEN AND WORSTED INDUSTRIES, and A. T. KING (B.P. 335,645–6, 6.7.29).—(A) Monoazo compounds, other than Fast Acid Blue RH and others known to be reducible with sulphur dioxide, which do not contain a  $\beta$ -naphthol residue, are reduced with a sodium sulphite of the composition  $\text{Na}_2\text{SO}_3 \cdot 2\text{NaHSO}_3$  in alcohol or pyridine under reflux. (B) Secondary disazo compounds in which the end component is  $\beta$ -naphthol or a derivative thereof are reduced at  $15^{\circ}$  with aqueous sodium hydrogen sulphite, preferably with addition of pyridine, whereby only the azo grouping remote from the  $\beta$ -naphthol residue is reduced.

C. HOLLINS.

**Manufacture of dye intermediates [1-chloro-anthraquinone-2-carboxylic acid].** W. SMITH, J. PRIMROSE, and SCOTTISH DYES, LTD. (B.P. 335,232, 15.4.29).—2-Methylanthraquinone is chlorinated in weak oleum at  $0^{\circ}$ , whereby the proportion of by-products may be used in the direct production of 1-chloroanthraquinone-2-carboxylic acid from 2-methylanthraquinone or from 2-*p*-toluoylbenzoic acid.

C. HOLLINS.

**Halogenation of isocyclic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,948, 3.4.29).—Aromatic or hydroaromatic compounds are chlorinated or brominated by treatment with hydrogen chloride or bromide in oleum or chlorosulphonic acid in presence of carriers. Dibenzanthrone with hydrogen bromide, oleum, and antimony at  $60$ – $65^{\circ}$  gives a dibromo-compound (navy-blue vat dye). The preparation of di-, tri-, and tetra-bromopyranthrones (orange to orange-red vat dyes), bromo-1:2:6:7-dibenzpyrene-3:8-quinone (yellow-orange vat dye), bromotoluene, b.p.  $138$ – $140^{\circ}/15$  mm., bromobenzanthrone, and bromo-iodoanthanthrone (blue-red vat dye), is also described.

C. HOLLINS.

**Storage of acetylene (B.P. 335,820. Converting olefines into hydrocarbons (B.P. 336,234).—See II. Naphthenates (B.P. 335,863).—See VII. Lactic and acetic acids (B.P. 335,596). 2:3[ $\beta$ ]-butylene glycol by fermentation (B.P. 335,280).—See XVIII.**

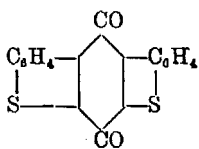
## IV.—DYESTUFFS.

## PATENTS.

**Manufacture of sulphurised dyes.** G. B. ELLIS. From CHEM. WORKS FORMERLY SANDOZ (B.P. 335,297, 6.7.29).—Hydroxylated phenazines are thionated in presence of molybdenum compounds. 3:7-Aminohydroxyphenazine, dissolved in sodium hydroxide solution, is heated with a solution of sodium polysulphide and molybdic acid at 110° for 30–32 hrs. to give a clear bluish-bordeaux-red sulphide dye. 3-Amino-7-hydroxy-2-methylphenazine similarly with ammonium molybdate or phosphomolybdic acid gives a dark red, as does its 8-carboxylic acid or 8-chloro-derivative; etho-safranone yields a bluish-red. C. HOLLINS.

**Manufacture of stable reduction compounds of [indigoid] vat dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,919, 11.5.29. Addn. to B.P. 334,878; B., 1930, 1144).—A 6:6'-dihalogeno-4:4'-dimethylthiindigo is reduced with alkali and a reducing agent (hyposulphite) other than alkali hydrogen sulphide, and the solution is treated with acid, or preferably with carbon dioxide. Alcohol is added with advantage during reduction. C. HOLLINS.

**Manufacture of vat dyes of the dithionaphthylenequinone [benzbisthionaphthenequinone] series.** I. G. FARBENIND. A.-G. (B.P. 314,527, 19.6.29. Ger., 29.6.28).—3:3'-Dicarboxy-2:2'-dithionaphthyl ketone, obtainable from *o*-thiolphenylglyoxylic acid and  $\alpha$ -dichloroacetone, is converted by heating with acetic anhydride into an anhydro-compound, m.p. 272–273°, which at 300° passes into benzbisthionaphthenequinone (annexed formula), m.p. 295°. The same product may be obtained from the 3-mono-carboxylic acid, m.p. 268–269°, prepared by condensing *o*-phenylglyoxylic acid with chloroacetone and causing the resulting 2-bromoacetyl-3-thionaphtheneic acid to react with *o*-thiolbenzaldehyde. The quinone is a golden-yellow vat dye for wool. C. HOLLINS.



**Manufacture of [vat] dyes of the dibenzanthrone series.** SOC. CHEM. IND. IN BASLE (B.P. 314,903, 4.7.29. Switz., 4.7.28. Addn. to B.P. 262,774 and 294,486; B., 1928, 517; 1930, 9).—4:4'-Dibenzanthronyl is heated at 140–150° in trichlorobenzene with addition of ferric chloride to give a blue vat dye fast to washing. C. HOLLINS.

**Preparation of indanthrone dyes.** NEWPORT CO. (B.P. 314,803, 28.6.29. U.S., 2.7.28. Addn. to B.P. 297,692; B., 1928, 847).—Halogenated indanthrones are dehalogenated to any desired degree by heating with a primary arylamine and basic copper acetate, with or without sodium acetate. 3:3'-Dichloro- or -dibromo-indanthrone, refluxed with aniline and basic copper acetate, gives a pure, brilliant indanthrone. C. HOLLINS.

**Manufacture of monoazo [acid] dyes for wool.** I. G. FARBENIND. A.-G. (B.P. 308,830, 28.3.29. Ger., 30.3.28).—Sulphonic acids of  $\beta$ -naphthylamine or 2:8-aminonaphthol, or *N*-derivatives thereof, are coupled with diazotised 5-nitro-2-aminodiphenylsulphone- $\alpha$ -sul-

phonic acids. Examples are: 5-nitro-2-aminodiphenylsulphone-3'-sulphonic acid  $\rightarrow$  methyl- $\beta$ -naphthylamine-7-sulphonic acid (reddish-blue), or  $\beta$ -naphthylamine-7-sulphonic acid (reddish-violet), or  $\gamma$ -acid (greenish-blue). Similar dyes are obtained from 4'-methyl and 4'-methoxy-derivatives of the diazo component.

C. HOLLINS.

**Manufacture of an azo [acid] dye for wool.** I. G. FARBENIND. A.-G. (B.P. 335,705, 30.8.29. Ger., 4.10.28. Cf. B.P. 15,163 of 1913; B., 1914, 784).—3:3'-Dichloro-4:4'-diamino-5:5'-dimethyltriphenylmethane, obtainable from benzaldehyde and 3-chloro-*o*-toluidine, is tetrazotised and coupled with 1-(2-chloro-5-sulphophenyl)-3-methylpyrazolone to give a yellow acid wool dye fast to light. C. HOLLINS.

**Manufacture of [metal complex compounds of azo] dyes.** SOC. CHEM. IND. IN BASLE (B.P. 335,523, 23.4.29. Addn. to B.P. 307,705; B., 1930, 983).—Chromium, or other metal or metals having at. wt. between 45 and 59, is introduced into dyes obtained by coupling the dye J-acid  $\rightarrow$  J-acid (acid-coupled) with a diazotised *o*-aminophenol, or with 2 mols. of a diazotised anthranilic acid, or with 1 mol. of a diazotised *o*-aminophenol and 1 mol. of another diazo component, always excluding components having a salicylic acid grouping. [Stat. ref.] C. HOLLINS.

**Manufacture of chromium compounds of azo dyes forming metallic compounds.** A. CAREMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,169, 16.5.29).—Chromable azo dyes containing at least one carboxylic or sulphonic acid group are prechromed in presence of organic bases, e.g., dimethylamine, aniline, cyclohexylmethylamine, pyridine. The process is applicable to the production of chromed dyes hitherto only obtainable in acid media, or not previously obtainable. Examples are: 4-chloro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  1-phenyl-3-methyl-5-pyrazolone (red) or 2:4-dihydroxyquinoline (bordeaux-red); 5-nitro-*o*-aminophenol  $\rightarrow$  Laurent acid (green); 1:2:4-aminonaphtholsulphonic acid  $\rightarrow$  1:8-naphtholsulphonic acid (blue), or phenylmethylpyrazolone (bordeaux-red), or 1-*p*-sulphophenyl-3-methyl-5-pyrazolone (bordeaux-red); anthranilic acid  $\rightarrow$  Schäffer acid (brown-red), 2:4-dihydroxyquinoline (yellow-orange), 1-phenylpyrazolone-3-carboxylic acid (red-yellow), or 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow); 5-sulphoanthranilic acid  $\rightarrow$  phenylmethylpyrazolone (yellow); 4-chloroaniline-3-sulphonic acid  $\rightarrow$  salicylic acid (yellow).

C. HOLLINS.

**Manufacture of insoluble azo dyes [ice colours and pigments].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 335,555, 21.6.29).—A 2:3-hydroxynaphthoic arylamide is coupled in substance or on the fibre with a diazotised amine of the type Ar·NH·Ar·X·Ar·NH<sub>2</sub>, where Ar is an aromatic residue and X a direct linking or an azo group, or of the type Ar·NH·P·NH<sub>2</sub>, where P is a condensed aromatic system. Examples are: 4-amino-4'-anilindiphenyl  $\rightarrow$  5-chloro-*o*-toluidide or *p*-chloroanilide (claret, becoming reddish dark blue on removal of nitroso-group with phenylhydrazine-*m*-sulphonic acid); 4-amino-4'-anilinoazobenzene  $\rightarrow$  4- or 5-chloro-*o*-toluidide,  $\alpha$ - or  $\beta$ -naphthylamide,

anilide, 4- or 5-chloro-*o*-anisidide (garnet, becoming black by removal of nitroso-group). C. HOLLINS.

**Acid wool dyes of the anthraquinone series.** G. KRÄNZLEIN, F. ROEMER, and E. KRONHOLZ, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,976, 11.11.30. Appl., 19.12.28. Ger., 23.12.27).—See B.P. 302,928; B., 1930, 502.

**Halogenated isocyclic compounds** (B.P. 335,948).—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Ageing of cotton contained in rubber goods.** G. BARR (Trans. Inst. Rubber Ind., 1930, 5, 31—47).—Dyed, rubbered balloon fabric on summer exposure may actually increase in strength for the first month or so on account of the "locking" effect of the perished rubber on the fibres. The practice of dyeing yellow the outer cotton ply of a doubled-rubbered fabric aids, however, the preservation not only of the rubber, but also of the cotton. Ultra-violet light causes preferential deterioration of the cotton, whereas tropical sunshine acts more rapidly on the rubber and sulphur, the difference being so great that it is impracticable to use the mercury lamp to forecast the value of any scheme of protection for tropical service. Aluminium finishes protect by virtue of their reflecting power, which excludes not only actinic light, but also heat rays. The portion of the solar spectrum most active in inducing deterioration in cotton appears to coincide with that producing maximum effect with silver bromide, viz., the violet and near ultra-violet. The deterioration of cotton, like that of rubber, on exposure to sunlight is essentially a process of oxidation; practically no change occurs in an evacuated glass tube. Traces of acid in cloth are of serious importance, particularly in view of the temperatures attained during vulcanisation; the rate of loss of strength is approximately proportional to the weight of acid in the cloth and also is doubled for a rise of about 8°.

D. F. TWISS.

**Determination of crude fibre by use of a new kind of filter ["Cella" filter].** K. FEIST and E. KUNTZ (Z. Unters. Lebensm., 1930, 59, 480—483).—The filtration of fibrous residues after treatment with sulphuric acid or alkali may be conveniently performed by means of "Cella" filters, composed of pure cellulose and having pores 0.5—3.0  $\mu$ . Repeat experiments with new filters gave very concordant results, but the re-use of filters causes an apparent increase in the amount of crude fibre and is not recommended. Employment of small-size filters with fine pores is not advantageous, owing to the increase in time required for filtration.

H. J. DOWDEN.

**Variations in the lignin content of pine wood.** P. KLASON (Svensk Pappers-Tidn., 1929, 32, 527—530; Chem. Zentr., 1930, i, 3773).—The average lignin content of samples of pine wood from northern and southern Sweden was 28.1%, with variations of 1% only. Hence climate does not appear to affect the lignin content. On the assumption that lignin consists of 70% of coniferylaldehyde and 30% of coniferyl alcohol, wood should contain 4.1% OMe; the observed value was 4.3%.

A. A. ELDRIDGE.

**Determination of mechanical wood pulp [in paper].** A. NOLL and F. HÖLDER (Papier-Fabr., 1930, 28, 700—702).—The bone-dry paper (4 g.) is disintegrated (special apparatus described) with 200 c.c. of hot water for 2 min., and the disintegrator washed down with a further 25 c.c. of water. The pulp is kept in a water-bath at 25° for 30 min., in order to obtain regular swelling of the fibres, 100 c.c. of *N*-potassium permanganate are added, and the mixture is kept at 25° for 1 hr., with continued stirring, and filtered. Then 30 c.c. of 0.1*N*-oxalic acid are added to 50 c.c. of water, brought to the b.p., acidified with sulphuric acid, and to this are added 10 c.c. of the filtrate. The excess of oxalic acid is titrated with permanganate. A table is given relating permanganate consumption with mechanical wood-pulp content.

T. T. PORTS.

**Determination of water in paper, pulp, and half-stuff.** M. KNORFF (Woch. Papierfabr., 1930, 61, 416—418; Chem. Zentr., 1930, i, 3506).—A discussion of the methods available.

A. A. ELDRIDGE.

**Moisture-proof determination of waxed papers.** C. A. THOMAS and H. J. REBOULET (Ind. Eng. Chem. [Anal.], 1930, 2, 390—391).—An apparatus is described for rapid determination (2 hrs.) of water-vapour transmission through waxed papers. Pinholes and imperfections in the paper before waxing cause large increases in transmission, whilst normal handling and creasing have not much effect.

J. LEWKOWITSCH.

**Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI. **Determination of crude fibre in cacao.** KÜRSCHNER and HANAK.—See XIX.

## PATENTS.

**Purified [vegetable] fibre.** R. A. GORTNER and W. F. HOFFMAN, Assrs. to NORTHWEST PAPER CO. (U.S.P. 1,757,768, 6.5.30. Appl., 30.1.26).—Such fibres, particularly wood fibres in chip or pulp form, are freed from mineral impurities (other than traces of silica) and colloidal organic colouring matter by electrolysis a 5—10% aqueous suspension of the material, using 220 volts *D.C.* Graphite electrodes are preferably used and are separated from the pulp by, e.g., fabric diaphragms. Hot or cold water may be used, optionally in conjunction with suitable reagents, and provision is made for continuously renewing the electrolyte in the electrode compartments. The purified material contains less than 0.05% of ash and does not discolour when heated at 60—70° for 100 hrs. Oxycellulose and hemicelluloses are not removed by this treatment.

D. J. NORMAN.

**Compressed [fibrous] products.** HALIZITE CORP., Asscs. of A. HAWERLANDER (B.P. 336,754, 20.9.29. U.S., 13.3.29).—Sawdust, wood shavings, etc., after being moistened with a volatile liquid (petrol), are treated with sufficient binding material (phenol-formaldehyde condensation product) to cover the surface of the fibrous particles and are finally subjected to heat and pressure.

F. R. ENNOS.

**Preparation of mercerised wood-fibre tissue.** M. O. SCHUR and R. H. RASCH, Assrs. to BROWN CO. (U.S.P. 1,749,003, 4.3.30. Appl., 5.1.27).—High- $\alpha$ -cellulose wood-fibre tissue is treated with caustic soda solution of mercerising concentration containing an oxidising

agent (hypochlorite, permanganate), washed with hot water, treated with an acid and reducing agent (sulphurous acid) to neutralise the last traces of alkali and oxidant, and finally washed free from the reaction products; the high-grade cellulose produced is suitable for nitration to yield artificial silk, films, lacquers, etc.

F. R. ENNOS.

**Colloided cellulose and indurated fibre.** F. G. HAHN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,757,755, 6.5.30. Appl., 8.2.28).—Cellulose in woven, non-woven, or sheet form is gelatinised without dissolution by treating it with an aqueous solution of a fixed caustic alkali containing 2–11 mols. of alkali per 100 mols. of water at a temperature between the f.p. of the reagent and about 20° above it. When the required degree of swelling is attained the material is compressed to the desired shape and the swelling agent removed by, e.g., washing with water.

D. J. NORMAN.

**Production of cellulose fatty acid esters.** K. WERNER (B.P. 336,349, 15.8.29).—Water is removed from air-dried cellulose before esterification by intimately mixing in a ball or roller mill at temperatures up to 50° with sufficient fatty acid and fatty acid anhydride to complete the esterification, but with an amount of catalyst insufficient to promote the reaction (0.02–0.05% of sulphuric acid or 0.05% of sulphuryl chloride calc. on the wt. of cellulose); esterification is subsequently carried out by gradual addition of a weak catalyst (sulphuryl chloride, phosphorus oxychloride), the temperature being allowed to rise to 60–70°.

F. R. ENNOS.

**Manufacture of (A) highly esterified acetylnitrocellulose, (B) artificial silk.** N.V. FABR. VAN CHEM. PROD., Assees. of J. N. ELGERSMA (B.P. 317,854–5, 21.8.29. Holl., 24.8.28).—After acetylating partly nitrated cellulose with acetic acid and acetic anhydride in the presence of sulphuric acid or a derivative thereof as catalyst, the excess of anhydride is removed by addition of the equivalent amount of water or an alcohol, and the whole is kept at 30–50° until the cellulose sulphate present has been transformed into acetate. The solution is then (A) precipitated with water and the highly esterified acetylnitrocellulose dried, dissolved in a suitable solvent, and worked up into the desired form; or (B) treated with sufficient sodium acetate to render the free sulphuric acid inactive, filtered, and spun into a bath containing aqueous acetic acid.

F. R. ENNOS.

**Cellulose ether compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,309, 15.4.29. U.S., 24.5.28).—A cellulose ether is dissolved in a mixture of an alcohol which is a solvent for the ether, e.g., butyl alcohol, with an aliphatic hydrocarbon or hydrocarbon mixture which is a non-solvent or a poor solvent therefor, e.g., gasoline, the two liquids being in such proportions that the mixture has a greater solvent power than the combined solvent power of the separate liquids.

F. R. ENNOS.

**Solvent for cellulose nitrate.** J. P. TRICKEY, Assr. to QUAKER OATS Co. (U.S.P. 1,756,228, 29.4.30. Appl., 3.8.28).—The use of esters of tetrahydrofurfuryl alcohol is claimed, these being water-white, stable in colour, free

from objectionable odour, compatible with gums and resins, and miscible with other solvents used in nitrocellulose lacquers.

D. J. NORMAN.

**Reducing the viscosity of nitrocellulose.** (A) G. H. TOZIER, and (B) P. C. SEEL, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,757,491 and 1,757,481, 6.5.30. Appl., 6.11.26).—(A) Nitrocellulose is digested for at least 8 hrs. with water under pressure in an acid-resisting autoclave at above 110° until the desired viscosity is reached. The nitrocellulose should remain completely immersed during the whole operation, and the steam used for heating is injected below the water level. The water thus introduced, together with any wash water which may be added to the system to facilitate the removal of decomposition products, flows away through a heat exchanger and a steam trap. An external circulating pump is provided. (B) The use of an acid-resisting autoclave is obviated by neutralising the acid as it is formed by the controlled introduction of alkali, e.g., 5% caustic soda solution, into the circulatory system.

D. J. NORMAN.

**Apparatus for preparation of cellulose xanthate.** M. DASSONVILLE (B.P. 337,149, 26.9.29. Fr., 1.7.29).—A form of jacketed rotating digester is described, which has a smooth interior and can be opened by separating into two halves.

B. M. VENABLES.

**Manufacture and working up of artificial materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,250, 4.6.29).—A true sulphonic acid or sulphinate of a derivative of an aliphatic or cycloaliphatic hydrocarbon containing at least 8 carbon atoms (sulphonated oleic acid) is incorporated with artificial masses made from or containing cellulose or its derivatives, or with solutions or baths for their production.

F. R. ENNOS.

**Manufacture of [cellulosic] sheets, films, or the like.** CELLULOID CORP. (B.P. 315,840, 19.7.29. U.S., 19.7.28).—A solution of cellulose or of a cellulose derivative is poured on to the surface of a forwardly moving inert liquid medium, e.g., mercury or a low-melting alloy, which is heated to evaporate the solvent, and the partially-dried sheet, after being stripped off, is passed over one or more drying drums and thence to the winding device.

F. R. ENNOS.

**Manufacture of films, ribbons, and sheet-like solids.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 318,250, 26.8.29. Fr., 31.8.28).—Liquid films are formed from solutions or pseudo-solutions by surface tension between two or more solid threads or plates serving as supports, and are then coagulated; the supports, which may be of silk, wire, or of the same composition as the film, may be removed from the manufactured product or not, as desired.

F. R. ENNOS.

**Rotary dewatering sieves for cellulose and the like.** J. STRINDLUND (B.P. 336,879, 27.12.29. Nor., 11.6.29).—A cylindrical sieve which rotates while partly immersed in the pulp mixture has a number of chambers below the sieve cloth, each of which is provided with an outflow pipe which is bent backwards with respect to the direction of rotation so that the outflow end remains immersed in the water passing into the interior of the



F. R. ENNOS.

F. R. ENNOS.

D. J. NORMAN.

## PATENTS.

C. HOLLINS.

C. HOLLINS.

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C. HOLLINS.

F. R. ENNOS

A. J. HALL.

**Composition of matter and its employment.** [Rendering balloon fabric impermeable to inflating gas.] C. M. CARSON, ASSR. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,750,460, 11.3.30. Appl. 6.1.27).—The fabric is coated with a mixture of 1 pt. of gelatin and fibrin with at least 3 pts. of rubber latex, together with glycerin and a hygroscopic electrically conductive salt (potassium chloride) which is chemically inert to



the other ingredients; after partly drying, the film is sprayed with formaldehyde, which tans the gelatin, and is subsequently dried. F. R. ENNOS.

Dye vats or jiggers and machines employing immersion rollers. F. HEWITT (B.P. 337,894, 12.9.29).

Application of liquids to [travelling] yarns or threads. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 337,433, 30.5.29).

Machine for mercerising fabrics. O. HOFFMANN (B.P. 337,712, 10.3.30).

Clarifying liquids (B.P. 336,865).—See I. Treatment agents for textiles (B.P. 317,039). Mothproofing agents (B.P. 335,547).—See III. Treatment of cellulose derivatives (B.P. 336,217).—See V.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Loss of nitrous products owing to degradation in the lead-chamber [sulphuric acid] process. A. SANFOURCHE and L. RONDIER (Bull. Soc. chim., 1930, [iv], 47, 952—958).—Experiments were carried out by passing a gaseous mixture of oxygen, nitrogen, sulphur dioxide, and nitric oxide into a vessel containing sulphuric acid, the temperature and conditions being those which prevail in the lead chambers. The reduction of the nitrous substances (to nitrous oxide and nitrogen) passes through a maximum as the temperature rises, this maximum increasing as the acid concentration decreases and moving towards the region of high temperature as it increases. The reduction becomes more pronounced with increasing sulphur dioxide concentration, except at high temperatures, when the reduction is virtually constant. The results indicate that the reaction takes place in sulphuric acid solution. S. K. TWEEDY.

Nitric acid produced from ammonia by modified pressure system. G. FAUSER (Chem. and Met. Eng., 1930, 37, 604—608; cf. B., 1928, 601).—Experiments on the industrial scale showed that in the oxidation of ammonia, the conversion is greater at atmospheric than at higher pressures, and that the rate of oxidation of nitric oxide increases as the pressure is raised. In a plant in which the former reaction is carried out at atmospheric pressure, the absorption apparatus, working under pressure, consists of a series of horizontal cylindrical vessels, cooled by water on the outside, in which the gases bubble through nitric acid or water on the countercurrent principle. Data in connexion with the design of the absorption system are given.

D. K. MOORE.

Ammonium sulphate from gypsum. B. WAESER (Brennstoff-Chem., 1930, 11, 396—398, 418—420).—Recent developments in the production of ammonium sulphate are briefly discussed, reference being made particularly to Baud's process (B., 1928, 12), to the Bayer cement process (B., 1926, 271), and to other developments in the manufacture of sulphuric acid and cement from gypsum (cf. Diehl, B., 1921, 506 A; Neumann, B., 1927, 106). The cost of converting ammonia into ammonium sulphate is estimated at 24 pf. per kg. of nitrogen, by direct combination with sulphuric acid, or 13.3 pf./kg., by the gypsum process. These costs may

actually be higher than the cost of production of the ammonia itself by synthetic methods.

A. B. MANNING.

Extraction of magnesia from dolomite. L. CAMBI (Giorn. Chim. Ind. Appl., 1930, 12, 438—441).—The technical possibility of Twynam's process (B., 1929, 851) is considered. Using a dolomite containing MgO 20.78, CaO 31.2, ( $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) 0.43,  $\text{CO}_2$  46.82, insoluble matter 0.19%, and sieving and levigating the milk prepared from the calcined dolomite in order to separate the heavy granules containing undecomposed dolomite and a large part of the impurities, it was found possible to prepare magnesia which, after washing and calcining, had the composition (%): MgO 98.91, CaO 0.39, ( $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) traces. The economic side of the process is not discussed. T. H. POPE.

Preparation of cupric acetate. Z. A. IOFA and L. L. KLYACHKO-GURVICH (J. Appl. Chem., Russia, 1930, 3, 361—365).—Cupric oxide is agitated for 1—2 hrs. with concentrated acetic acid and the acetate (containing 1—2% CuO) is stirred at 80° with the solution obtained from a previous crystallisation; after filtration the solution is diluted with solution remaining from the first operation and cooled to crystallise.

CHEMICAL ABSTRACTS.

Dust control in phosphate rock grinding. W. H. GABELER (Ind. Eng. Chem., 1930, 22, 1077—1080).—The Davison Chemical Co.'s plant at Baltimore for grinding phosphate rock consists of 12 Raymond mills, with an average capacity of 4 tons per hr. each. To overcome the dust nuisance, Draco two-compartment filters with cotton-napped bags were installed, each designed to handle the air from two mills. The air volume passing through one unit was 900 cub. ft. per min. from each vent, i.e., 1800 cub. ft. per min. The static pressure at the intake to the filter was 1 in., and the suction at the exhaust 2 in. The average load was 2 h.p. A recovery of 32 lb. of pulverised rock per hr. was obtained from two mills, equivalent to 1 ton per day and representing 0.2% of the capacity. This amounted to a dust concentration, assuming 99% efficiency, of 2.05 grains per cub. ft. Comparative screen tests on material from the mills and the filter showed, respectively, 91.9 and 99.9% through 100-mesh, 81.9 and 99.5% through 150-mesh, 67.0 and 96.5% through 200-mesh, and 51.3 and 83.9% through 300-mesh. The power consumption for the whole system does not exceed 15 h.p.-hrs., and the only labour costs are for inspection and lubrication; maintenance charges over 6 months were negligible. W. J. WRIGHT.

Change of the water-soluble phosphoric acid content of superphosphate during storage in bulk. III. Relation of the variety of raw phosphate and of the conditions of its manufacture to the decline of the water-soluble phosphoric acid content. T. SHŌJI, E. SUZUKI, and E. NANAI (J. Soc. Chem. Ind., Japan, 1930, 33, 418—419 B; cf. B., 1930, 903).—The change of the water-soluble phosphate content during storage at the ordinary temperature and at 60° of superphosphates made from phosphate rock from various districts has been studied: only in a few cases is the water-soluble content preserved by storage at

the higher temperature. Variations of procedure during the manufacture of superphosphate, such as alteration of the quantity and concentration of the sulphuric acid used and of the degree of fineness of the mineral, are without influence on the diminution of the water-soluble content.

H. F. GILLBE.

**Reversion of the citrate-soluble phosphate in Rhenania phosphate.** K. SCHARER (Landw. Versuchs-Stat., 1930, 111, 1—10).—Samples of Rhenania phosphate, after storage in corked bottles for a year, showed slight decreases in the proportion of citrate-soluble phosphate. These were, however, within the limits of error officially allowed in this test. A. G. POLLARD.

**Soluble sulphurs [polysulphide sprays].** R. P. TUCKER (Cal. Dep. Agric., Mon. Bull., 1930, 19, 422—429).—The atomic structure of alkali polysulphides is examined.

A. G. POLLARD.

**Determination of small quantities of oxygen in gases.** H. R. AMBLER (Analyst, 1930, 55, 677—680).—The combustion of hydrogen in excess of oxygen in the presence of platinum wire at red heat is examined with a view to the determination of oxygen in excess of hydrogen. Ammonia was not formed in the presence of nitrogen and hydrogen in sufficient quantities to introduce error, and the method was found suitable for all proportions of oxygen up to the explosion limit (about 4%) and for higher proportions if the pipette is strong enough. The accuracy of the method may be reduced to 0.1% of the total gas if oxides of carbon are present.

D. G. HEWER.

**A Western nitrogen-fixation plant.** G. N. WESTBY (Ind. Eng. Chem., 1930, 22, 1099—1103).—A description is given of the plant and arc process used by the American Nitrogen Products Co., at La Grande, Washington, prior to the destruction of the factory by fire. The product contained 96.5—99.0%  $\text{NaNO}_2$ , according to the market requirements. With a production of 200 tons per month, the cost of the nitrite was 4.55 cents per lb.

W. J. WRIGHT.

**"Dry-ice."** D. H. KILLEFFER (Ind. Eng. Chem., 1930, 22, 1087—1091).—The development of the solid carbon dioxide ("Dry-ice") industry in America is recorded. For preserving perishable goods, Dry-ice shows a marked economy compared with ice, the available refrigeration per lb. being about double. Refrigerators can maintain a temperature of 5° for ten days without recharging. For cooling large quantities of materials, as distinct from maintaining a low temperature, Dry-ice is uneconomical. Suitable methods of storage and distribution of large quantities of the material during the winter or summer are described.

W. J. WRIGHT.

**Carbonised molasses waste.** KILP. **Determination of lead tetraethyl in gasoline.** CATLIN and STARRETT.—See II. **Magnesite.** DWORZAK.—See VIII. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI. **Potash shale as fertiliser.** HEATH. **Magnesium salts as fertilisers.** DIX and BISCHOF.—See XVI. **Liquor aluminii acetico-tarici and liquor aluminii acetici.** HOLDERMANN.—See XX. **Protection against toxics.** TURNER.—See XXIII.

## PATENTS.

**Manufacture of nitric acid.** M. BATTEGAY, Asst. to CALCO CHEM. Co., INC. (U.S.P. 1,756,532, 29.4.30. Appl., 3.2.27. Fr., 19.10.26).—Nitrosylsulphuric acid is circulated downwards through narrow towers filled with Raschig rings and heated electrically at the lower end at 60—110°, while a current of substantially pure oxygen is passed upwards through the towers. The addition of small quantities of ceric sulphate, ammonium metavanadate, or chromic acid to the acid liquid materially accelerates the rate of oxidation of the nitrogen peroxide therein.

A. R. POWELL.

**Manufacture of nitric acid.** W. W. TRIGGS. From E. I. Du Pont de Nemours & Co. (B.P. 336,233, 27.3.29).—In the catalytic oxidation of ammonia, the gases leaving the oxidiser at 700—900° pass first to a cooler in which their temperature is reduced to about 250°, and thence to a condenser in which they are rapidly cooled to about 20°, water vapour being thereby condensed and oxidation of nitric oxide to nitrogen peroxide begun. The condensate flows back in countercurrent to the ascending hot gases, and by careful regulation a condensate containing the minimum amount of nitric acid may be drawn off and conducted to the absorbers. The gases from the condenser pass to an oxidiser in which they are completely converted into nitrogen peroxide, oxidation being accelerated under the pressure maintained throughout the process, and thence reach the absorbers where they are bubbled through a countercurrent of successively more dilute acid. Nitric acid of 69% concentration may be obtained. [Stat. ref.]

W. J. WRIGHT.

**Manufacture of phosphoric acid and hydrogen.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,109, 24.8.29).—Phosphorus or phosphorus acids of a lower stage of oxidation than phosphoric acid are heated at 250—500° under pressure with anhydrous phosphoric acid.

W. J. WRIGHT.

**Synthetic production of ammonia.** R. S. RICHARDSON, Asst. to CHEM. ENG. CORP. (U.S.P. 1,750,649, 18.3.30. Appl., 12.3.27).—After the gases leave the converter, part of the ammonia is condensed and removed before fresh gases are introduced, being thus obtained in a pure state, and after admission of the gases the remainder of the ammonia is condensed, thereby absorbing any impurities from the gases or the system.

W. J. WRIGHT.

**Wire-gauze catalysts [for the oxidation of ammonia].** C. TONIOLO, and "AZOGENO" SOC. ANON. PER LA FABR. DELL'AMMONIACA SINTETICA E PROD. DERIV. (B.P. 337,680, 6.1.30. It., 25.7.29).—The catalysts comprise two or more metal (e.g., platinum) wire gauzes of different mesh laid one above the other so that the wires of the different gauzes lie at different angles to one another.

A. R. POWELL.

**Manufacture of anhydrous sodium sulphide.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 336,251, 10.6. and 6.8.29).—Sodium sulphate is reduced by hydrogen or other gaseous reducing agent at 500—600°, the mass being maintained in the solid state during the reaction. Reduction is accelerated if a catalyst is employed, such catalyst consisting of sodium sulphide

or caustic soda, or metals or mixtures containing them, *e.g.*, the crude melt from the sodium sulphide manufacture.

W. J. WRIGHT.

**Manufacture of ammonium carbamate and the carbamates and carbonates of the alkali metals and of magnesium.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,206, 1.7.29).—The reaction of ammonium bicarbonate with liquid ammonia is effected with 2–3 times the amount of ammonia theoretically required, so that conversion takes place at ordinary temperatures. The process may also be carried out with gaseous ammonia, which, under the pressure employed, forms a solution with part of the salt.

W. J. WRIGHT.

**Manufacture of ammonium sulphate.** F. G. LILJENROTH, ASSR. to KUNSTDÜNGER-PATENTVERWERTUNGS A.-G. (U.S.P. 1,758,449, 13.5.30. Appl., 24.12.28).—A mixture of finely-ground calcium cyanamide and calcium sulphate is heated under pressure with water and carbon dioxide to produce ammonium sulphate and calcium carbonate. The latter is collected and dissolved in nitric acid to obtain calcium nitrate and carbon dioxide under pressure for use in the first stage of the process.

A. R. POWELL.

**Inhibiting the corrosive effect of brines towards iron and steel.** O. V. MARTIN, ASSR. to MARTIN-COLVIN Co. (U.S.P. 1,750,761, 18.3.30. Appl., 26.10.27).—The brine is treated with milk of lime sufficient to liberate about 0.05% of magnesium hydroxide from the magnesium chloride present in the brine. The  $p_H$  of the solution is thus reduced to a value at which the dissolved salts cease to promote the corrosion of the metal.

A. R. POWELL.

**Production of alimentary salt.** P. WOOG (B.P. 319,203 and Addn. B.P. 336,279, [A] 5.7.29, [B] 8.7.29. Fr., [A] 17.9.28, [B] 18.2.29).—(A) An alimentary saline preparation, which contains all the salts in sea water without alteration, is obtained by concentrating the sea water until salts separate in a crystalline form, and then evaporating it at not above 105°, so that it does not become superheated; an agglomeration of crystals, which hinders evaporation, is prevented by constant stirring. (B) To the mixture obtained as described an insoluble magnesium salt or finely-divided magnesium metal may be added. The magnesium salt may consist of partly hydrated magnesium carbonate, which may be prepared by adding an alkali carbonate to the paste.

W. J. WRIGHT.

**Making magnesium chloride from chlorine and a magnesium base.** T. GRISWOLD, JUN., and R. M. HUNTER, ASSRS. to DOW CHEM. Co. (U.S.P. 1,756,247, 29.4.30. Appl., 2.7.28).—Magnesium oxide, hydroxide, or carbonate is treated with chlorine and sulphur dioxide in the presence of water to obtain a solution of magnesium chloride and sulphate from which the latter is separated by evaporation and crystallisation. The magnesium chloride mother-liquor is evaporated to obtain the dry salt for the production of metallic magnesium by fused electrolysis, the chlorine liberated in this process being used in the preparation of fresh quantities of the chloride.

A. R. POWELL.

**Refining and purifying barium sulphate.** H. V. FARR, ASSR. to MALLINCKRODT CHEM. WORKS (U.S.P. 1,752,244, 25.3.30. Appl., 4.3.26).—The powdered sulphate is heated at 130° with a concentrated solution of a salt of another alkaline-earth metal capable of forming a complex barium salt. The product is hydrolysed with water, and the extremely fine barium sulphate produced is purified by washing. Various references to such double salts are given.

W. J. WRIGHT.

**Production of naphthenates of heavy and alkaline-earth metals.** I. G. FARBENIND. A.-G. (B.P. 335,863, 16.1.30. Ger., 22.2.29).—Naphthenic acid is boiled with aqueous sodium hydroxide, a solution of a salt of a heavy metal or alkaline-earth metal is added directly to the boiling liquid, and the precipitate, washed by hot decantation, is maintained molten at about 130° until dry; the whole operation is conducted in the same vessel. The preparation of lead manganese and cobalt zinc naphthenates is described.

C. HOLLINS.

**Manufacture of [crystalline] zinc sulphide.** E. C. GASKILL (U.S.P. 1,758,741, 13.5.30. Appl., 17.12.26).—Zinc sulphide ores mixed with carbonaceous material are heated by electrical resistance to 1000° in a vacuum or in a current of nitrogen or steam free from oxygen, whereby pure zinc sulphide volatilises and condenses in settling chambers and bag plant as fine crystals which can be ground to yield a dense white pigment.

A. R. POWELL.

**Production of basic lead chromate.** A. STEWART, ASSR. to U.S. CHEM. PRODUCTS Co., INC. (U.S.P. 1,751,295, 18.3.30. Appl., 9.12.27).—An aqueous suspension of lead sulphate is treated at 70° with a soluble alkali dichromate and sufficient soluble alkali hydroxide to give a product containing about 55%  $PbCrO_4$  and 45%  $PbO$ . The basic lead chromate, after settling, is washed, filtered, and dried at 120°.

W. J. WRIGHT.

**Manufacture of zeolites.** PERMUTIT Co. (B.P. 319,746, 26.9.29. U.S., 27.9.28).—Solutions of aluminium sulphate and sodium silicate are mixed, and to this mixture is added, before gelation, a solution of aluminium sulphate. The proportions of the reagents are such that (a) the total alkali content of the silicate and aluminate solutions exceeds that required to form neutral sulphate with the sulphuric acid of the aluminium sulphate, and (b) the ratio  $Na_2O : Al_2O_3$  in the final mixture is 1:1.

W. J. WRIGHT.

**Obtaining pure glauconite [for water-softening].** H. KRIEGSHEIM and W. VAUGHAN, ASSRS. to PERMUTIT Co. (U.S.P. 1,757,374, 6.5.30. Appl., 4.2.28).—Crude greensand is thoroughly washed with water to remove clay and fine particles, carefully graded into sizes, and vigorously agitated with hot dilute sodium hydroxide solution to remove humus. The purified sand is then passed through a hot solution of sodium silicate ( $d$  1.02) to restore silica removed by the previous treatment, agitated with very dilute aluminium sulphate solution to remove free alkali, and finally scrubbed thoroughly with warm water.

A. R. POWELL.

**Production of elemental sulphur.** R. C. BENNER and A. P. THOMPSON, ASSRS. to GEN. CHEM. Co. (U.S.P.

1,751,066—8, 18.3.30. Appl., [A—C] 8.9.26).—(A) A mixture of sulphide ore and carbonaceous material is subjected to the action of an oxidising gas in counter-current, and the gaseous products at 350° are passed over a catalyst, such as bauxite, to complete the reduction to elemental sulphur. (B) Sulphide ore is treated with a carbonaceous material comprising a considerable amount of hydrocarbons, and the gaseous products are treated with an oxidising gas and passed over a catalyst as in (A). (C) In the processes described in (A, B), reduction is carried out at temperatures above 1200°, so that the slag may be removed in a fused condition.

W. J. WRIGHT.

**Apparatus for purifying sulphur and obtaining carbon disulphide and sulphur oxides.** G. JAKOVA-MERTURI (B.P. 310,972, 3.5.29. Fr., 4.5.28).—The lower portion of a furnace is divided vertically into three compartments in each of which are set two superimposed cast-iron retorts, all of which can be put into communication by means of pipes and valves; the centre ones also communicate with an upper chamber. Temperatures of 800—900°, 450—500°, and 130°, respectively, may be attained in the three compartments. In the upper portion of the furnace, in addition to the chamber referred to, is a second one in which are placed horizontally three water-cooled, sheet-steel cylinders, open at one end, and communicating by a pipe with a cooled receiver. In manufacturing carbon disulphide, two retorts in an end compartment are loaded with brown coal and the centre ones with sulphur, the volatilised sulphur passing into the former pair and combining with the coal. For the production of roll sulphur the first two retorts are cut off and sulphur vapour from the centre pair is condensed in the last two, from which the sulphur is run off. To obtain flowers of sulphur, only the centre retorts are utilised, the sulphur vapour passing through the upper chamber into the cooled cylinders, and any oxides of sulphur being condensed in the receiver. Sulphur ore may be used instead of sulphur, arrangements being then made for dehydrating and the removal of tar.

W. J. WRIGHT.

**Separation and recovery of gases [sulphur dioxide from smelter gases].** R. L. HASCHE, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,758,398, 13.5.30. Appl., 17.8.27).—The gases, freed from dust and fume, are passed through silica gel or active charcoal at 25°, whereby the sulphur dioxide is absorbed. The adsorbed gas is liberated by passing steam through the adsorbent and the greater part of the steam is condensed at as high a temperature as possible. The gases then pass through a water-cooled condenser to remove most of the remaining water and are finally dried by means of sulphuric acid before passing to the refrigerating apparatus for the production of liquid sulphur dioxide.

A. R. POWELL.

**Production of phosphorus oxychloride.** J. S. DUNN, F. BRIERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 337,123, 6.9.29).—Natural calcium phosphates are pretreated at 300° with phosgene to decompose carbonate, and, after removal of the calcium chloride by washing, the product is dried and treated with phosgene.

W. J. WRIGHT.

**Purification of triaryl phosphates.** W. GIBSON and C. R. HENSHAW, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,781,225, 11.11.30. Appl., 23.10.29. U.K., 24.10.28).—See B.P. 322,057; B., 1930, 95.

**Vessels proof against chemicals etc.** (B.P. 316,134).—See I. **Smelter-furnace by-products** (U.S.P. 1,755,845). **Utilisation of dolomite etc.** (U.S.P. 1,749,210—1).—See X. **Complex iron compounds** (B.P. 335,965).—See XX. **Breathing cart-ridges** (B.P. 337,170).—See XXIII.

## VIII.—GLASS; CERAMICS.

**Ultra-violet window glazing.** H. E. BECKETT (Dept. Sci. Ind. Res., Building Res. Bull. No. 8, 1930, 13 pp.).—The literature dealing with the therapeutic value of ultra-violet radiation and the behaviour of ultra-violet light-transmitting glasses is reviewed, and the amount of radiation received by a window facing north is examined.

M. PARKIN.

**Russian and Slovakian magnesite.** E. DWORZAK (Feuerfest, 1930, 6, 145—150).—Chemical analyses of a number of magnesite bricks of Austrian, Slovakian, and Russian origin are given, and a comparison is made of the colour, composition, and physical properties both of the bricks and of the granular, calcined material. Russian magnesite bricks have a higher magnesia content (> 92% MgO) than the Slovakian, with consequent greater refractoriness and refractoriness under load. A lower flux content makes them less vitreous and more porous, and, owing to the lower firing temperature, the sp. gr. is somewhat lower than that of the Slovakian bricks. In spite of the higher porosity, the Russian bricks are equally resistant to slag attack. The chemical composition of the Russian calcined magnesite is the same for all granulations, viz., about 91% MgO and 3% Fe<sub>2</sub>O<sub>3</sub>. A grain size of 0—5 mm. gives the most satisfactory degree of vitrification both with the Russian and Slovakian material, and if larger grades are used a certain proportion of fines must be added to act as a bond. In steel works up to 15% of open-hearth furnace slag, with a grain size of about 5 mm., may be added to the Russian magnesite to assist vitrification.

F. SALT.

**Effect on boiler-furnace walls of gases from bagasse and coffee-pod fuels.** F. W. FREISE (Feuerfest, 1930, 6, 150—152).—A study was made of the injurious effect of gases rich in potassium on the walls of furnaces fired with sugar-cane residues or with coffee-pod waste. Most of the potassium was present as carbonate, which readily decomposes. A special test furnace was constructed, in which different types of refractories were tested. Bricks containing less than 50% Al<sub>2</sub>O<sub>3</sub> lasted for short periods only. Furnaces exposed to vigorous attack should be built of bricks with not less than 60% Al<sub>2</sub>O<sub>3</sub>. Zirconia bricks conferred no further advantages, but a paste made up with ground zirconia and water-glass gave good results either as a mortar or as a coating for those parts of the walls directly exposed to the gases.

F. SALT.

**Friction of dry solids in vacuo.**—SHAW and LEAVEY.—See I. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI.

## PATENTS.

**Tunnel kilns.** V. LASTOVICKA (B.P. 336,171, 23.12.29).—A tunnel kiln for firing bricks has a longitudinal recess extending along one side from the chimney, near the entrance end of the tunnel, to a point near the middle, opposite which is the firebox. The kiln is surmounted by a drying tunnel, vents being provided in the kiln roof. F. SALT.

**Manufacture of enamel ware.** W. E. DOUGHERTY (B.P. 336,797, 17.10.29).—Titania is a suitable opacifier for glasses free from chlorine, or for glasses containing chlorine if a suitable salt of the calcium group is present. If traces of iron are present, relatively small amounts of tin oxide or zirconia, or both, added to the titania, will remove the yellowish cast in the enamel. Titanium may be used in this way as an opacifier also in the ground coat. F. SALT.

**Semi-porcelain bodies for manufacture of pottery.** J. E. TAMS (B.P. 336,172, 27.12.29).—A semi-porcelain body, either for casting or jollying, contains asbestos powder, ball clay, ground stone, and/or ground felspar, roughly in the proportions of 50, 40, 10, and 10 pts., respectively. F. SALT.

**Manufacture of ceramic material.** A.C. SPARK PLUG Co. (B.P. 315,196, 8.7.29. U.S., 7.7.28).—An aluminium silicate is fused in the presence of fluxes, and the fused mass is subjected to sudden cooling in a stream of water, if necessary under pressure. Crystal growth is thus arrested, and porcelain bodies made with this fused product as the non-plastic ingredient have greater mechanical strength owing to continued and additional crystalline growth during the firing of the porcelain. The process is particularly valuable in the case of sillimanite, mullite, and andalusite. F. SALT.

**Methods and apparatus for feeding molten glass.** L. MELLERSH-JACKSON. From BALL BROTHERS Co. (B.P. 337,988—9, 12.11.29).

**Feeding the glass batch to a glass furnace.** J. E. POLLAK. From HAZEL-ATLAS GLASS Co. (B.P. 337,950, 24.10.29).

**Heating apparatus for making safety glass etc.** DU PONT VISCOLOID Co. (B.P. 337,547, 3.10.29. U.S., 27.10.28).

## IX.—BUILDING MATERIALS.

**The rotary kiln in cement manufacture.** III—VII. W. GILBERT (Cement, 1930, 3, 561—566, 699—704, 946—953, 1196—1206, 1472—1484).—The description and results of a 6-day process test on a typical wet-process rotary kiln are given. The kiln was 8.5 ft. in diam. and 202 ft. long, with an inclination of 1 in 25. During the test 263.8 tons of coal (dry) produced 928.9 tons of clinker. The total heat loss from the shell of the kiln was calculated to be equivalent to approx. 327 lb. of coal per hr., and from the cooler 67 lb. per hr., the figures representing conditions on a quiet day, and with a partly roofed kiln. By substituting a double layer of firebrick, 9 in. in total thickness, for the usual 6-in. lining, 76.7% of this loss might be saved, though the increased first cost and maintenance must be offset. The test indicated 29.6% of excess air in

the kiln and 74% efficiency of the coal as fired. The latter figure might be improved by reducing within limits the water content of the slurry by cutting down the excess air, or by otherwise reducing the temperature of the exit gases.

C. A. KING.

**Iron and steel used in cement manufacture.** ANON. (Cement, 1930, 3, 1305—1315).—The heavy wear on the mechanical parts of cement plant has suggested a description of the characteristics and mechanical properties of the different forms of iron and steel as applied to operating conditions. Although the production of wrought iron has decreased considerably by reason of its supersession by mild steel, the superior resistance of the former to shock, heat, and oxidising conditions is noted. C. A. KING.

**Accelerated soundness test [for concrete aggregates].** H. F. KRIEGER (Civil Eng., 1930, 1, 120—122).—Considerable variation in the results of the sodium sulphate immersion test will be obtained unless the temperature is controlled closely, due to the effect of temperature of saturation of sodium sulphate; e.g., between 20° and 30° the concentration of the solution changes from 20% to above 41% Na<sub>2</sub>SO<sub>4</sub>. For sewage-disposal media at least 20 pieces should be taken; for concrete aggregates at least 50 pieces 1—2 in. in diam., and for finer materials the sample should consist of 1 kg. carefully screened. A solution of 400 g. of sodium sulphate per litre is prepared at 30°, and set aside for 12 hrs.; at all times excess crystals should be present. The sample, dried at 105° and cooled, is immersed in the solution for 19 hrs. at 30°, examined, dried, and the loose fragments are separated. The test should be run for 5 cycles on concrete aggregates, and for 20 cycles on material for sewage-filtering purposes. An individual specimen breaking into three or more pieces or losing more than 20% of its weight by chipping is considered to have failed, and a sample having 20% or more of its constituent pieces failing is unsound; 15—20% indicates "doubtful soundness" and necessitates a second trial. C. A. KING.

**Sinkage [of pulp wood].** III. Changes in the water-gas system in logs during seasoning and flotation. G. W. SCARTH and R. D. GIBBS (Canad. J. Res., 1930, 3, 80—93; cf. Gibbs, A., 1930, 1322).—Observations on the production of gas in logs by fermentation during seasoning and flotation are recorded.

C. W. GIBBY.

**Sinkage studies.** IV. Mechanism of absorption of water by wood blocks. G. W. SCARTH (Canad. J. Res., 1930, 3, 107—114; cf. A., 1930, 1322).—Studies of the rate of penetration of water into white pine-wood blocks show that the limiting factor in the sinking of logs is the rate of escape of air by dissolution and diffusion.

R. K. CALLOW.

**Evaluation of sieve analyses [of cement] and Abrams' modulus of fineness.** A. HUMMEL (Zement, 1930, 19, 355—364; Chem. Zentr., 1930, i, 3478).

**Coal tar.** CHADDER and SPIERS.—See II. Ammonium sulphate from gypsum. WAESER.—See VII.

## PATENT.

**Cement for electric lamp caps** (B.P. 316,866).—See XI.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Metallography of some ancient Egyptian implements.** (Sir) H. C. H. CARPENTER and J. M. ROBERTSON (J. Iron Steel Inst., 1930, 121, 417—454).—Photomicrographs of various ancient iron implements show that the Egyptians were familiar with carburising and water-quenching; the latter process may have been known in 1200 B.C., but tempering was probably not discovered until considerably later. H. F. GILLBE.

**Agglomeration of blast-furnace dust and treatment of iron carbonate ores by the Greenawalt process.** R. CORDONNIER (Rev. Mét., 1930, 27, 467—478).—The material to be treated is mixed with 2—8% of powdered fuel and 5—20% of water. The mixture is fed by a charging carriage on to a mechanical hearth. Combustion is initiated by ignition with blast-furnace gas (or mineral oil) and continues under the action of a fan. The agglomerate resembles a very porous coke in texture, is highly permeable to gases, and may be smelted with great economy. Volatile impurities are removed and the percentage of iron is increased. In treating carbonates the charge is covered with a mixture of calcined ore and powdered fuel, to prevent uneven evolution of gas. E. H. BUCKNALL.

**Influence of liquid as against solid pig-iron additions on the operation of the open-hearth furnace.** F. WEISGERBER (Stahl u. Eisen, 1930, 50, 1489—1494).—Using 20—30% of pig iron in the open-hearth steel process, the addition of the pig iron in the molten state increases the capacity of the furnace by 10%, reduces the quantity of pig iron required for efficient operation by 10%, reduces the fuel consumption by 15%, and slightly increases the consumption of dolomite. The resulting economies amount to about 2 RM. per ton of steel. A. R. POWELL.

**Influence of melting conditions on the micro-structure and mechanical strengths of grey cast irons containing various amounts of carbon and silicon.** A. L. NORBURY and E. MORGAN (J. Iron Steel Inst., 1930, 121, 367—392).—The influence of melting conditions and superheating, of addition of steel, graphite, silicides, and oxide, and of streams of hydrogen, nitrogen, and carbon dioxide on molten mixtures of Swedish iron, Armco iron, and ferrosilicon has been studied. The refining influence of superheating on the graphitic structure has been confirmed, and addition of small quantities of steel has a similar effect; when, however, 40% and 10% ferrosilicons, 60% carbon silicide, and 5% nickel silicide are added to melts of whiteheart malleable iron, grey test bars of good mechanical properties are produced. Nickel silicide and 10% ferrosilicon alloy readily with the iron, but 40% ferrosilicon and calcium silicide alloy more slowly. Nitrogen and hydrogen, bubbled simultaneously or separately through the molten iron, tend to improve the metal by bringing slag to the surface, but are otherwise without influence; carbon dioxide causes the appearance of considerable quantities of slag, consisting probably of silicates. Iron oxide additions cause the removal of manganese, silicon, and carbon by oxida-

tion, without causing appreciable reduction in strength of the cast metal. The strength and hardness of pearlitic irons increase as the total carbon content decreases and as the distance below the eutectic composition increases; areas of supercooled graphite with ferrite lower considerably the strength and hardness of pearlitic irons. In ferritic irons the strength and hardness increase with decreasing size of the graphite particles and with increasing distance below the eutectic, whereas increase of the silicon content lowers the strength and increases the hardness. H. F. GILLBE.

**Pressure-welding of iron.** H. ESSER (Arch. Eisenhüttenw., 1930—1, 4, 199—206; Stahl u. Eisen, 1930, 50, 1500).—Pieces of pure iron and of iron with increasing carbon content were welded by pressure *in vacuo* at various temperatures and the tensile strength of the welds was determined; the shape of the ends welded was made conical so as to ensure fracture taking place at that point. Under a welding pressure of 2 kg./mm.<sup>2</sup> the strength of the welds in pure iron increases with rise of welding temperature to a maximum at 900°, then falls sharply, and finally increases again; at 900° continuous growth of the crystals through the weld is observed, but at 910° a distinct line of demarcation is observable along the joint. The strongest welds are obtained with metal having a very fine-grained structure; the strength of welds made with single crystals varies with the orientation of the crystal surfaces in contact. Highly polished surfaces form the strongest welds at much lower temperatures than roughened surfaces, e.g., maximum strength in the first case is obtained by welding at 970°, but if the surfaces are roughened with grade 1G emery maximum strength of the weld occurs with a welding temperature of 1280°. With increasing carbon content the temperature range for obtaining strong welds by pressure becomes more restricted.

A. R. POWELL.

**Tempering of severely quenched special steels.** A. MICHEL and P. BÉNAZET (Rev. Mét., 1930, 27, 501—508).—Previous work on the tempering of austenitic steel (B., 1930, 287) is now extended to a study of the effects of adding chromium, alone or with nickel, tungsten, or cobalt, by means of the Chevenard differential dilatometer. Typical quenched structures have been obtained on cooling steel specimens 4 mm. in diam. in air from 1250° (sometimes 1150°). In chromium steels increase in the Cr : C ratio renders such structures more easily obtainable and more difficult to destroy by tempering. This result is explained on the basis of formation of Cr<sub>3</sub>C<sub>2</sub> and a ferrite rich in chromium. Steels containing 5% W in addition to chromium exhibit a part of the  $\gamma$ — $\alpha$  transformation on heating. This is an effect intermediate between the two types of behaviour previously observed (*loc. cit.*). Tempering after severe quenching produces a fine dissemination of the carbides. Such treatment may be employed prior to hardening alloy steels of commerce. E. H. BUCKNALL.

**Pearlitic rustless cast steel.** V. ZSÁK (Gießerei, 1930, 17, 339—342; Chem. Zentr., 1930, i, 3348).—Steel is rendered rustless by 12—18% Cr; Acl is shifted to about 800°, and the eutectoid is at 0.3% C. For good incorrodibility the carbon content should be



0.1—0.3%. Steel containing 0.25—0.3% C and 18% Cr is recommended for thin-walled castings; manganese (0.4%), silicon (0.5%), phosphorus and sulphur (0.03%), and nickel (0.4%) have little effect. A. A. ELDRIDGE.

#### Properties of some steels containing chromium.

A. R. PAGE and J. H. PARTRIDGE (J. Iron Steel Inst., 1930, **121**, 393—415).—The mechanical strength and toughness and the resistance to oxidation and scaling at temperatures up to 900° have been determined for various chromium steels. Increase of the chromium and silicon contents from 4.7% and 0.16% to 6% and 0.31% hardly influences the mechanical properties of the steel. Steels containing about 5.6% Cr cannot be air-hardened if more than 2% Si is present, and although silicon up to 3.5% tends to make the steel brittle without impairing its strength at the ordinary temperature, at higher temperatures the strength is considerably reduced; up to 5.8—8% Cr and up to 1.6—3.1% Si produce steel of poor strength above 700°, although of greater strength than low-chromium steels at the ordinary temperature. At 900° steels containing 1.0—1.5% Si are markedly deficient in ductility. Nickel-chromium austenitic steels are stronger and less plastic when hot than the silicon steels, but there is little advantage in increasing the nickel and chromium contents from 9% to 13%. The elongation and reduction of area under load are fairly constant for nickel-chromium steels at all temperatures, whereas with silicon steels both diminish as the temperature rises. The impact values show that all the steels are relatively brittle and that heating increases the toughness; steel containing 5.99% Cr, 0.31% Si, and 0.57% C shows a remarkably high impact value at about 400°. Chromium steels containing 1.5% Si are highly resistant to oxidation at a red heat and the scale formed is adherent. All the silicon-chromium steels examined have about the same coefficient of expansion, whilst the nickel-chromium steels have a relatively high coefficient, and in all cases the coefficient increases with rise in temperature.

H. F. GILLBE.

#### Alloys for use at high temperatures. Nickel-chromium and complex iron-nickel-chromium alloys. I. W. ROSENHAIN and C. H. M. JENKINS.

II. C. H. M. JENKINS, H. J. TAPSELL, C. R. AUSTIN, and W. P. REES (J. Iron Steel Inst., 1930, **121**, 225—236, 237—314).—Alloys and steels containing up to 60% Cr together with nickel and, in some cases, other elements have been examined with regard to their endurance under load at 800° or 650°. Under conditions of stable equilibrium a second constituent appears besides the nickel-rich solid solution in binary chromium-nickel alloys at 800° when the chromium content reaches 40%, and the mechanical strength diminishes simultaneously. Alloys containing 50—60% Cr possess poor mechanical properties; at 800° the 30% chromium alloy has the maximum endurance, but at 650° a lower chromium content is preferable. A 90/10 alloy containing a little carbon is stronger than the 80/20 alloy. Sand-casting appears to produce a stronger material than chill-casting. The majority of the nickel-chromium-iron alloys investigated consist of nickel-austenitic iron solid solution, but some are influenced by the  $\gamma$ — $\alpha$  transformation and exhibit very high values of the

mechanical properties at the ordinary temperature. Although the behaviour of these alloys is satisfactory at 650°, at 800° and above the binary nickel-chromium alloys are preferable. Deoxidation prior to casting, preferably with magnesium, is essential with the nickel-chromium alloys, and when iron is present in addition the rate of pouring and the temperature of the molten metal are also of great importance. An improvement of the mechanical properties of certain of the ternary casting alloys occurs if carbon, silicon, tungsten, titanium, or molybdenum is present. Thus an alloy containing Cr 30%, Ni 30%, Fe 33.5%, W 4%, C 1.5%, and Si 1% has at 800° a life of 50 days under a stress of 6 tons/in.<sup>2</sup> The strongest of such alloys has a high ratio between the tensile strengths at 800° and at the ordinary temperature. Additions to the ternary alloys increase the hardness and reduce the ductility at high temperatures, and rolling or forging becomes practically impossible, but when such processes were possible the resulting metal had a shorter life than the cast; the ratio of tensile strengths at 800° and the ordinary temperature is relatively low for the rolled alloys. Brinell tests and microscopical examination of various alloys after slight straining and annealing indicate that, in general, cast material is stronger at high temperatures than are the rolled alloys, provided that the temperature of the test approaches the recrystallisation temperature of the metal; at lower temperatures the wrought material is usually the stronger. Investigation of the recrystallisation processes in binary nickel-chromium alloys indicates that, after thorough rolling, only the 70/30 alloy hardens appreciably on re-annealing, whereas the cast alloys all harden markedly when annealed after slight deformation. The recrystallisation temperature of slightly deformed and annealed alloys is 50—100° below that of the cast alloy. Discussion of the difference of endurance of the cast and wrought alloys shows that the cause cannot yet be fully explained. H. F. GILLBE.

**Determination of oxygen in steel.** G. THANHEUSER and C. A. MÜLLER (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., Düsseldorf, 1929, **11**, 87—94; Chem. Zentr., 1930, i, 3581).—The silica tube must be carefully cleaned before each experiment. After outgassing the graphite crucible and its content of 30—40 g. of electrolytic iron, the blank value is determined; the sample is added after the fused mass has been cooled nearly to the f.p., and the blank value is redetermined after the experiment. The fused mass must not be used repeatedly. A. A. ELDRIDGE.

**Determination of silicon in steel and iron.** H. WOLF and R. HEILINGÖTTER (Chem.-Ztg., 1930, **54**, 878—879).—Complete separation of the silicon from steel cannot be effected by a single evaporation with hydrochloric acid or with sulphuric acid by any of the published methods. At least four evaporations are required with intermediate filtrations. Tests on a steel containing 4.12% Si showed that the results after one evaporation were uniformly 0.12% low, whatever modifications in the dissolution of the sample and evaporation of the solution were used.

A. R. POWELL.

**Electrometric titration of chromium in steel and ferrochromium.** F. SPINDECK (Chem.-Ztg., 1930,

54, 890).—The steel (0.5–3 g.) is dissolved in 16% sulphuric acid and the chromium oxidised with persulphate and silver nitrate, any permanganic acid destroyed by boiling with a little 1:1 hydrochloric acid, and the cold solution titrated electrometrically with ferrous ammonium sulphate solution after addition of manganese sulphate. Fischer's type of stirring apparatus is used together with two platinum electrodes and an Emich filter tube filled with a solution made by mixing solutions of 2.926 g. of ammonium vanadate and 4.9 g. of ferrous ammonium sulphate with 50 c.c. of 1:5 sulphuric acid and diluting to 500 c.c. A. R. POWELL.

**Etching figures in iron and steel.** V. N. SVETCHNIKOV (Rev. Mét.: 1930, 27, 512).—In an additional note (cf. B., 1930, 1113), the author discusses work by Portevin (B., 1923, 781 A). Special reference is made to effects of coring in solid solutions on the regularity of etching figures. E. H. BUCKNALL.

**Economical scaling of sheet iron.** E. DWORZAK (Przemysl Chem., 1930, 14, 361–373).—For cleaning 1 ton of sheet iron, 1 mm. thick, with sulphuric acid, 40 kg. of 15% acid are required, and 2–3% of the metal is dissolved; the addition of such preparations as naphthalenesulphonic acid or "Vogel's economiser" inhibits the reaction. In the presence of the latter reagent only 17 kg. of sulphuric acid are required, and only 0.78% of iron is dissolved. Scaling at the optimum temperature (30°) is complete in 30 min.

R. TRUSZKOWSKI.

**Chemical methods of protecting metals and alloys from corrosion.** J. COURNOT and J. BARY (Rev. Mét., 1930, 27, 479–485).—A review of known methods. E. H. BUCKNALL.

**Activation by copper sulphate in the flotation of blende.** M. MORTENSON (Tidsskr. Kjem. Berg., 1930, 10, 118–119).—The time required for the activation of blende by copper sulphate solution depends on the iron content of the mineral, being longest for blende rich in iron (marmatite). The rate of activation also increases with temperature and with the presence of acid in the liquid. The electrical conductivity of blends varies greatly with the iron content; a pure iron-free blende from Spitsbergen showed a resistance of  $6 \times 10^6$  ohms per cm.<sup>3</sup>, whilst that of marmatite from Røros containing 15.4% Fe was only  $8 \times 10^{-1}$  ohm.

H. F. HARWOOD.

**Refining of Alagirski distilled zinc.** N. P. ASEEV, V. V. DOLIVO-DOBROVOLSKI, and B. F. GRASCHTSCHENKO (Coloured Metals, 1930, pp. 40).—The most suitable temperature for the liquation of distilled zinc (Fe 0.09–0.193, Pb 1.13–2.12, Cd 0.024–0.231%) from the Alagirski works is about 450°, the period of heating depending on the degree of impurity. The minimum content of lead in the purified zinc was 0.83%, which is considerably below the limits established by Spring and Romanoff (1.5%) or Rössler and Edelmann (1.7%). Metallographic investigation of the zinc to be liquated shows that, with 0.06% Fe or more, crystals of FeZn<sub>7</sub> are developed, and that solid solutions of iron in zinc are practically absent. This result is in discord with the earlier view, that solid solutions containing up to 0.7% Fe exist, but is confirmed by the work of Ogawa and

Murakami (A., 1929, 141). Micrographs taken after liquation show a characteristic development and accumulation, both of crystals of FeZn<sub>7</sub> and of lead alloys. Repeated liquation yields no noteworthy results. Redistillation of the Alagirski zinc yields a highly pure metal containing 0.03–0.05% Pb, but only when definite conditions of temperature, pressure, amount of distillate, condensation, etc. are observed. Electrolytic refining of the zinc is not practicable, owing to the lack of cheap electrical energy. T. H. POPE.

**Influence of temperature on the toughness of aluminium alloys.** T. KOBAYASHI (Mem. Ryojun Coll. Eng., 1930, 3, 145–153).—The variations with temperature of the toughness of a number of commercially important aluminium alloys has been determined by an impact method for the interval 20–550°.

H. F. GILLBE.

**Rapid determination of tin and antimony in alloys with a high copper content.** A. M. BELOUSOV (J. Appl. Chem., Russia, 1930, 3, 437–439).—The sample is dissolved in nitric acid, the solution being treated at the b.p. with 3% ammonium nitrate solution, boiled, and kept on the water-bath for 1 hr. The mixture is centrifuged, the washings and the sediment being heated with hydrochloric acid; either tin or antimony is determined volumetrically. Low results are obtained for antimony in the absence of tin: hence addition of a known quantity may be necessary.

CHEMICAL ABSTRACTS.

**Determination of cadmium in mill and smelter products.** W. E. KECK, G. L. OLDRIGHT, and F. K. SHELTON (Univ. Utah Tech. Paper, 1930, No. 12, 15 pp.).—The sample is dissolved in a mixture of nitric acid and potassium chlorate, the residue being evaporated with hydrochloric acid. The residue from this is dissolved in dilute sulphuric acid and treated with coarse iron filings. After boiling, filtering, and washing the residue, hydrochloric acid is added to the filtrate and cadmium is precipitated by boiling with ammonium sulphide; it is redissolved and reprecipitated, finally being redissolved and reprecipitated as cadmium ammonium phosphate and weighed as the pyrophosphate.

CHEMICAL ABSTRACTS.

**Electrodeposition of chromium.** V. P. ILINSKI, N. P. LAPIN, and L. N. GOLTZ (J. Appl. Chem., Russia, 1930, 3, 309–320).—Increase in the concentration of sulphate ions above 3 g. of chromic sulphate per litre decreases the current efficiency, improves the lustre and probably the hardness of the deposited metal, and widens the permissible limits of current density.

CHEMICAL ABSTRACTS.

**Electrolytic production of antimony.** F. VOGEL (Metallbörse, 1930, 20, 537–538, 650–651; Chem. Zentr., 1930, i., 3349–3350).—Optimal conditions are specified. A solution containing sulphuric acid (100–160 g.), hydrofluoric acid (20 g.), and antimony (200 g. per litre) may be used. The anodes (95–97% Sb) may contain lead, tin, iron, nickel, zinc, copper, bismuth, and the noble metals as impurities. A. A. ELDRIDGE.

**Determination of bismuth in lead ores by internal electrolysis.** E. M. COLLIN (Analyst, 1930, 55, 680–682).—The method described previously (B., 1930, 773)

has been modified so that it may be used in the case of ores without preliminary smelting, the difference being in the pretreatment of the ore to effect its decomposition and dissolution. The ore is dissolved in concentrated hydrochloric acid, with or without the help of zinc, and, after evaporation to dryness, the residue is taken up with dilute (1:1) nitric acid, hot water is added, and the liquid is filtered. After the addition of potassium permanganate (until no longer decolorised) and hydroxylamine hydrochloride, the solution is electrolysed and the combined bismuth and copper deposit is separated as before; the bismuth is then dissolved in 1:1 nitric acid and the solution electrolysed. If less than 1 mg. Bi is present, it is determined colorimetrically. D. G. HEWER.

**Friction of dry solids *in vacuo*.** SHAW and LEAVEY.—See I. **Coke fines [for zinc smelting].** DAMM and WESEMANN.—See II. **Magnesite.** DWORZAK.—See VIII. **Iron and steel in cement manufacture.** ANON.—See IX. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI.

## PATENTS.

**Smelting of ores [in a blast furnace] and recovering by-products therefrom.** F. T. SNYDER (U.S.P. 1,755,845, 22.4.30. Appl., 8.6.25).—The ore is smelted with bituminous coal in a blast furnace under such conditions that the volatile distillation products of the coal are recovered by passing the gases through suitable condensers and scrubbers. To prevent caking in the upper zones a large part of the uncondensed gases from the scrubbers is passed back through the upper zones of the furnace so as to remove therefrom the more readily condensable distillates of the coal. A recovery of over 100 lb. of ammonium sulphate per ton of coal used is claimed, owing to the presence of lime in the charge.

A. R. POWELL.

**Manufacture of iron and steel.** H. HAGEMANN (B.P. 336,954, 18.7.29).—Pig iron is blown until the carbon is reduced to 0.02%, tapped into the ladle, and treated with an alloy of iron with 18–22% Al and 40–50% Si until completely deoxidised. Just prior to casting, manganese low in carbon is added to bring the composition of the cast metal up to 0.5% Mn, 0.02% C, 0.09% Cu, and only traces of silicon.

A. R. POWELL.

**Production of slags for use in the manufacture of wrought iron.** A. M. BYERS Co., Asscs. of A. H. BEALE, H. A. BRASSERT, and F. WILLE (B.P. 315,858, 20.6.29. U.S., 21.7.28).—Slag for use in the Aston process of making wrought iron is prepared by melting tap cinder and iron roll-scale under non-reducing conditions in a hearth furnace, the charge being so proportioned that the product contains 60–75% FeO, 5–15% Fe<sub>2</sub>O<sub>3</sub>, and 10–12% SiO<sub>2</sub>.

A. R. POWELL.

**Moulding of liquid or plastic iron, steel, and other metals and alloys difficult to mould.** P. MÜLTHAUP (B.P. 336,688, 26.7.29).—The moulding appliances consist of cast, sintered, or pressed tungsten.

A. R. POWELL.

**Manufacture of magnetic [iron-nickel] alloys.** W. S. SMITH, H. J. GARNETT, and W. F. RANDALL (B.P. 336,948, 22.6.29).—Alloys of iron with 30–50% Ni,

up to 10% Cr or Mn, and free from carbon are annealed completely, cooled, subjected to a straining operation, e.g., winding on a conductor, and reannealed at a temperature between 500° and 700° at least 50° above the magnetic transformation point. A. R. POWELL.

**Chromium or chromium-nickel steel alloys.** F. KRUPP A.-G. (B.P. 337,349, 25.7.29. Ger., 26.6.29).—Alloys containing 0.07–1% C, 18–25% Cr, 7–12% Ni, and more than 2% V and/or Ti are claimed. The amount of vanadium and/or titanium added is at least twice the carbon content, the function of these metals being to combine with the carbon and thus stabilise the austenitic structure of the steel. A. R. POWELL.

**Production of titanium steel.** W. and H. MATHEUSIUS (B.P. 337,715, 12.3.30. Ger., 16.3.29).—Molten steel after decarburisation and deoxidation is tapped in a strong stream from one ladle into another containing briquettes of a titanium thermic mixture.

A. R. POWELL.

**High-speed tool-steel alloy.** G. R. COULS (U.S.P. 1,756,282, 29.4.30. Appl., 1.9.27).—Steel containing substantially 20% W, 1% Cu, 4% Cr, 1.5–2.5% V, and 73% Fe is claimed.

A. R. POWELL.

**Electrodes or rods for welding [non-rusting steel].** J. H. ATTCHISON, H. BULL, and L. JOHNSON (B.P. 337,589, 25.10.29).—The rod is prepared by drawing down a billet consisting of a shell of chromium steel containing 20% Cr and 0.1% C surrounding a core of nickel, the weight of the core being about 10% of that of the steel. When it is desired to introduce other elements into the weld, e.g., tungsten, molybdenum, or manganese, the nickel core is provided with a sleeve of steel containing a high percentage of these elements.

A. R. POWELL.

**Protection of iron and steel. Cementation processes for ferrous metals.** A. FOLLIET and N. SAINDERICHIN (B.P. 337,562 and 337,635, [A] 10.10.29, [B] 28.11.29).—(A) Iron or steel articles are heated at 850–1050° in a mixture of fine-grained aluminium, granular carborundum, 2–5% of the chloride of chromium, nickel, or manganese, and 1–2.5% of ammonium chloride, whereby a coating of aluminium containing chromium, nickel, or manganese is produced which resists scaling up to 1500°. (B) The steel is heated in a mixture of chromite, granulated aluminium, and chromic chloride, whereby the outer layer is converted into a chromium steel. The chromate may be replaced by ilmenite, calcined pyrolusite, garnierite, or wolfram for the production of cemented layers of titanium, manganese, nickel, or tungsten steels, respectively.

A. R. POWELL.

**Inhibitor material [for use in steel pickling].** L. J. CHRISTMANN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,755,812, 22.4.30. Appl., 18.9.29).—Tetramethyldiaminodiphenyl disulphide is claimed as an inhibitor in sulphuric acid pickling baths.

A. R. POWELL.

**[Inhibitor for use in baths for the] cleaning and protection of metal surfaces.** J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,750,651, 18.3.30. Appl., 19.11.27).—An inhibitor for sulphuric acid pickling baths comprises a sulphonated arylthiazole com-

pound, *e.g.*, the aldol condensation product of aniline hydrochloride, ethylene glycol, and a sulphonated mercaptobenzthiazole. A. R. POWELL.

**Production of coated metal bodies [galvanised iron].** F. B. DEHN. From AMER. ROLLING MILL CO. (B.P. 337,399, 29.7.29). The galvanising bath contains zinc alloyed with 3–20% Al, preferably 8%, and is floated on a lead bath through which the pickled steel sheets are passed first. After passing through the galvanising bath the treated sheets are heated at a temperature just above the m.p. of the coating to produce a finely-crystallised structure in the coating.

A. R. POWELL.

**Manufacture of coated iron and steel articles.**

E. M. JONES, ASST. to PARKER RUST-PROOF CO. (U.S.P. 1,750,270, 11.3.30. Appl., 13.6.27).—The articles are pickled and boiled in a bath containing acid phosphates of iron, zinc, or manganese, together with finely-divided silica in suspension; a layer containing phosphate and silicate of iron is formed.

A. R. POWELL.

**Coated metal [iron] and its manufacture.** F. W. DE JAHN and J. G. DELY, ASSRS. to CHEM. RES. & DESIGNING CORP. (U.S.P. 1,755,686, 22.4.30. Appl., 10.10.29).—Sheet iron or steel is passed through a bath of molten lead containing 0.05–0.5% Bi, 0.05–0.2% Cu, up to 0.1% Hg, and 3–8% Sn. At the point of entry of the metal into the bath the latter is covered with a layer of fused zinc chloride and at the lowermost portion of the bath the coated metal is subjected to the forcible impingement of jet currents to dislodge adhering particles of foreign matter.

A. R. POWELL.

**Manufacture of [steel] articles hardened by nitrogenisation.** F. KRUPP A.-G. (B.P. 312,349 and 337,404, 29.4.29. Ger., 2.5.28).—Articles of steel containing (A) 0.4–0.5% C, 0.25–0.35% Si, 0.5–0.6% Mn, 1.2–1.5% Cr, 0.3% Mo, and 0.5% V, or (B) 2% C, 12% Cr, 0.8% W, and up to 1% Si, Mn, V, and/or Mo, are heated at 500° in ammonia for 70 hrs.

A. R. POWELL.

**Separation of zinc, tin, lead, bismuth, cadmium, antimony, arsenic, and similar elements from sulphide ores or secondary products by means of blast-smelting.** E. J. KOHLMAYER (B.P. 336,670, 19.7.29).—A mixture of zinc or lead sulphide ore with pyrites is melted in a rotating drum-furnace the length of which is equal to its diameter, so that the heating flame passes forward and backwards through the furnace. The slag is skimmed off and the charge blown with air, whereby the zinc or lead is oxidised and volatilised, leaving a residue of ferrous sulphide. By continuing the air blast and occasionally adding small quantities of lime the iron may be completely converted into calcium ferrite, which is suitable for smelting in the blast furnace for the recovery of its iron content. The method is also applicable to the recovery of tin, bismuth, cadmium, arsenic, and antimony from pyritic ores. Addition of a small quantity of iron to the charge after removing the slag accelerates the removal of the volatile metal.

A. R. POWELL.

**Reduction of zinciferous materials.** E. H. BUNCE and G. T. MAHLER, ASST. to NEW JERSEY ZINC CO. (U.S.P. 1,749,126—7, 4.3.30. Appl., 4.1.28).—(A) Roasted

zinc ore is reduced with carbon in the usual way and the gases and vapours are passed through a briquetted mass of zinc oxide and carbon or through a tower packed with fireclay and/or carbonaceous material, at such a temperature that the lead and other impurities in the zinc vapour are condensed and entrapped in the porous mass while the zinc vapour passes to the usual condensing system. (B) The operation is carried out in a single continuous operation by charging the briquetted mass of zinc oxide and carbon into the top of a vertical retort, maintaining the temperature of the upper layers at 800–900° so that the lead is entrapped therein, and removing the purified zinc vapours from a point adjacent to the charging door.

A. R. POWELL.

**Treatment by volatilisation of zinc ores and mixtures thereof.** A. FOLLIET and N. SAINDERICHIN (B.P. 337,636, 28.11.29. Fr., 19.10.29).—Crushed zinc sulphide ore is passed in a thin layer through a furnace into which is blown a blast of air at 650–800°; in the first zone oxidation and removal of the sulphur is effected, and in the second carbon is added to cause reduction and volatilisation of the zinc.

A. R. POWELL.

**[Zinc] alloy for bearing surfaces.** H. C. HALL (B.P. 336,881, 30.12.29).—The alloy comprises zinc with 8–18% Al, 0–1% Cu, and 0–1% Ni, and is suitable for use up to 200°.

A. R. POWELL.

**Utilisation of dolomite or magnesian limestone for production of magnesium etc.** H. H. DOW and E. O. BARSTOW, ASSRS. to DOW CHEM. CO (U.S.P. 1,749,210—1, 4.3.30. Appl., [A] 30.10.26, [B] 8.11.26).—(A) Powdered dolomite made into a slurry with water is treated with the chlorine produced in the electrolytic magnesium cell and with sulphur dioxide, whereby a solution of magnesium chloride and a residue of calcium sulphate are obtained. (B) Chlorine from the cell is caused to react with sulphur dioxide in the presence of water and the resulting solution of hydrochloric and sulphuric acids is treated with calcium chloride to remove the latter acid. The resulting hydrochloric acid solution is neutralised with dolomite and heated with sufficient calcined dolomite to precipitate all the magnesium as hydroxide, which is washed free from calcium compounds and dissolved in a further portion of the hydrochloric acid to obtain magnesium chloride. The calcium chloride produced is returned to the first stage of the operation.

A. R. POWELL.

**Refining of magnesium and its alloys.** I. G. FARBENIND. A.-G. (B.P. 336,498, 13.12.29. Ger., 2.1.29).—Iron may be removed from magnesium and its alloys by stirring into the molten alloy at 900° about 1.5% of finely-powdered manganese and keeping the mixture at 700° for 2 hrs., whereby the iron and manganese settle to the bottom of the pot and the purified magnesium may be decanted off; it contains less than 0.02% Fe.

A. R. POWELL.

**Improving the mechanical properties, especially the compression yield point, of articles made from magnesium and its alloys.** I. G. FARBENIND. A.-G. (B.P. 337,706, 20.2.30. Ger., 11.3.29).—The metal or alloy is extruded into rods at 300–350° at a rate between

half the normal rate and the full normal rate and the metal is quenched immediately it issues from the press.

A. R. POWELL.

**Casting of magnesium and its alloys.** J. E. HOY, Assr. to Dow CHEM. Co. (U.S.P. 1,751,038, 18.3.30. Appl., 3.7.26).—The air in the mould is replaced by sulphur dioxide and sulphur vapour by igniting a quantity of sulphur in the sprue just prior to casting the metal.

A. R. POWELL.

**Light metal [aluminium] alloy.** H. T. TILLQUIST and J. HÄRDÉN (B.P. 337,099, 17.8.29).—Aluminium-chromium alloys are produced by melting aluminium and chromium in hydrogen or in a vacuum in a high-frequency induction furnace. The alloys are suitable for introducing small quantities of chromium into aluminium or other aluminium alloys.

A. R. POWELL.

**Light-metal [aluminium] alloy.** BIRMINGHAM ALUMINIUM CASTING (1903) Co., LTD., and P. PRITCHARD (B.P. 337,558, 9.10.29).—An alloy of aluminium with 3.5% Mg and 0.5% Mn is claimed. When subjected to anodic oxidation a dense, strong, and adherent film is produced which is highly resistant to corrosion.

A. R. POWELL.

**Casting of molten metals or alloys [of lead].** H. HARRIS (B.P. 336,727, 27.8.29).—The molten lead or lead alloy is fed from an overhead melting vessel through a series of nozzles into the space between two parallel horizontal drums revolving in opposite directions in such a way that the metal is in a plastic semi-solid state as it is squeezed between the drums, and issues therefrom in the form of a sheet.

A. R. POWELL.

**Treatment of complex ores of copper, cobalt, and nickel.** R. S. CARRERAS (B.P. 336,584, 10.7.29).—The ore is dissolved anodically in dilute sulphuric acid containing 5% of sodium chloride and the solution is electrolysed for the recovery of copper, using insoluble anodes. The electrolyte is then evaporated fractionally for the recovery of cobalt and nickel sulphates from which the metals are recovered by electrolysis.

A. R. POWELL.

**Smelting of [chromium, manganese, and vanadium] ores.** ELECTRO METALLURG. Co., Assces. of F. M. BECKET (B.P. 309,594, 10.4.29. U.S., 13.4.28).—Ferro-alloys of the above metals are obtained by the silicothermic reduction of their oxides or alkaline-earth salts in a self-propagating reaction mixture of such a composition that the slag formed contains a relatively high percentage of the desired metal which is subsequently recovered by reduction with carbon in an electric furnace. *E.g.*, a mixture of vanadium pentoxide, ferro-vanadium-silicon, lime, steel scrap, and sodium nitrate is ignited to obtain ferrovanadium containing 35% V, 7% Si, and <0.3% C, and a slag containing about 10% V. The slag is then smelted with carbon to produce the ferrovanadium-silicon alloy for the first operation.

A. R. POWELL.

**Working up of chromium ores.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,970, 22.7.29).—Chromite is mixed with 1–2% of sodium nitrate and calcined in air at 800–1100° for 30 min. prior to the usual roasting treatment with lime and sodium carbonate.

A higher extraction of chromium is obtained in a shorter time after the pretreatment.

A. R. POWELL.

**Manufacture of hydrocarbon-containing metals and alloys.** BAYERISCHE METALLWERKE A.-G. (B.P. 316,103, 22.7.29. Ger., 20.7.28).—Tungsten, chromium, vanadium, or titanium, or their alloys or oxides are heated below 2500°, preferably at 1400–1600°, in an atmosphere of hydrocarbon vapour, *e.g.*, acetylene, benzene, or naphthalene, containing 1–2% of ammonia, whereby a hard surface layer is produced suitable for metal-cutting or wire-drawing.

A. R. POWELL.

**Manufacture of tungsten.** J. GRAY. From A. PACZ (B.P. 337,160, 3.10.29).—A solution of sodium tungstate is mixed with silver nitrate and the mixture is poured into hydrochloric acid to obtain a mixture of tungstic acid and silver chloride. After washing and drying, the powder is mixed with thorium nitrate and reduced in hydrogen. The resulting metal powder is pressed into bars which are sintered in hydrogen under such conditions that the whole of the silver is removed by volatilisation. The ingots are then swaged and drawn into wire as usual. The silver used in the process should be 0.5–10%, and the thorium about 1.5%, of the tungsten used.

A. R. POWELL.

**[Tungsten-cobalt] alloy for cutting tools.** B. E. FIELD, Assr. to HAYNES STELLITE Co. (U.S.P. 1,750,796, 18.3.20. Appl., 29.8.25. Renewed 11.1.29).—The alloy contains at least 35% W and 30% Co together with up to 12% Cr and 0.20–1.25% B.

A. R. POWELL.

**Dressing of residues containing metal.** HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 319,327, 13.9.29. Ger., 20.9.28).—The material is ground and washed or air-leigated to remove dross etc. and leave a residue of fairly clean metal which is melted in an induction furnace. The dross etc. is smelted separately or treated chemically to recover its content of valuable metal.

A. R. POWELL.

**Casting of metals.** I. SUGIMURA, K. MAGARISAWA, and M. YASUMOTO (B.P. 337,286, 8.1.30).—Two immiscible metals, such that one has a lower m.p. and higher  $\delta$  than the other, are melted together, stirred, and allowed to settle until the lighter metal floats in a liquid mass just below the surface of the heavier. On casting the mixture the heavier metal forms a tightly adherent sheath around a core of the lighter metal. Examples of such pairs of metals are cast iron-copper, cast iron-lead, zinc-lead, and aluminium-lead; the first-named metal in all cases forms the core of the casting.

A. R. POWELL.

**Production of age-proof welds by the fusion-welding method.** F. KRUPP A.-G. (B.P. 316,555, 25.7.29. Ger., 30.7.28).—The welding wire used is an alloy of iron with 0.1% Al and 0.6% Mn or other deoxidising agents, 1% Ni, about 1% Cr, and 0.4% Mo or 1% W. The wires are preheated at 800–1000° in hydrogen to eliminate oxygen, sulphur, carbon, and phosphorus.

A. R. POWELL.

**Welding of bronze.** L. J. TIBBENHAM (B.P. 336,402, 1.10.29).—The welding rod is coated with a mixture of 49% of boric acid, 9% of borax, 13% of iron oxide, 17% of cupric oxide, and 12% of sodium silicate solution.

A. R. POWELL.

**Sealing or joining of metal objects to insulating material.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 337,491, 12.8.29).—The metal objects comprise ring- or disc-shaped parts composed of an alloy of 50–90% Fe with one or more of the elements tungsten, molybdenum, tantalum, niobium, cobalt, or silicon, and up to 5% Cr, Al, or Mg. The composition of the alloy is such that its coefficient of expansion is within  $\pm 20\%$  of that of glass.

A. R. POWELL.

**Electrodeposition of chromium.** C. H. PROCTOR, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,749,443, 4.3.30. Appl., 9.3.29).—A bath containing (per gal.) 8–48 oz. of chromium trioxide, 0.08–0.5 oz. of sulphuric acid, and 0.25–1.25 oz. of "finely-ground chrome-iron ore" is claimed.

A. R. POWELL.

**Stripping of chromium plating.** TERNSTEDT MANUFG. CO., Assees. of J. F. McCULLOUGH (B.P. 336,532, 11.2.30. U.S., 6.3.29).—The articles are treated anodically at 12 volts in a 6–12% solution of oxalic, citric, or tartaric acid at 25–30°.

A. R. POWELL.

**Electrolytic production of light metals [magnesium].** E. O. BARSTOW, Assr. to DOW CHEM. CO. (U.S.P. 1,755,380, 22.4.30. Appl., 9.11.25).—The electrolysis is conducted in a steel vessel which acts as the cathode and is provided with a centrally disposed carbon anode surrounded by a fireclay diaphragm. Outside this diaphragm is a rectangular box the end walls of which are submerged below the surface of the electrolyte, whilst the side walls extend well above the surface. The box is provided with two transverse partitions, one extending well below the other, and outside the longer partition a propeller is fixed so that its motion draws the surface layers of the electrolyte through the box in such a way that the suspended particles of magnesium are trapped between the two partitions in the box and there caused to coalesce to a mass of molten metal.

A. R. POWELL.

**Tilting gear for crucible furnaces.** J. H. LUDLOW, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 338,013, 25.11.29).

**[Sand-moulds for] casting of stainless steel.** W. E. MARTIN and J. A. BERLYN (B.P. 337,413, 29.7.29).

**Catalysts for ammonia oxidation** (B.P. 337,680). **Non-corrosive brine** (U.S.P. 1,750,761). **Magnesium chloride** (U.S.P. 1,756,247). **Sulphur dioxide from smelter gases** (U.S.P. 1,758,398).—See VII. **Enamel ware** (B.P. 336,797).—See VIII.

## XI.—ELECTROTECHNICS.

**Electrical method and instrument for measuring liquid consistency.** C. E. FAWKES (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 543–547).—The consistency load impressed on a submerged rotor impeller turning in the liquid under standard conditions is electrically indicated on a visual dial. From the readings obtained and the sp. gr. of the liquid, viscosity in c.g.s. units can be calculated.

S. S. WOOLF.

**Process control with the "electric eye" [photoelectric cell].** C. A. STYER and E. H. VEDDER (Ind. Eng. Chem., 1930, 22, 1062–1069).—Various types of

photoelectric cells are described. These are suitable, when used in conjunction with the necessary current-amplifying devices, for, *e.g.*, indicating and recording the density of smokes, controlling the turbidity of the thickener overflow in the concentration of metallurgical slimes, counting such objects as hot ingots or bags of salts etc. discharged from a conveyor, or for use in the paper industry. Other examples given, with quantitative data, include the automatic recording of concentration of copper sulphate solution, by colour changes, and of sulphuric acid, by refractive index. C. IRWIN.

**Photoelectric cells in chemical technology.** A. J. McMASTER (Ind. Eng. Chem., 1930, 22, 1070–1073).—The characteristics of vacuum and gas-filled photoelectric cells are described (*cf.* preceding abstract). The latter are the more sensitive and the former the more constant. Relations of cell currents and light units are given, together with the variation of effect with varying wave-length of light. Relays are also referred to. Whenever cells are used for quantitative measurements, regular calibration from a standard is necessary. They may be used in the measurement of optical properties of materials, or the determination of the light-transmissive power, reflecting power, or absorption factor of metals, glass, tiles, other ceramic materials, paints, varnishes, cellulosic products, etc.

C. IRWIN.

**Gas-analysis apparatus.** JARRIER.—See I. **Nitrogen-fixation plant.** WESTBY.—See VII. **Titration of chromium in steel and ferrochromium.** SPINDECK. **Chromium.** ILINSKI and others. **Antimony.** VOGEL. **Bismuth in lead ores.** COLLIN.—See X. **Electrical properties of rubber.** CURTIS and others.—See XIV. **Conductometer for sugar factories.** ŠANDERA.—See XVII. **Universal thermostat.** FINK. **Quartz lamp and wine analysis.** HEIDUSCHKA and MÖHLAU.—See XVIII.

## PATENTS.

**Electric-furnace resistor.** F. A. FAHRENWALD (U.S.P. 1,755,350, 22.4.30. Appl., 19.5.24).—A cast-metal resistor consisting of a sinuous grid having straight legs of ribbed section, connected at alternate ends by integral loops, some of which have outwardly-extending prongs, is claimed.

J. S. G. THOMAS.

**Metal-arc and metal-vapour lamps. Electric-discharge tubes.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 336,208 and 336,222, 5.7.29).—(A) In an electric-discharge tube containing a mixture of metals or an alloy, *e.g.*, cadmium amalgam, which preferably forms an electrode of the tube, a channel separated from the discharge space is provided to return the condensed metal to the part of the tube containing the alloy or mixture and deliver it below the surface thereof. (B) To prevent deposition of metallic vapour on the tube wall, that part of the wall to which metal is liable to adhere is coated with, *e.g.*, calcium fluoride, which prevents such deposition. Alternatively, the tube wall may be coated with potassium chloride, which reacts with deposited metallic vapour, *e.g.*, magnesium, to form products which are volatile or do not absorb the radiation emitted by the discharge.

J. S. G. THOMAS.

**Manufacture of thermionic electrodes of vacuum-electric tube devices.** WESTINGHOUSE LAMP CO., Assecs. of M. N. FREDENBURGH (B.P. 315,324, 11.7.29. U.S., 11.7.28).—Alkaline-earth carbonates precipitated in a predetermined minimum time at or above 85°, from an approx. 15% solution of soluble salts, *e.g.*, the nitrates of barium and/or strontium and calcium, the last-named being present to the extent of 10–20% of the total anhydrous salts, by addition of a solution of *N*-ammonium carbonate, are converted into oxides, which are applied to the electrodes. J. S. G. THOMAS.

**[Electrode for] photosensitive cells.** ARCTURUS RADIO TUBE CO., Assecs. of S. RUBEN (B.P. 336,397, 30.9.29. U.S., 11.1.29).—A copper electrode, coated with cuprous oxide by heating at about 1000° and etched by a solution of ammonium chloride, is partly encased in an insulating compound, *e.g.*, beeswax and resin. J. S. G. THOMAS.

**Manufacture of coloured electric glow lamps.** I. G. FARBENIND. A.-G. (B.P. 315,313, 5.7.29. Ger., 11.7.28).—Lamps are varnished with a solution of a polymerised vinyl ester in an organic solvent containing a colouring matter, *e.g.*, aminoazobenzene. J. S. G. THOMAS.

**Cement for caps for electric incandescence lamps etc.** GEN. ELECTRIC CO., LTD., Assecs. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 316,866, 5.7.29. Ger., 4.8.28).—A fatty drying oil, *e.g.*, linseed or tung oil, which is but little decomposed by heat, together with, if desired, hexamethylenetetramine or other condensing agent for artificial resins, is added to cements consisting of a spirit solution of artificial resins and a filler. J. S. G. THOMAS.

**Alkaline storage battery.** J. F. MONNOT (U.S.P. 1,780,935, 11.11.30. Appl., 31.7.28. U.K., 4.11.27).—See B.P. 297,996; B., 1928, 864.

**Composition of matter. [Insulating material.]** M. DARRIN, Assr. to F. N. BURT CO., LTD. (Re-issue 17,867, 11.11.30, of U.S.P. 1,644,711, 11.10.27).—See B., 1928, 129.

**Luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., Assecs. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 336,490, 4.12.29. Ger., 13.7.29).

**Light-sensitive devices.** COMP. DES LAMPES (B.P. 377,477, 6.8.29. Fr., 10.4.29).

**Thermostats** (B.P. 337,093). **Measuring humidity** (B.P. 336,855).—See I. **Pure *n*-butyl alcohol** (B.P. 335,683).—See III. **Purified fibre** (U.S.P. 1,757,768). **Sheet material** (U.S.P. 1,757,010).—See V. **Magnetic alloys** (B.P. 336,948). **Welding electrodes for steel** (B.P. 337,589). **Magnesium** (U.S.P. 1,749,210—1 and 1,755,380). **Light metal alloy** (B.P. 337,558). **Treatment of complex ores** (B.P. 336,584). **Metal-insulation joints** (B.P. 337,491). **Chromium** (U.S.P. 1,749,443 and B.P. 336,532).—See X.

## XII.—FATS; OILS; WAXES.

**Determination of butter fat.** E. SCHWEIZER and J. GROSSFELD (Z. Unters. Lebensm., 1930, 59, 494—501).—During the course of three years a number of

samples of butter, margarine, and mixtures of the two have been examined to determine the constancy of the Reichert–Meissl value and the butyric acid value. The mean percentage deviation from the normal values of 27.0 and 20.0 were found to be  $\pm 10.0$  and  $\pm 7.4$ , respectively, for samples of normal butter. The mean of the Reichert–Meissl values (37 samples) was 28.7, which corresponds closely with the van Raalte value of 28.4. The ratio of the butyric acid value to the Reichert–Meissl value was found to exceed in all cases the value 0.66 (mean of 37 samples, 0.713), and thus confirms van Raalte's observation that for pure butter the "xylol percentage number" should exceed 66.0. In addition to these characteristic figures, the values of Juckenack's difference (saponification value — Reichert–Meissl value — 200) and Grossfeld's difference (saponification value — 1.5  $\times$  butyric acid value) have been studied and their significance in the detection of adulteration is exemplified. H. J. DOWDEN.

**Which drying process gives a reliable determination of fatty acids in soap?** J. DAVIDSOHN (Chem. Umschau, 1930, 37, 293—296).—The volatility of the fatty acids of the kernel oils leads to inaccuracy in this determination. Mixtures of dried coconut and tallow fatty acids were studied under various conditions of temperature etc. The losses at 100° are too great to permit the use of this temperature in drying, and are not reduced by the admixture of paraffin wax. Goldschmidt's second method (Z. Deuts. Oel- u. Fett-Ind., 1920, 40, 407), in which the desiccated ethereal extract of the fatty acids is evaporated to constant weight at 60°, gives reliable results, but the treatment with anhydrous sodium sulphate need not be prolonged beyond 10 min. and the process may be shortened by evaporating the last traces of ether at 50° in an air current.

E. LEWKOWITSCH.

**Apparent iodine value of elæostearic acid.** J. VAN LOON (Chem. Umschau, 1930, 37, 269—271).—The curve of apparent iodine value of  $\beta$ -elæostearic acid is similar to that obtained for tung oil (B., 1930, 996) and shows a kink at iodine value 180, corresponding to two-thirds saturation of the acid.  $\beta$ -Elæostearic acid appears to undergo slight polymerisation on keeping under alcohol. There is evidence of the presence in tung oil of a hitherto unknown highly unsaturated acid, which is fully halogenated under the conditions studied.

E. LEWKOWITSCH.

**Value of by-products of saccharin manufacture to the oil and fat industry.** W. HERZOG (Chem. Umschau, 1930, 37, 296—298).—The products formed by the condensation of *p*-toluenesulphonamide with fatty acid chlorides, *e.g.*, *p*-toluenesulphopalmitamide, m.p. 100–102°, yield salts with alkalis and alkaline-earths that are of value as emulsifying agents. The preparation and properties of chloroamine, chloramine-T, etc., and their use as preservatives for edible fats (cf. G.P. 408,647; B., 1925, 375) or in disinfectant soaps (cf. G.P. 401,011; B., 1924, 987), or possibly for determination of iodine value (cf. Margosches and Frischer, B., 1927, 727) are described. E. LEWKOWITSCH.

**Chinese tung oil seeds.** ANON. (Bull. Imp. Inst., 1930, 28, 267—272).—Tung oil, extracted from one



sample of nuts from *Aleurites Fordii* and from two samples of *A. montana*, had the following respective characteristics: yield (from moisture-free kernels) 58.5%, 62.6%, 59.8%;  $d_{15}^{25}$  0.9428, 0.9381, 0.9405; acid value 0.44, 12.1, 1.5; saponif. value 192.3, 191.5, 192.6; iodine value (Wijs, 3 hrs.) 179.2, 163.1, 170.0; unsaponifiable matter 0.9, 0.6, 0.7%;  $n_{40}^{20}$  1.5155, 1.506, 1.509; heat test (Browne, min.) 11, 24, 20.5; solidif. point of fatty acids 30.0°, 38.2°, 41.1°. The residual respective meals, as prepared from the kernels, had (%): moisture 10.3, 9.7, 12.8; crude proteins 37.9, 37.9, 47.9; fat 0.4, 1.6, 0.7; carbohydrates etc. (by diff.) 35.9, 33.4, 24.7; crude fibre 7.8, 7.1, 6.8; ash 7.7, 10.3, 7.1%; the nutrient ratios were 1:1.0, 1:1.0, 1:0.5; and food units 132, 132, 146. Meals from the whole nuts are also described.

E. H. SHARPLES.

**Blown linseed oil and sulphur-treated linseed and wood oils.** A. HOLLANDER (Farben-Ztg., 1930, 36, 118—120).—The enhanced water resistance of "vulcanised" and blown oils (cf. B., 1930, 382) is illustrated by further data, experimental details being quoted. The gel formation in such films before drying is a combination of the ordinary "oxypolymerisation" with sulphur adsorption by the "molecular valencies" of the double linkings. The latter effect hinders the oxygen absorption due to the "main valencies," and hence retards drying and degradation processes. The drying of these oils (with the exception of "wood oil fatice") is considered to occur mainly by "desolvation."

S. S. WOOLF.

**Perilla oil.** H. P. KAUFMANN (Allg. Oel- Fett-Ztg., 1930, 27, 39—40; Chem. Zentr., 1930, i, 3735).—The following values were obtained: iodine value (Kaufmann) 204, 204; thiocyanogen value 124, 129. Of the total fatty acids 0.69, 0.66% represents unsaponifiable matter; 7.6, 6.7% saturated acids; 3.9, 10.7% oleic acid; 44.3, 33.6% linoleic acid; and 44.2, 49.0% linolenic acid (20.1, 21.3% of which is in the  $\alpha$ -form). The oil contains unsaponifiable matter 0.7, 0.7; saturated acids 7.2, 6.3; oleic acid 3.7, 10.1; linoleic acid 41.9, 31.9; linolenic acid 41.7, 46.4%.

A. A. ELDRIDGE.

**Sunflower seed from Southern Rhodesia.** ANON. (Bull. Imp. Inst., 1930, 28, 272—276).—Six representative samples of varieties of sunflower seed from S. Rhodesia contained 6.6—7.7% of moisture; the oil contents, on moisture-free seeds, and the respective acid values, were 30.7, 0.8; 29.6, 0.9; 29.3, 0.8; 29.2, 0.8; 26.1, 0.6; 31.8, 0.5%. The first and last compare favourably with the quality usually crushed in Europe.

E. H. SHARPLES.

**Dhupa kernels (*Vateria indica*) from India.** ANON. (Bull. Imp. Inst., 1930, 28, 279—281).—Extraction of the kernels of *V. indica* yielded 23.2% of greenish-white fat (on dry kernels) having  $d_{15}^{25}$  0.8585, m.p. (open tube) 34.2°, acid value 1.0, saponif. value 190.3, iodine value (Wijs, 3 hrs.) 45.9, unsaponifiable matter 0.9%,  $n_{40}^{20}$  1.4588, solidif. point of fatty acids 52.7°. The residual meal had moisture 8.8%, ether extract 4.1%, crude proteins 6.2%, crude fibre 5.0%, ash 2.1%, carbohydrates etc. (by diff.) 73.8%, nutrient ratio 1:13.4,

food units 100. The meal was free from cyanogenetic glucosides and alkaloids, but had a bitter taste which would render it unsuitable for use as a feeding-stuff.

E. H. SHARPLES.

**Moringa aptera seed from Egypt.** ANON. (Bull. Imp. Inst., 1930, 28, 276—279).—The kernels (4.9% of moisture) from the seeds (wt. 0.5 g., 51.2% of shell) of *M. aptera* on extraction yield 50% of oil having  $d_{15}^{25}$  0.9151,  $n_{40}^{20}$  1.461, acid value 0.5, saponif. value 188.2, iodine value (Wijs, 3 hrs.) 71.2, unsaponifiable matter 0.5%, solidif. point of fatty acids 28.1°. The residual meal has moisture 8.7%, crude proteins 48.6%, fat 2.6%, carbohydrates etc. (by diff.) 28%, crude fibre 6.6%, ash 5.5%, nutrient ratio 1:0.7, food units 156. The meal is free from cyanogenetic glucosides, but contains a saponin and, probably, a substance of an alkaloidal nature. Both the oil and meal are very similar to those from *M. pterygosperma* (from which Ben oil is derived).

E. H. SHARPLES.

**American cherry-kernel oil.** G. S. JAMIESON and S. I. GERTLER (Oil & Fat Ind., 1930, 7, 371—372, 387).—The expressed oil (32—40%) from the kernels of the sour cherry (cf. Rabak, B., 1917, 159) after refining was pale in colour and had (figures for the crude oil in parentheses):  $d_{15}^{25}$  0.9183 (0.9176),  $n_D^{25}$  1.4740 (1.4742), acid value 0.09 (4.39), iodine value (Hanus) 115.8 (118.7), saponif. value 190.7, Reichert-Meissl value 0.3, Polenske value 0.2, unsaponifiable matter 0.5% (0.66%), saturated acids (corr.) 7.7% (lead salt-ether method), unsaturated acids (corr.) 87%, iodine value 127.9. The composition of the oil is given as: acids (%): oleic 46.85, linoleic 40.11, myristic 0.19, palmitic 4.04, stearic 2.79, arachidic 0.72. The refined oil, which has a bland flavour and good keeping properties, should be suitable for use as a salad oil and in cosmetic manufacture.

E. LEWKOWITSCH.

**Abutilon oil.** L. JOLSON (Masloboino Zhir. Delo, 1929, No. 8, 22—25).—Cultivated abutilon seeds contain moisture 9.7, oil 16.5, crude protein 19.8, cellulose 16.9, ash 5.35, and nitrogen-free extract 31.75%; the oil has  $d_{20}^{20}$  0.9265, iodine value 131.7, saponif. value 190.6, acid value 4.42, unsaponif. matter 0.80%, and is similar to cottonseed and soya-bean oil. The fatty acids of the oil have f.p. 19.9°, iodine value 124.2, neutralisation value 190.3.

CHEMICAL ABSTRACTS.

**Fatty oil of coriander.** G. NORKIN (Masloboino Zhir. Delo, 1929, No. 8, 25—26).—Coriander seed contains, in addition to the essential oil, a fatty oil having  $d_{15}^{25}$  0.919,  $n_D^{25}$  1.4688, acid value 7.84, saponif. value 192.15, iodine value 93.81, Reichert-Meissl value 0.49; the acids have mol. wt. 291.4,  $n_D^{25}$  1.4678, neutralisation value 196.5, iodine value (Hübl) 97.46. The colour reactions of the oil and the properties of the sodium soap are recorded.

CHEMICAL ABSTRACTS.

**Study of the official method of bleaching test of the American Oil Chemists' Society.** C. MA and J. R. WITTHROW (Ind. Eng. Chem. [Anal.], 1930, 2, 374—377).—The results of experiments with varying conditions justified the manipulative details prescribed in the official test; full bleaching can be obtained, however, in 1 min. at temperatures between 92° and 125.°

On either side of these limits the degree of bleaching decreases. No increased bleaching is obtained by increasing the amount of fuller's earth beyond 7%, or prolonging the treatment beyond 5 min. The Hess-Ives tint photometer (cf. Kress and McNaughton, B., 1916, 449) is useful, and the degree (%) of bleach may be satisfactorily expressed as  $100(T-L)/(100-L)$ , where  $T$  and  $L$  are the total percentage luminosities of the bleached and unbleached oils, respectively.

E. LEWKOWITSCH.

**Action of silent [electric] discharge on oils, fats, and fatty acids.** V. Catalytic properties of added substances. I. Y. IWAMOTO (J. Soc. Chem. Ind., Japan, 1930, 33, 247—248 B; cf., *ibid.*, 1929, 32, 93 B).—Oleic acid was subjected for 6 hrs. to the action of the silent discharge in an atmosphere of hydrogen and in the presence of 5% of various metallic and metallic-soap catalysts, and the iodine value, stearic acid content, etc. of the products were determined. Platinum and palladium act as powerful positive catalysts for the hydrogenation; uranium, iron, bismuth, and aluminium oxides are weaker in action. Bismuth oxide and ferric chloride appear to accelerate polymerisation rather than hydrogenation. Copper, zinc, and magnesium oxides, titanium chloride, and cobalt hinder both polymerisation and hydrogenation, whilst silver oxide and zinc chloride entirely inhibit any action of the silent discharge.

E. LEWKOWITSCH.

**Colour reactions of [fatty] oils.** W. H. DICKHART (Oil & Fat Ind., 1930, 7, 391).—10 C.c. of the oil, acidified with 1 c.c. of concentrated hydrochloric acid, are shaken for 1—2 min. with 10 c.c. of amyl alcohol, and the liquids allowed to separate. Soya-bean oil (also admixed with 9 vols. of tung oil) produces a deep blue colour persistent for 24 hrs. Linseed and perilla oils give a blue colour fading in 24 hrs. to yellow and pale blue, respectively. Olive, cottonseed, arachis, sunflower, rape (yellow after 24 hrs.), and tung oil solutions are colourless. Crude cottonseed and palm oils give red colours.

E. LEWKOWITSCH.

**Analysis of olein.** H. P. KAUFMANN [with R. WALTHER] (Allg. Oel-Fett-Ztg., 1930, 27, 4—9; Chem. Zentr., 1930, i, 3735).—A discussion. Normal oleins contain more saturated and higher unsaturated fatty acids than was previously supposed.

A. A. ELDRIDGE.

**Transformation of fatty acids.** STADNIKOV and VOSSCHINSKAJA.—See II. **Waxed paper.** THOMAS and REBOULET.—See V. **Soap in paints.** BALDWIN. **Determination of insolubles and wax in shellac.** STILLWELL.—See XIII. **Colloid chemistry of butter.** KING. **Determination of fat in chocolate.** WISEMAN.—See XIX.

#### PATENTS.

**Extraction of fatty oil.** M. SATO and (A) C. ITO, (B) Y. ISHIDA (B.P. 336,273—4, 10.6.29).—The material is extracted with (A) alcohol under pressure at a temperature above the b.p. of the solvent and below 120°, or (B) with a hot (above 65°) mixture of alcohol and light petroleum (and/or benzene) in the proportions, e.g., 9:1, and the oil is separated from the extract by cooling to below 50°. A continuous process is described

in (B), by which soya beans yielded 19.5% of a pale oil and a meal rich in nitrogen. E. LEWKOWITSCH.

**Refining of edible oils.** R. B. HILL, Assr. to BROWN Co. (U.S.P. 1,749,976, 11.3.30. Appl., 30.11.23).—Oils are decolorised by treatment with 0.05—0.15% of a 30—75% (by wt.) solution of sulphuric acid. The oils may be further treated with alkali and the soap-stock coagulated with powdered bentonite (ardmorite).

E. LEWKOWITSCH.

**Manufacture of lubricating grease.** E. N. KLEM-GARD (U.S.P. 1,749,251, 4.3.30. Appl., 17.3.27).—Smooth transparent buttery greases are prepared by heating paraffin oil with 5—25% of (commercial) aluminium stearate at 168—289° until the mixture is dehydrated and the stringy texture destroyed, and then cooling without agitation.

E. LEWKOWITSCH.

**Preparation of emulsions.** K. HATTORI (U.S.P. 1,752,176, 25.3.30. Appl., 31.3.26).—Protalbic acid (e.g., 2—10 pts. per 1000) is used as emulsifier for cod-liver oil (with or without, e.g., lactic acid), camphor, etc.

E. LEWKOWITSCH.

**Soap-making and -forming apparatus.** L. H. NELLES (B.P. 337,430, 2.5.29).

**Hydrogenation** (B.P. 336,569).—See I.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Metallic soaps.** II. **Effect of soaps in paints—preparation of paints.** J. T. BALDWIN (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 492—506; cf. B., 1930, 67).—The consistency and settling and weathering properties of a series of paints containing lithopone, zinc oxide, basic carbonate and basic sulphate white lead, and titanox, in vehicles containing a comprehensive range of metallic soaps, were observed, and the results are tabulated.

S. S. WOOLF.

**"Chalking" of paints.** H. WOLFF (Farben-Ztg., 1930, 36, 221—223).—The seriousness of chalking as a paint defect depends on the purpose for which the paint is intended. White paints chalk more than tinted paints, the main cause of chalking being the destruction of the binding vehicle due to the influence of actinic rays; thus admixture of stand oil with a boiled-oil paint reduced chalking considerably. The addition of 0.05% of lampblack to a white paint reduced the extent of chalking by 10% in 6 months and by 30% in 1 year, whilst for 0.1% of tinter the corresponding figures were 47% and 60%. Chalking does not always proceed at a uniform rate: cases are instanced of paints that chalked heavily for 9 months, and then showed a period of 6—9 months in which chalking was much less pronounced before the final disintegration of the film set in. This is explained by the stratification of paint films (previously discussed by Blom and by Wolff; cf. B., 1929, 609, 103), chalking pigments forming a pigment-rich outer layer, whilst the surface layers of paints based on non-chalking pigments have relatively low pigment concentration.

S. S. WOOLF.

**Causes of floating of pigments in paints and enamels.** C. A. HOLKESVIG (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 477—489).—

Colour floating or flooding is a condition of concentration of colour at the surface of a film, due to presence of certain pigments in true or colloidal solution, or to differences in ease or rate of dispersion of pigments. The use of insoluble pigments or pigments of similar dispersion rates in the particular vehicle employed, and the continuation of grinding until differences of dispersion are equalised, will reduce or eliminate floating.

S. S. WOOLF.

#### Synthetic resins in enamels and house paints.

L. K. SCOTT, E. J. PROBECK, and O. MILETI (Amer. Paint and Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 437—449).—A description of exposure tests, the results of which indicate the superior durability of "glyptal" enamels and house paints when compared with "4-hr. enamels" and normal house paints, respectively.

S. S. WOOLF.

The "aridyne"—a standard unit of [paint] drying power. C. F. CARRIER (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 510—519).—The "aridyne," a standard for use in evaluation of driers, is defined as the drying power of 0.064 oz. of oil-soluble metallic lead dispersed in a volatile solvent. "Soligen" driers are suggested as a suitable basis for the preparation of standard drying solutions.

S. S. WOOLF.

Accelerated testing of protective coatings. M. F. MORAM, JUN. (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 548—564).—Eighteen paints were exposed to normal weathering and to five different accelerated weathering cycles and the results are tabulated and discussed.

S. S. WOOLF.

Colour control in the paint plant. M. R. PAUL and J. P. ST. GEORGE (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 419—436).—An account of an investigation into the preparation and maintenance of colour standards by spectrophotometric methods. The practical limits for commercial colour matching are indicated.

S. S. WOOLF.

Selective adsorption by pigments. C. P. VAN HOEK (Farben-Ztg., 1930, 36, 267—269).—Theories explaining the relative durabilities of exterior paints containing white lead, zinc oxide, lithopone, or titanium white in terms of basicity and tendency to soap formation are summarised and shown to be inadequate. Selective adsorption of fatty acids, and retention of the products of oxidation in the film by pigments, are considered to be significant factors leading to non-hardening films. This must be taken in conjunction with the degree of ultra-violet light absorption of the pigment, with proportionate protection of the binding vehicle, and from this aspect zinc oxide is to be regarded as the most durable pigment of the range, being superior to white lead for resisting severe weather conditions. These views are correlated with the "chalking" qualities of lithopone and titanium white, and other theories of chalking are briefly examined.

S. S. WOOLF.

Micrography of coloured pigments. I. Sienna. ANON. (Farben-Ztg., 1930, 36, 120—121).—A summary of the work of H. Wagner and R. Haug on the examination of eleven samples of Italian and indigenous siennas, including optical properties, physical properties, *e.g.*,

*sp. gr.*, state of subdivision, oil absorption, etc., chemical composition, microscopical examination, and a scheme of specification.

S. S. WOOLF.

[Nitrocellulose] solvents. A. HEINEMANN (Farben-Ztg., 1930, 36, 270; cf. Zimmer, B., 1930, 1119).—Attention is drawn to the effect of the molecular complexity of solvents on the size of the nitrocellulose-solvent complexes formed, and hence on the viscosity of nitrocellulose solutions. The significance of the presence of the hydroxyl group in solvents is also indicated.

S. S. WOOLF.

Lacquer studies. I. Development of an abrasion test for use with nitrocellulose lacquers. W. KOCH (Ind. Eng. Chem. [Anal.], 1930, 2, 407—409).—A modification of Sward's abrasion test (B., 1929, 825) is described, in which carborundum powder is driven by a regulated air current against the test film, which is inclined at 45° to the blast, the first appearance of actual holes in the film being taken as the end-point. The "abrasion factor" is recorded as (weight of abrasive used)/(film thickness in cm.  $\times$  1000), a standard lacquer being used for comparison. More than 24 hrs.' ageing of the very thin films used (0.0015—0.0025 cm. thick) has no effect on the abrasion factor.

E. LEWKOWITSCH.

Blooming of varnish films. W. KRUMBHAAR (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 520—532).—The following conclusions are drawn from the behaviour of trial varnishes. To be free from bloom, varnishes should contain sufficient calcium to neutralise free acidity and as much manganese as possible (regarding colour). Zinc, lead, and cobalt driers, stand oils, and blown oils favour bloom. Linseed oil (in the proportion of at least 1½ pts. of oil to 1 pt. of fused resin) gives non-blooming varnishes, as does wood oil in special combination with linseed oil and bodied at not above 210°. The blooming tendencies of resins vary greatly, that of Sierra Leone gum being very low, whilst ester gum and other synthetic resins are unsuitable for use in non-blooming varnishes. The nature and method of addition of solvents used are not significant, with the exception that solvents containing fractions boiling above 221° should be avoided.

S. S. WOOLF.

Resistance to tackiness of certain paint, varnish, and lacquer coatings on the interior of cedar-lined receptacles. F. RABAK (Ind. Eng. Chem., 1930, 22, 1136).—Tests on a number of finishes stored in a small cedar-lined safe show that vapours emanating from the cedar lining act as a slow solvent for the vehicles of certain coatings, *e.g.*, oil enamels, varnishes, asphaltum, etc., softening the films to the extent of tackiness. Paints free from or containing relatively little drying oil, *e.g.*, flat wall paints, lacquers, radiator bronze, were satisfactory, showing no stickiness over a period of 18 months.

S. S. WOOLF.

Determination of wax in shellac. A. G. STILLWELL (Ind. Eng. Chem. [Anal.], 1930, 2, 387).—Powdered shellac (5 g.) is dissolved by boiling in 150 c.c. of water containing 3 g. of sodium carbonate. After cooling to below 15° and filtering through a cotton-wool and asbestos plug in a specially prepared Soxhlet apparatus, the

precipitate is well washed successively with water and with 50 c.c. of 70% alcohol, dried in an oven, and extracted with carbon tetrachloride. The solvent is evaporated and the extract weighed. Results are obtained within 3 hrs. and agree with those from the official method.

E. LEWKOWITSCH.

**Determination of "insolubles" in shellac.** A. G. STILLWELL (Ind. Eng. Chem. [Anal.], 1930, 2, 420—421).—It has been found that the official extraction method fails to remove from the shellac 0.3—0.5% of a wax (m.p. 93°) which passes into varnishes and should not be included as "insoluble." A more rapid method is described in detail in which the powdered lac (5 g.) is dissolved in 50 c.c. of a mixture of alcohol and carbon tetrachloride (2 : 1): the solution is filtered by siphoning through a wad of cotton-wool, and the insoluble portion is washed with hot alcohol, extracted for 1 hr., and dried. The results agree with those obtained by the standard method when extraction is carried to completion.

E. LEWKOWITSCH.

**Determination of the m.p. of resins.** M. RANGASWAMI (J. Oil Col. Chem. Assoc., 1930, 13, 287—288).—Durrans' "mercury method" (cf. B., 1929, 690) is modified by the use of small amounts, e.g., 0.2 g., of resin and by conducting the test in a crucible in place of a test-tube. Within limits, the amount of mercury used does not affect the accuracy of the determination, if heating be sufficiently slow. Typical results are quoted.

S. S. WOOLF.

**New colour reaction for rosin.** H. C. COHEN (Farben-Ztg., 1930, 36, 121).—1 c.c. of a solution of 1 vol. of chlorosulphonic acid in 5 vols. of chloroform is added to 1 c.c. of a 10% chloroform solution of the material under test. Rosin gives a relatively permanent violet-red coloration, albertol and ester gum a rose-pink, drying oils give no colour. One drop of a 2% rosin solution in linseed oil gives a deep red coloration with 1 c.c. of the reagent.

S. S. WOOLF.

**Measurement of liquid viscosity.** FAWKES. Photoelectric process control. STYER and VEDDER; McMASTER.—See XI. Blown and sulphur-treated oils. HOLLANDER.—See XII.

#### PATENTS.

**Manufacture of synthetic resins.** I. G. FARBENIND. A.-G. (B.P. 314,810, 1.7.29. Ger., 2.7.28).—Solvent naphtha, freed from phenols and bases, is condensed below 90° with an alkyl ether of a *m*-substituted phenol, particularly of a *m*-cresol or *m*-5-xyleneol, in presence of a condensing agent (stannic chloride, ferric chloride, fluoboroacetic acid, sulphuric acid) other than aluminium chloride. The resins (m.p. 148°, 138°, 113°, 133°, and 94°, respectively, in the five examples) are miscible in all proportions with petrol and with drying oils.

C. HOLLINS.

**Decoration of [hard] surface-covering material [e.g., linoleum etc.].** ARMSTRONG CORK CO., ASSEES. OF J. C. MCCARTHY (B.P. 337,924, 8.10.29. U.S., 21.11.28).

**Production of thin plates of artificial resin.** C. A. JENSEN (B.P. 337,530, 13.9.29).

**Azo pigments** (B.P. 335,555).—See IV. Mercerised wood-fibre (U.S.P. 1,749,003). Cellulose ether com-

positions (B.P. 312,309). Solvent for cellulose nitrate (U.S.P. 1,756,228). Reducing the viscosity of nitro-cellulose (U.S.P. 1,757,491 and 1,757,481).—See V. Naphthenates (B.P. 335,863). Crystalline zinc sulphide (U.S.P. 1,758,741).—See VII. Coloured glow lamps (B.P. 315,313).—See XI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Fractionally coagulated crêpe [rubber].** LONDON COMMITTEE OF THE CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1930, 28, 294—299).—In fractional coagulation of latex in two successive stages, the first fraction is the richer in acetone-soluble constituents, but the plasticity of both fractions is little different from that of the control sample. Both fractions give vulcanised products of good tensile strength, but vulcanisation is much the more rapid and "flatter" with the first fraction, and the product ages better.

D. F. TWISS.

**Swelling of vulcanised rubber in liquids.** J. R. SCOTT (Trans. Inst. Rubber Ind., 1930, 5, 95—118). The time-swelling curve for vulcanised rubber immersed in an organic solvent consists of an early markedly curved portion representing the initial rapid absorption and "saturation" and an indefinitely prolonged slight slope representing a slow continued absorption, termed the "swelling increment." This increment is probably caused by gradual depolymerisation of the rubber arising from the presence of dissolved oxygen in the swelling liquid; it is greatly increased by addition of depolymerising agents, such as acids or copper salts, or of oxidising agents to the liquid, and decreased by antioxidants and exclusion of oxygen. The swelling maximum is determined by the chemical character of the liquid rather than by its physical properties, but the swelling time (disregarding the "increment") seems to be determined by the physical properties, particularly the viscosity. Increase in temperature and exposure to light increase the swelling maximum. The presence of organic accelerators or of sulphur increases the depolymerisation effect as indicated by the swelling increment, but the swelling maximum is diminished by all factors which reduce the extensibility of the rubber, such as progressive vulcanisation or the presence of reinforcing constituents. A swelling test should be of value for the selection of antioxidant substances and for the detection of injurious ingredients in constructional materials intended for use with rubber.

D. F. TWISS.

**Action of zinc oxide containing iron in rubber mixtures.** R. DITMAR and C. H. PREUSZE (Caoutchouc et Gutta-Percha, 1930, 27, 15,219—15,220).—From the point of view of chemical and mechanical results the presence of iron oxide in zinc oxide, up to 2% on the latter, is completely without modifying influence on the effect of the zinc oxide as a compounding ingredient for rubber. The only observable influence is a tendency to impart a slight colour to otherwise white vulcanisates.

D. F. TWISS.

**Influence of increasing content of different fillers on the calender behaviour of unvulcanised rubber mixtures.** R. DITMAR and C. H. PREUSZE (Caoutchouc et Gutta-Percha, 1930, 27, 15,212—15,214).

—The results of observations are recorded of the effect of various proportions of chalk, zinc oxide, carbon black, and silica gel on the ease of calendering to smooth sheet, and the degree of contraction of the sheet in the next 5 min. Adsorbent fillers decrease the contractive effect and facilitate smooth calendering. D. F. TWISS.

**Permeability of rubber mixings.** W. C. DAVEY and T. OHYA (Trans. Inst. Rubber Ind., 1929, 5, 27—30).—Experiments on the rate of diffusion of hydrogen through sheets of rubber attached to cambric for support, using a constant-flow method and applying an interferometer to the refractometric determination of the diffused hydrogen, show that vulcanisation increases the permeability, but that the presence of an organic accelerator and particularly of an antioxidant or softener improves the result. The presence of mineral fillers in the rubber assists the passage of hydrogen, but the effect is less with finer fillers such as zinc oxide and carbon black. D. F. TWISS.

**Effect of different conditions of storage on the hardness of rubber.** LONDON COMMITTEE OF THE CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1930, 28, 289—293).—Samples of rubber, on storage for 6 months at 0° after their arrival in this country, invariably become hard and frozen, storage at 15° also sometimes leading to the same result; the samples stored at 0°, however, generally require less milling to reach a fixed degree of plasticity. The length of time for which the coagulum remains in the serum on the plantation has no effect on the hardness of smoked sheet rubber, but storage in this country leads to general hardening, the effect being particularly marked in a dried atmosphere. No hardening was observed in an atmosphere of nitrogen. D. F. TWISS.

**Heats of combustion of rubber, gutta-percha, and balata.** T. H. MESSENGER (Trans. Inst. Rubber Ind., 1930, 5, 71—86).—After purification by extraction with water and acetone, respectively, and successive dissolution in benzene, precipitation with alcohol, re-dissolution in chloroform, and re-precipitation with acetone, the dried rubber, gutta-percha, and balata gave substantially the same heats of combustion, viz., 11,000 g.-cal. per g. This result confirms the close chemical similarity between these hydrocarbons and gives no indication of any difference in degree of polymerisation. Disaggregation of rubber by milling or exposure to sunlight also left the heat of combustion unaltered, the alteration effected therefore being presumably physical in character rather than one of real depolymerisation. In an appendix, values obtained by various experimenters for the C:H ratio in rubber, gutta-percha, and balata are compared. D. F. TWISS.

**Effect of temperature, pressure, and frequency on the electrical properties of rubber.** H. L. CURTIS, A. T. MACPHERSON, and A. H. SCOTT (Physical Rev., 1929, [ii], 33, 1080).—The dielectric constant, power factor, and resistivity of rubber depend on the percentage of sulphur used in its vulcanisation, on the temperature and pressure, and, for the first two properties, on the frequency at which measurements are made. L. S. THEOBALD.

**Standardisation of vulcanisation testing [of rubber].** J. R. SCOTT (Trans. Inst. Rubber Ind., 1930, 5, 139—150).—The details of procedure for vulcanisation testing, particularly for the evaluation of raw rubber, are discussed, the following factors receiving consideration: composition and preparation of the test mixture, interval between mixing and vulcanisation, conditions of canisation, interval before tensile testing; character of the tensile tests, and degree of vulcanisation to be adopted for comparative purposes. D. F. TWISS.

**Determination of adsorptive power of amorphous carbon [for rubber] with methylene-blue.** R. DITMAR and C. H. PREUSZE (Gummi-Ztg., 1930, 45, 243—244; cf. Carson and Sebrell, B., 1929, 1024).—To 50 c.c. of a 0.01% solution of methylene-blue at 15° are added small portions from a bulk of 0.5 g. of the carbon black with shaking, until the solution is decolorised; the mixture is then titrated with the methylene-blue solution until faint colour persists, the "adsorptive power" of the carbon black being expressed as the number of parts of methylene-blue adsorbed by 100 pts. The adsorptive power varies widely for different grades of black, and has no simple relation with their relative rates of sedimentation from water or with their appearance in ultra-violet light. Highly adsorptive carbon black retards the rate of vulcanisation of a rubber mixture and also tends to reduce the amount of "overflow" during vulcanisation, so that the sp. gr. of the vulcanisate is greater than for a similar mixture containing a less adsorptive black. The relative ease of wetting by rubber, which is a feature of practical importance in carbon blacks, can be estimated by observing the difference in appearance of two small sheets of masticated crêpe rubber which have been sprinkled very thinly with the two samples of black under comparison and kept at 70° for 45 min. D. F. TWISS.

**Comparison of carbon blacks.** DAWSON and HARTSHORNE.—See II. Ageing of cotton contained in rubber goods. BARR.—See V.

#### PATENTS.

**Preservation of rubber.** J. TEPPEMA, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,781,306, 11.11.30. Appl., 30.11.26).—See B.P. 281,616; B., 1929, 66.

**Method of uniting rubber and other substances.** L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,781,649, 11.11.30. Appl., 26.4.28).—See B.P. 310,461; B., 1930, 472.

**Disintegration of rubber (B.P. 336,828).** Vessels proof against chemicals etc. (B.P. 316,134).—See I. Mercaptobenzthiazole (B.P. 335,567).—See III. Sheet-like solids etc. (B.P. 318,250).—See V.

#### XV.—LEATHER; GLUE.

**Measurement of the swelling of hide by expression of the water.** H. OWRUTSKY (Collegium, 1930, 427—430).—When pelt was swollen in a lime liquor and then weighted, the amount of water removable by pressure (as determined at regular intervals) was found

to diminish as the process was continued. Delimed pelts were next treated with water and with 0.1*N*-solutions of various acids, the thickness of the swollen pelts was measured, the pelts were submitted to pressure, and the weight of water expressed was noted. It was shown that the swelling of the pelt could be measured by the amount of expressed water, but not by the thickness of the swollen pelt, since the pelt swollen in distilled water was thicker than that swollen in 0.1*N*-acids. The water-swollen pelt lost most water (14.1%) by expression, and pelt swollen in 0.1*N*-hydrochloric acid lost least (5.36%), so that the greatest degree of swelling was shown in hydrochloric acid. The water in alkali-swollen pelt is in a state of weaker combination with collagen than is the water in acid-swollen pelt, since it is more readily expressed. D. WOODROFFE.

**Determination of the best conditions of tannage with extracts of *Rhus cotinus*, L., and *Carpinus duinensis*, Scop., at different  $p_H$  values.** A. N. ANFINOV and A. P. PISSARENKO (Collegium, 1930, 419—427).—Examination of *Rhus cotinus*, L., extract liquors ( $d 1.0615$ ) and *Carpinus duinensis*, Scop., extract liquors ( $d 1.028$ ) of different  $p_H$  values showed that the higher was the  $p_H$  value, the greater was the amount of precipitated material. At  $p_H$  8, 75% of *Rhus cotinus* was precipitated. When hide powder was treated with the respective liquors, the maximum amount of material was found to be absorbed by the powder from liquors having  $p_H$  2. Pieces of pelt tanned in these liquors exhibited minimum thickness and swelling and gave the best finished leather when liquors having  $p_H$  5—6 were used. D. WOODROFFE.

**Fluorescence test for [identification of] tanning materials.** C. VAN DER HOEVEN (Collegium, 1930, 414—419).—The fluorescence observed in ultra-violet light on wadding soaked in the tannin solution (a) without further treatment, (b) with the addition of acid or alkali, respectively, is given in tabular form for 62 tanning extracts and materials, synthetic tannins, and sulphite-cellulose extracts. The most characteristic colours were obtained with mangrove bark, ulmo, sulphite-cellulose extract, and certain synthetic tannins. D. WOODROFFE.

#### PATENTS.

[Rotary] apparatus for tanning skins, hides, etc. E. G. WILSON (B.P. 337,408, 22.7.29).

Treatment agents for leather (B.P. 317,039).—See III.

### XVI.—AGRICULTURE.

**Examination of soils.** K. UTESCHER (Z. Pflanz. Düng, 1930, 18A, 203—217).—The value of chemical analyses, notably the acid-extraction processes of the Prussian Geological Institute, in determining the nature and properties of soils is emphasised. A. G. POLLARD.

**Soil colloids. I. Base exchange and soil acidity. II. Factors influencing the dispersion of soil colloids in water.** A. N. PURI (Mem. Dept. Agric. India, 1930, 11, 1—38, 39—51).—I. Experiments with a number of soils have shown that when a soil is exhaustively treated with 0.05*N*-hydrochloric acid and washed free from excess the amount of hydrogen ion

retained by it is a characteristic constant for the particular soil. A part of the hydrogen-ion content can be replaced by any other cation by exhaustive treatment with a neutral salt; the free acid simultaneously thereby liberated is then equivalent to the amount of cation entering the soil complex. The "acidoid" or soil completely saturated with hydrogen ions has been studied as a complex acid in a number of reactions characteristic of acids. Experiments on sucrose inversion were carried out on 6 soils from various parts of the world, but they all indicated the same dissociation constant, the value being close to that of acetic acid. The main result of these investigations is to show that the acidoid possesses the characteristics of a true acid, and that the reactions take place in three distinct stages, in which 1, 2, and 3 equivalents of hydrogen ion take part. The first hydrogen ion is replaced by exhaustive treatment with a neutral salt and the suspension is then neutral. Only the first hydrogen ion can invert sucrose, hydrolyse ethyl acetate, decompose zinc sulphide, or show "exchange acidity." The second hydrogen ion can be replaced by heating the suspension with calcium or barium carbonate in a current of air. Normal salt formation by replacing the third hydrogen ion takes place in the presence of a large excess of alkali. The methods of determining lime requirements of acid soils are discussed in the light of the tribasic acidoid theory, and large-scale experiments on liming have shown that neither liming nor manuring alone much improves the soils. A combination of the two is necessary, and the optimum dose of lime is different in the presence of different manures. After the maximum value for the crop yield with any manure, there is a distinct tendency for the yield to fall. The toxicity of soils is confined to the first replaceable hydrogen ion, and a simple method for its measurement is outlined.

II. The dispersion of soils has been studied as nearly as possible under field conditions by leaving the soil in contact with water for 24 hrs. and determining the dispersed clay (0.002 mm.) by the pipette method. This value is termed the "dispersion factor," and the dispersion coefficient is defined as the percentage of the total clay which can pass into suspension on being left in contact with water for 24 hrs. It is proposed to give the name "clayic acid" to the soil completely saturated with hydrogen ions and to call the salts derived therefrom "clayates." The dispersion coefficients of clayic acid and a number of clayates have been determined; the lithium and sodium salts give considerably higher values than any other. All good soils give a comparatively low dispersion coefficient and all barren alkali soils give a high value. Soils rich in exchangeable sodium give a high dispersion coefficient and a very low rate of percolation to water and whilst treatment with gypsum increases the permeability, it lowers the dispersion coefficient. Highly acid soils give a very low dispersion coefficient and a very high rate of percolation. Irrigated soils give a comparatively higher dispersion coefficient than unirrigated soils. E. S. HEDGES.

**Microflora of leached alkali soil.** J. E. GREAVES and J. D. GREAVES (Bot. Gaz., 1930, 90, 224—230).—The restoration of bacterial activities in alkali soils following leaching varied with the concentration and

nature of the salts present, and is probably due to the removal of inhibitory substances by the saline solutions and a subsequent stimulation of the organisms by the remaining salts. In this respect leaching is less effective in the presence of chlorides than of sulphates. In Ashby media inoculated with soils sulphates stimulated nitrogen fixation to a greater extent than any other salts examined.

A. G. POLLARD.

**Origin, nature, and isolation of the inorganic base-exchange compound of soils.** E. TRUOG and J. A. CHUCKA (J. Amer. Soc. Agron., 1930, 22, 553—557).—Minute quantities of base-exchange material in soil can be detected by treatment with aqueous magenta (0.5%) and washing with water and alcohol, any such material being coloured. The material, which appears to be formed from feldspars under alkaline conditions of weathering, was separated from bentonite and a silt loam sub-soil.

CHEMICAL ABSTRACTS.

**Effect of silicic acid [in soil].** W. KRÜGER and G. WIMMER [with O. RINGLEBEN, O. UNVERDORFEN, J. GRIMM, and H. LÜDECKE] (Z. Ver. deut. Zucker-Ind., 1930, 80, 771—812).—Literature concerning the inter-relationships between soil phosphates and silica in plant nutrition is discussed. The favourable effect of silica on the growth of plants is increased by the presence of ferrous sulphate. The latter in the absence of silica decreases crop yields by reducing the assimilability of phosphates. The bearing of this on the work of Lemmermann is discussed.

A. G. POLLARD.

**Determination of the total surface area of soils, clays, and similar substances.** H. KURON (Z. Pflanz. Düng., 1930, 18A, 179—203).—The adsorption of water by soils, their mechanical fractions, sand, and permutit is examined. At lower vapour pressures adsorption follows the Ostwald-Freundlich rule in all cases, and calculations of total surface area of the materials may be made directly. The point of inflexion of drying-out curves of soil and its constituent fractions occurs at the same vapour pressure. The water adsorption of a soil at various vapour pressures can be represented as the sum of the adsorptive values of its constituent fractions. Drying-out curves for permutit are of a similar nature to those of natural zeolites.

A. G. POLLARD.

**Simplified method of determining "sticky point" of soils.** A. N. PURI (Agric. J. India, 1930, 25, 206—209).—The method of Keen and Coutts (B., 1928, 905) has been modified to adapt it for use in both the field and laboratory, and comparative results for 48 Indian soils are detailed.

E. HOLMES.

**Rapid determination of the nitrogen requirement of soils.** J. PÁZLER (Z. Zuckerind. Czechoslov., 1930, 55, 87—90).—The sample (100 g. of soil passing 2-mm. sieve) is shaken for 1 hr. with 1% potassium sulphate solution in a nearly filled 500-c.c. Stohmann flask. After filling to the mark, the suspension is filtered and 300 c.c. of the filtrate are used for nitrate determination by means of Devarda's alloy. Nitrate contents so determined agree closely with those by Mitscherlich's method for nitrogen-deficient soils. For richer soils

the Mitscherlich values are definitely higher. The presence of calcium carbonate in soils tends to increase the nitrate values recorded by the method described.

A. G. POLLARD.

**Determination of phosphate and potash requirements of soils by the citric acid method and Sigmond's nitric acid method.** W. JESSEN and W. LESCH (Z. Pflanz. Düng., 1930, 18A, 218—235).—None of the methods of Neubauer, of Lemmermann, or of Sigmond gives a sharp line of demarcation between normal and phosphate-deficient soils. Sigmond's method is simpler and more definite for soils rich in carbonate, but has no advantage for soils of low carbonate content. No relationship exists between the mechanical composition of soils and the solubility of their phosphate contents. All purely chemical methods give similar values for the potash requirement of soils, but in heavy soils these differ from the results of pot experiments and the seedling method. In clay soils rich in hydrochloric acid-soluble potash the assimilation of potash by plants is greater than the total exchangeable potash present, and in these soils chemical methods fail to give definite information as to assimilable potash contents.

A. G. POLLARD.

**Determination of the nutrient content and nutrient changes in soils by the citric acid method and other processes.** J. HASENBÄUMER and R. BALKS (Z. Pflanz. Düng., 1930, 9B, 456—473).—The nutrients removed from soil in four successive crops were much greater than the decrease in citric-soluble material in the soil. The loss of citric-soluble nutrients under these conditions approximated to the loss of water-soluble nutrients, and this general agreement was maintained in differently fertilised soils. The loss of citric-soluble phosphate and potash during cropping tended to become less than the loss of root-soluble material (Neubauer) as the clay content of the soils increased. The citric-soluble potash content of soil samples varied considerably with the distance from plant roots at which they were taken. Corresponding variations in the citric-soluble phosphate contents were small. In general, variations in the citric-soluble, water-soluble (Dirks and Scheffer), and root-soluble phosphates in soils were similar, but the values were not strictly proportional. Niklas' method (absorption of potash and phosphate by *Aspergillus*) is less sensitive to differences in soil treatment, but yielded values in general agreement with the above.

A. G. POLLARD.

**New microbiological method for determining the fertiliser requirement of soils.** H. NIKLAS, H. POSCHENRIEDER, and J. TRISCHLER (Z. Pflanz. Düng., 1930, 18A, 129—157).—Factors influencing the growth of *Aspergillus niger* in soil cultures are examined and a method is developed for determining the phosphate and potash requirement of soils by means of the weight of the mycelium produced. Addition of 1% of citric acid to the nutrient solution minimises the growth of other soil organisms and maintains a fairly constant  $p_H$  during the test. Chalk present in the soil is initially neutralised by excess of citric acid. Results obtained are closely in agreement with those of Neubauer tests.

A. G. POLLARD.



**Influence of different methods of making stall manure on the losses during stacking, and the utilisation of the manure by plants (with special reference to the Krantz hot-fermentation process).** R. SAILER (Landw. Versuchs-Stat., 1930, 111, 63—153).—Comparison is made of open-yard manure and hot- and cold-fermented pit manure. Losses of dry matter and nitrogen during rotting were greatest in yard manure both in summer and winter. Hot- and cold-fermented pit manures suffered similar losses in winter, but in summer the nitrogen loss during hot fermentation was the greater. In all cases summer losses were greater than the corresponding winter losses. Losses in hot-fermenting stacks occurred mainly in the early (loosely stacked) stage. Weed seeds lost their germinative power during hot fermentation, but remained viable in cold stacks. The quantity of drainage liquor was greatest in hot-fermenting stacks and less in yard manure, although the chemical composition of the liquor was practically the same in all cases. In field trials the efficiency of the manures was in the order yard manure < cold-fermented < hot-fermented. Hot-fermented manure showed no superiority in increasing the efficiency of artificial fertilisers. In sterile soils there was little or no utilisation of hot-fermented manure.

A. G. POLLARD.

**Effect of barnyard manure on a calcareous soil.** D. W. PITTMAN (J. Amer. Soc. Agron., 1930, 22, 549—552).—The amount of available phosphorus is largely influenced by the amount of manure applied. Low yield of sugar beet on unmanured soil is due to lack of soluble phosphorus in the early growing season as well as to lack of nitric nitrogen. Farm manure is therefore essential to sugar beet on highly calcareous soil.

CHEMICAL ABSTRACTS.

**Intake of nutrients by plants.** E. HASELHOFF, F. HAUN, and W. ELBERT (Landw. Versuchs-Stat., 1930, 111, 11—62).—The yields and mineral contents of a number of crops in various stages of growth in differently fertilised soils are recorded and discussed.

A. G. POLLARD.

**Dependence of the intake of nutrients [by plants] on the reaction of the substrate.** P. DAHM (Ber. deut. Bot. Ges., 1930, 48, 312—316).—No definite relationships were observed between the reaction of the substrate and the assimilation of nutrients in water or in pot cultures. The difficulties of maintaining constant  $p_H$  in nutrient solutions and of determining small quantities of the constituents are greater than are usually supposed. Under the conditions of the experiment lime was more important as a nutrient than as a neutralising agent.

A. G. POLLARD.

**Action of various nitrogenous fertilisers on acid soils.** D. MEYER (Z. Pflanz. Düng., 1930, 9B, 474).—The injurious effects of cyanamide on acid soils previously referred to (cf. Meyer and Obst, B., 1930, 208) are limited mainly to acid soils poor in humus and of low biological activity.

A. G. POLLARD.

**Interpretation of fertilising experiments with cyanamide.** E. J. PRANKE (Amer. Fertiliser, 1927, 67, No. 11, 60—66).—When calcium cyanamide is exposed to the air, 8% may be converted into dicyano-

diamide in 1 year; this form of nitrogen is, however, effective as a fertiliser if it does not exceed 10% of the total quantity present. The reaction of calcium cyanamide with superphosphate and with soil acids is also discussed.

CHEMICAL ABSTRACTS.

**Potash shale as a source of potash for growing plants.** H. C. HEATH (Bot. Gaz., 1930, 90, 121—150).—Applications of potash shale to soils in pot cultures increased the potash intake of a number of crops. Roasting the shale considerably increased its water-soluble potash content and the response of plants to its application. Roasting with calcium carbonate or gypsum was still more effective. Plants having nearly neutral sap benefited more from shale applications than those with more acid sap. Plant injury from excessive applications of lime to a peat and a sandy soil could not be ascribed to depressed assimilability of potash, but was due to reduced photosynthesis resulting possibly from the immobility of iron in the overlimed soil.

A. G. POLLARD.

**Grassland manuring.** G. T. GARRATT (J. Min. Agric., 1930, 37, 778—781).—Notes on the difficulties of rotational grazing of intensively manured pasture on Cambridgeshire Boulder Clay in a season of abnormally low rainfall are given.

E. HOLMES.

**Nutrition of seedlings and its influence on the rooting of cereals.** KLEEMANN (Z. Pflanz. Düng., 1930, 9B, 433—456).—Growth of cereals is largely influenced by the activity of enzymes in the seed prior to actual germination. These activities are stimulated and root development is subsequently improved by moistening the seeds (to 35—40% moisture content) and storing for 3 days in a saturated atmosphere at 17—18°. Still better results are obtained if respiration losses are minimised by storage of the seeds in a vacuum. Convenient alternative conditions of storage and of drying are recorded. Respiration losses during drying are reduced by working at low temperatures or in a vacuum. The improved primary root production and increased growth of treated seed result from the production of a supply of soluble food reserve, notably carbohydrate, by enzymic action.

A. G. POLLARD.

**Nitrate fertilisers for oats in Iowa.** R. A. PENDLETON (J. Amer. Soc. Agron., 1930, 32, 663—668).—Early additions of sodium nitrate produced a larger increase in yield than late applications, but the latter accentuated the increase of protein in the grain. Phosphate did not increase the protein content. Superphosphate without nitrate had little effect.

CHEMICAL ABSTRACTS.

**Fertiliser trials with magnesium salts for oats to determine the mode of action of magnesium.** W. DIX and S. BISCHOF (Z. Pflanz. Düng., 1930, 18A, 158—179).—Magnesium sulphate increased, and the oxide and carbonate decreased, the acidity of soil. In field trials, magnesium additions decreased the crop yields in the order sulphate > carbonate = oxide > lime. Analyses of crops from treated soils are recorded and the intake of calcium and magnesium and their translocation in various organs of the plant discussed in the light of their rôle in plant nutrition.

A. G. POLLARD.

**Seed treatment for controlling covered smut of barley.** R. W. LEUKEL (U.S. Dept. Agric., Tech. Bull. 207, 1930).—Various types of fungicides are compared. Dusting powders were effective where seeds were sown in soils of average moisture content (less than 25% saturation decreased the efficiency of the materials), and were not influenced by soil reaction or by temperature variations within normal ranges. Seed injury by dusts was less than that by liquid treatments.

A. G. POLLARD.

**Effect of superphosphates on the germination of maize.** C. O. ROST (J. Amer. Soc. Agron., 1930, 22, 498—507).—Large amounts of 46% superphosphate appeared to be more toxic than equivalent amounts of 16% superphosphate. Heavy applications retard germination less when they have been in intimate contact with the soil for several weeks before planting.

CHEMICAL ABSTRACTS.

**Concentration of certain constituents of the soil solution under orchard conditions.** E. L. PROBSTING (Hilgardia, 1930, 5, 35—59).—Seasonal variations in the sulphate, nitrate, calcium, and magnesium contents of the soil solution were observed, the actual values being influenced by soil treatment and intercropping and by the nature of the orchard trees. The concentration of potassium remained practically the same throughout the season. An excess of total cations over total anions existed in the soil solution. This was greater under peaches than under pears, and varied with the soil treatment.

A. G. POLLARD.

**Citrus rust mite and its control.** W. W. YOTHERS and A. C. MASON (U.S. Dept. Agric., Tech. Bull. 176, 1930).—Sulphur, which was the most effective agent examined, kills the rust mite by fumes arising from its oxidation. Actual contact with the insect is unnecessary.

A. G. POLLARD.

**Growth of certain conifers as influenced by different fertiliser treatments.** A. C. MCINTYRE and J. W. WHITE (J. Amer. Soc. Agron., 1930, 22, 558—567).—The favourable effect of fertilisation is described.

CHEMICAL ABSTRACTS.

**Indications that available nitrogen may be a limiting factor in hard winter wheat production.** P. L. GAINES and M. C. SEWELL (J. Amer. Soc. Agron., 1930, 22, 639—641).

**Phosphate rock grinding.** GABELER. **Superphosphate.** SHOJI and others. **Rhenania phosphate.** SCHARER. **Polysulphide sprays.** TUCKER. **Nitrogen-fixation plant.** WESTBY.—See VII. **Sunflower seed.** ANON.—See XII.

PATENT.

**Ammonium sulphate** (U.S.P. 1,758,449).—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Conductometric analysis in sugar factories.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1930, 55, 90—93).—A special conductometer is described and its use in the analysis of molasses etc. explained.

A. G. POLLARD.

**Carbonised molasses waste.** KILP.—See II. **Firing of boiler furnaces with bagasse fuel.** FREISE.

—See VIII. **Starch fermentation.** SYPNIEWSKI and others.—See XVIII. **Detection and determination of *p*-hydroxybenzoic acid.** WEISS.—See XIX.

PATENTS.

**Promotion of fermentation** (B.P. 335,972 and 336,207). **Lactic and acetic acids** (B.P. 335,596).—See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

**Fermentation phenomena of the *Nymphæa cocci* and their behaviour with regard to yeast.** J. GRÜSS (Woch. Brau., 1930, 47, 473—475).—The production of carbon dioxide by *Coccus zymophyllosepticus* from dextrose is greatly, and the decomposition of cellulose by a mixture of *C. phyllosepticus* and *C. zymophyllosepticus* is slightly, increased by the presence of calcium carbonate. The former action, in absence of calcium carbonate, is increased by aeration. In mixed culture yeast suppresses the activity of *C. zymophyllosepticus*, which does not increase the production of carbon dioxide by the yeast. In fermentations of dextrose in presence of sulphur the production of carbon dioxide by the coccus exceeds the production by yeast, which is much below that in normal fermentations; under these conditions the coccus produces more hydrogen sulphide than does yeast. The cocci become weaker in their power of hydrolysing cellulose when cultivated on artificial media. The production of carbon dioxide from dextrose varies inversely with that of hydrogen and methane.

F. E. DAY.

**Malt dust as a harmful factor in fermentation.** K. SCHUSTER (Woch. Brau., 1930, 47, 481—483).—Comparative laboratory fermentations of normal worts and of worts containing a proportion of extract from malt dust indicate that the latter are unsatisfactory, both as regards the course of fermentation and the quality of the yeast produced. The beers in bad cases are not quite bright and not easily cleared by filtration. The importance of "polishing" malt is stressed, and it is suggested that too fine grinding may give rise to similar effects by increased dissolution of husk constituents.

F. E. DAY.

**Continuous process of distillery-starch malt fermentation.** W. SYPNIEWSKI, A. JOSZT, and M. KUNIŃSKI (Przemysł Chem., 1930, 14, 392—405).—A laboratory apparatus is described for the continuous fermentation of rye- and potato-starch malts. A constant supply of sterile malt flows into the apparatus, which at the same time delivers fermented malt; yeast is added only at the beginning of the process. Yields of 52—54 litres of alcohol from 100 kg. of starch are readily obtained.

R. TRUSZKOWSKI.

**Simple universal thermostat for fermentation laboratories with arrangement for fermentation experiments, refractometry, and pycnometry.** H. FINK (Woch. Brau., 1930, 47, 461—464).—A rectangular water-bath of 100 litres capacity is heated by a flat, electrical heating element. This is controlled through a relay by an Ostwald-Luther or other thermoregulator, and the temperature, equalised by a

motor-driven stirrer, can be maintained constant to  $0.2^{\circ}$ . The same motor actuates a rack for holding fermentation vessels, the speed and motion of which can be varied. A stand attached to the length of the bath carries a number of gas burettes connected by flexible capillaries to the fermentation vessels. The shaking rack is readily removable to allow special stands for the immersion refractometer or a number of pyknometers to be fitted into the bath. F. E. DAY.

**Influence of the distilling apparatus and of other factors on the quality of spirit.** T. CHRZASZCZ and J. RESZETNIK (Przemysl Chem., 1930, 14, 415—429, 437—448).—The quality and potability of potato spirit obtained in 132 distilleries have been studied. A satisfactory product is obtainable only when sound potatoes are used and infection of the malt is avoided. The spirit obtained from abnormal fermentation, characterised by production of hydrogen sulphide, foam, etc., is invariably of poor quality. The greater the concentration of alcohol obtained from a given rectifier, the purer is the product. Only continuous-process apparatus should be used; periodic and two-column stills give inferior products. The material used for the construction of stills is of minor importance. The potability of spirits depends chiefly on the absence of furfuraldehyde and other aldehydes and acids. The decolorisation of permanganate by rectified spirits is due chiefly to furfuraldehyde. R. TRUSZKOWSKI.

**Determination of carbon dioxide in beer.** J. BLOM and B. KRAUSE (Woch. Brau., 1930, 47, 471—473).—To well-cooled beer 50% caustic soda solution is introduced, if possible by a special piercing pipette and compressed air. The weight of the beer is determined before and after the introduction of the soda solution. Then 20 g. of the alkaline beer are weighed into a 50-c.c. wide-mouthed flask provided with an inlet tube through which acid and air free from carbon dioxide can be drawn to the bottom of the flask. An outlet tube, provided with a small cotton-wool filter, connects with two 200-c.c. flasks in series, containing, respectively, 50 c.c. and 10 c.c. of 0.1*N*-baryta. These have splash-traps, and are shaken continuously during the absorption. 20 C.c. of 25% sulphuric acid are introduced into the 50-c.c. flask and air is drawn through the apparatus, slowly at first. After about 10 min. the air current is increased, and 15 min. later the apparatus is disconnected and the excess of baryta titrated with 0.1*N*-hydrochloric acid. Alternatively, the carbon dioxide may be absorbed in caustic potash solution and weighed.

F. E. DAY.

**Sulphurous and lactic acids in the production of fruit wines.** RÜDIGER and K. SICHERT (Wein u. Rebe, 1930, 11, 560—568; Chem. Zentr., 1930, i, 3495).—The addition of lactic acid (2—6 g. per litre) to perry prepared from pears low in acid is recommended in place of that of sulphurous acid. A. A. ELDRIDGE.

**Colorimetric method for the determination of the amount of potassium ferrocyanide required for the clarification [of wine].** A. HANAK (Z. Unters. Lebensm., 1930, 59, 506—511).—A method is described for the rapid determination of iron in wine. Into each

of two similar tubes (*A* the test sample, *B* the standard) are measured 25 c.c. of wine, and to one sample, *A*, are added a few drops of ferric chloride solution (0.05%). A measured volume of potassium ferrocyanide solution (0.5%) is now added to each tube, followed by 1—2 drops of hydrogen peroxide (30%) or dilute nitric acid. The average amount of ferrocyanide solution required is 0.6 c.c., but if, after the addition of peroxide, the colour in *B* be not more intense than that in *A*, insufficient has been added, and the process must be commenced afresh, using 0.8 c.c. Some wines may require 1 c.c. or more. The sample *B* is now diluted with water or a dilute solution of yellow dye to colour equality with *A*. The amount of ferrocyanide required to clarify *A* may now be derived from the amount of solution used, and the ratio of the resultant volumes when colour equality is reached. The addition of gum arabic to the wine helps to prevent flocculation of the Prussian-blue. H. J. DOWDEN.

**Use of the quartz lamp in wine analysis.** A. HEIDUSCHKA and E. MÖHLAU (Pharm. Zentr., 1930, 71, 689—691).—Provided the wine is decolorised by animal charcoal, the adulteration of normal wine by raisin or currant wine is detected by the blue to violet luminescence which the latter shows under the quartz lamp, and which is not given by natural wine. The glycerin-water mixtures which were formerly used as standards for the determination of the intensity of luminescence have been replaced by ethyl-alcoholic solutions of sodium saccharin. The latter standards can be prepared very accurately by weighing, are not hygroscopic, and show no change in luminescence after 4 months.

C. RANKEN.

**Amylase and catalase reactions of milk.** WEINSTEIN. **Diastatic activity of honey.** LAMPITT and others.—See XIX.

## PATENTS.

**Promotion of fermentation.** DARCO SALES CORP. (B.P. 335,972 and 336,207, 3.7.29. U.S., [A] 29.4.29, [B] 2.4.29).—(A) An improved yield of fermentation products is obtained by fermenting a molasses solution of medium sp. gr., which is treated as in (B), and raising later the sp. gr. of the partly fermented solution above that of the initial solution by the addition of a molasses solution of high sp. gr. (B) The time of fermentation is shortened and a purer and greater yield of alcohol is obtained by the addition to the fermenting solution of an organic nitrogenous substance and an actively adsorbent material such as vegetable char. A molasses solution of greater sp. gr. than is ordinarily possible can thus be fermented. C. RANKEN.

**Production of lactic and acetic acids [by fermentation].** W. W. TRIGGS. From WISCONSIN ALUMNI RES. FOUNDATION (B.P. 335,596, 20.6.29).—Pentoses and/or hexoses, e.g., hydrolysed wood, corn-cobs, straw, molasses, etc., are fermented by means of a new ferment (morphology etc. given) isolated by known methods from fermenting silage or sauerkraut. The enzyme grows best at  $28-30^{\circ}$  and at  $pH$  6.5, and is anaerobic. It converts pentoses into acetic and lactic acids, hexoses into lactic acid, without production of gas or ethyl alcohol. C. HOLLINS.

**[Microbiological] preparation of 2 : 3[ $\beta$ ]-butylene glycol from carbohydrates.** T. H. VERHAVE, SEN. (B.P. 335,280, 26.6.29. Addn. to B.P. 315,263; B., 1930, 1088).—After a first fermentation as described in the prior patent, the alcohol is removed by distillation, and the residue added to a second batch or used as solvent, in place of water, for the next carbohydrate mash. Preferably air is blown through the fermenting mash and alcohol recovered from the exit gases. The presence of  $\beta$ -butylene glycol does not retard its formation in the succeeding batches, and relatively high glycol concentrations are reached. C. HOLLINS.

## XIX.—FOODS.

**Qualities of combined wheats as affected by type of bin, moisture, and temperature conditions.** I. F. C. FENTON and C. O. SWANSON (Cereal Chem., 1930, 7, 428—448).—Details are given of the variation in moisture content of wheat during harvesting, also the effect of the amount of moisture on the keeping quality of the wheat, 15% of moisture being considered the maximum safe amount. Wheats of higher moisture content are improved in keeping quality by moving by means of compressed air. E. B. HUGHES.

**Variations in properties of acetone extracts of common and durum wheat flours.** A. W. WALDE and C. E. MANGELS (Cereal Chem., 1930, 7, 480—486).—Comparison of acetone extracts of Marquis and durum flours showed the former to contain a sterol, probably an ester of sitosterol, which was not present in the durum flour. Acetone extract of flour contains little phosphatide, but a relatively large amount of unsaponifiable matter. E. B. HUGHES.

**Proteolysis in bread doughs.** W. E. BROWNLEE and C. H. BAILEY (Cereal Chem., 1930, 7, 487—517).—In sampling the doughs, quick freezing is followed by grinding and acetone dehydration. Several methods of detecting change in the composition of the proteins have been used, including peptisation by salt solutions, precipitation of the primary proteins by heavy-metal salts, measurement of viscosity of the water extract, and fractional analysis of a water extract of the protein. No alteration in the structure of the gluten was detected during the process of fermentation. E. B. HUGHES.

**Test for ropiness in bread.** H. H. BUNZELL and M. FORBES (Cereal Chem., 1930, 7, 465—472).—The method depends on the measurement of the catalase activity in the bread. A totally enclosed apparatus is described in which a solution of hydrogen peroxide is added to a water suspension of the bread, and the evolved oxygen measured. In applying the method to flour, a biscuit is made from flour, water, and baking-powder and the product is baked.

E. B. HUGHES.

**Gluten and non-gluten proteins.** M. J. BLISH (Cereal Chem., 1930, 7, 421—427).—Methods used for the characterisation of the flour proteins involve processes which probably alter the structure. Gliadin is the only protein of which the chemical individuality has been definitely established. The ratio of gliadin to total

protein is fairly constant, but the ratio of gluten to non-gluten proteins varies with different flours.

E. B. HUGHES.

**Normal and modified foaming properties of whey-protein and egg-albumin solutions.** P. N. PETER and R. W. BELL (Ind. Eng. Chem., 1930, 22, 1124—1128).—Whey-proteins have good foaming and emulsifying properties. The best conditions for the utilisation of these properties are given, and as whey-protein is cheaper and has an excellent food value its use in place of egg products is suggested. It can replace egg-white or -yolk in those uses which are dependent on whipping or emulsifying properties, such as in salad dressings, cake icing, ice cream, etc., but it cannot replace eggs in uses which are dependent on heat-coagulative properties such as baked or cooked products.

B. W. TOWN.

**Wheat-protein test digestion studies.** C. F. DAVIS (Cereal Chem., 1930, 7, 518—525).—When using a "high heat" electric heater, capable of evaporating 210 c.c. of water from 300 c.c. in 20 min., the Kjeldahl method is quicker than the Gunning modification.

E. B. HUGHES.

**Gasometric determination of carbon dioxide in baking powder.** J. R. CHITTICK, F. L. DUNLAP, and G. D. RICHARDS (Cereal Chem., 1930, 7, 473—480; cf. Hertwig and Hicks, B. 1929, 187).—The A.O.A.C. gasometric method (cf. J. Assoc. Off. Agric. Chem., 1927, 10, 36) gives concordant and accurate results. Although no correction is applied for the vapour tension of the reaction mixture, this is balanced (at ordinary temperatures) by the carbon dioxide dissolved in the liquid.

E. B. HUGHES.

**Biuret reaction in cereal chemistry.** H. KÜHL (Z. ges. Getreidewesen, 1930, 17, 28—32, 67—70; Chem. Zentr., 1930, i, 3496).—The extract (50 : 1000, 94 c.c.) is treated with 0.1N-sodium hydroxide (6 c.c.), heated at 50°, and then treated with 10 drops of 2% copper sulphate solution, stirring meanwhile. The colour is observed before and after the solution is boiled. The test can be used to determine the age of flour. The reaction is not appreciably affected by maltose or diastase.

A. A. ELDRIDGE.

**Influence of the fat content on the keeping quality of milk.** H. BARKWORTH (J. Min. Agric., 1930, 37, 803—806).—An examination of the results of tests of 4000 samples of milk, extending over 6 years, indicates that if rich milks are considered in terms of butter fat content there is no commercial difference in keeping quality between poor and rich milks. E. HOLMES.

**Catalase test [of milk].** G. ROEDER (Milch. Forsch., 1930, 9, 516—590; Chem. Zentr., 1930, i, 3371).—In Lobeck's test shaking causes no variation in the apparent catalase value, but at high hydrogen-ion concentration the action of the enzyme is considerably retarded. The catalase is more active when present in high concentration and less active when that of the hydrogen peroxide is high.

A. A. ELDRIDGE.

**Catalase reaction of milk.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 514—515).—Milk which had

been treated by the holding process was accidentally contaminated with raw milk and a considerable rise in the catalase value was caused thereby (cf. B., 1929, 374).  
H. J. DOWDEN.

**Amylase detection in milk.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 513—514).—The earlier work on the determination of the degree of heating to which milk has been subjected (B., 1929, 374) has been extended to cover the flash treatment at 85°. It is imperative to use pure unswollen starch grains, preferably potato starch. The test works satisfactorily with sour milk and the temperature limits are 35—40°.

H. J. DOWDEN.

**[Detection of] artificial whole-milk powder.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 515—516).—The powder was observed to contain a relatively high proportion of large fat globules, and the characteristics of the isolated fat suggested adulteration. Extraction with light petroleum gave a yield of 18% of fat (refractometer reading at 25° 62·8°, at 40° 53·0°), whereas normal whole-milk powder gave only 1% (reading at 40° 43·3°). Examination of the fat proved that the powder had been prepared from skim-milk powder with the addition of 17—18% of olive oil.

H. J. DOWDEN.

**Watering of buttermilk.** G. KEMPINSKI (Milch. Forsch., 1930, 9, 506—510; Chem. Zentr., 1930, i, 3371).—Owing to the progressive decomposition of lactose with increase in acidity the density of the serum diminishes and cannot therefore be employed to determine the degree of watering; the ash content is, however, constant.

A. A. ELDRIDGE.

**Influence of pasteurisation on the conservation of butter.** L. PIK (Chem. Listy, 1930, 24, 396—398).—Pasteurisation of buttermilk at 85—95° leads to coagulation of lactalbumin, as a result of which the rate of development of bacteria in the pasteurised product is retarded.

R. TRUSZKOWSKI.

**Colloid chemistry of butter.** "Histology of technical substances." N. KING (Kolloid-Z., 1930, 52, 319—332).—A considerable amount of previously published literature on butter is reviewed from the colloid-chemical point of view, particularly in regard to the relation between structure and physical properties, and chemical and bacteriological changes. Butter is discussed as an emulsion of the fat-in-water type and also as the water-in-fat type, and a new view of its structure is advanced, according to which butter is a disperse system of water droplets and fat globules in liquid fat, the fat thus appearing in two forms. The fatty globules are complex in structure, being composed of an adsorbed layer of protein and an inner layer of oriented crystallites of the fat. The structure, composition, and reactions of the aqueous phase are discussed and also the distribution of bacteria. An account is given of the rôles played by the water-fat and air-fat boundaries in the chemical and bacteriological changes and of their effects on the flavour and odour of the butter.

E. S. HEDGES.

**Masking of the colour reaction for margarine.** B. HEPNER and S. ZALC (Przemysl Chem., 1930, 14, 412—415).—Baudouin's furfuraldehyde reaction is not given

by sesamé oil previously treated by acids or subjected to temperatures above 180°; this applies also to margarine heated above 180° after addition of crude sesamé oil. The development of rancidity in sesamé oil leads to the production of a green coloration with hydrochloric acid alone; this may mask the red coloration due to Baudouin's reaction. When phenolic dyes, such as Sudan Yellow, have been added to the margarine, these should be removed by extraction with potassium hydroxide solution before applying the test. In rancid margarine the Baudouin reaction may fail to give positive results even though the product originally answered to the test.  
R. TRUSZKOWSKI.

**Oil absorption of shell eggs.** T. L. SWENSON and H. H. MORTERN (Science, 1930, 72, 98).—Eggs dipped in oil at the ordinary and under reduced pressure show a diminution in the loss of weight during storage. Penetration is marked around the air cells.

L. S. THEOBALD.

**Composition of meal from pressed and dried potatoes.** B. LAMPE (Z. Spiritusind., 1930, 53, 298).—Pulped potatoes from which the juice has been expressed are dried below 65° till the moisture content of 60% is reduced to 20%. By drying for approx. 6 hrs. at not above 100°, a final moisture content of 10—11% is obtained. The dried potatoes rapidly crumble in water, show an intact starch grain, and contain, if the potatoes are new, 1·49 and 3·65% of ash and protein, respectively, calc. on dry matter. For old potatoes the corresponding values are 2·04 and 4·38, and approximate to 40—50% of those obtained from flakes made from unpressed potatoes.

C. RANKEN.

**Composition of European and Californian almonds.** C. V. HART (Ind. Eng. Chem., 1930, 22, 1128—1129).—Analyses are recorded. No significant difference in composition was detectable. B. W. TOWN.

**Further comparison of Californian and imported almonds.** G. PITMAN (Ind. Eng. Chem., 1930, 22, 1129—1131).—Californian almonds show a somewhat greater shearing stress than imported varieties, which could not be accounted for by differences in hemicellulose content. Almonds from different districts of California had practically the same composition.

B. W. TOWN.

**Do fu : an oriental food.** J. S. HEPBURN and K. S. SOHN (Amer. J. Pharm., 1930, 102, 570).—This preparation of the soya bean contains total solids 13·4, moisture 86·6, ether extract 4·65, crude protein 4·2, ash 0·2, and nitrogen-free extractives 3·91%.

H. E. F. NOTTON.

**Nutritive values of some typical Indian hays.** F. J. WARTH (Mem. Dept. Agric. India, 1930, 11, 73—84).—The composition and digestibility of 30 typical Indian hays are recorded. The protein content varied between 18 and 2%, and these figures were found to be correlated with the percentages of soluble ash, and to decrease in similar samples at increased maturity. The amount of digestible protein in the samples decreased more rapidly than the total amount of protein; samples with less than 2·5% of total protein contributed practically no digestible protein to the diet. E. HOLMES.

**Nutritive value of gram husk.** F. J. WARTH and L. C. SIKKA (Mem. Dept. Agric. India, 1930, 11, 85—99).—Feeding tests on calves indicated that gram husk is comparable with wheat bran as a feeding-stuff. It has a distinctly lower starch-equivalent value than wheat bran, and provides no digestible protein; on the contrary, it has a depressing effect on the digestibility of the protein in the rest of the ration. Despite this, gram husk must be considered a concentrated food of merit. Observations are appended regarding European and American figures for starch-equivalent values.

E. HOLMES.

**Vitamin-D and the antirachitic activation of foods by irradiation with ultra-violet light.** F. L. GUNDERSON (Cereal Chem., 1930, 7, 449—455).—An outline of the known relationship which exists between occurrence of rickets, vitamin-D, and irradiation of foodstuffs, with special reference to cereals. Antirachitic potency of irradiated foods is stable during normal storage and household cooking, but may be partly destroyed at the higher temperatures of commercial baking.

E. B. HUGHES.

**Microscopy and histo-chemistry of certain fruits [citrus fruits, pears, plums, bananas, and tomatoes].** A. NIETHAMMER (Z. Unters. Lebensm., 1930, 59, 501—506).—The endocarp of oranges contains crystals of calcium oxalate, which can be isolated and identified by treatment of the fruit with phosphoric acid followed by sublimation under reduced pressure. After storage for several weeks, slow resorption of the crystals occurs with the concomitant formation of acetaldehyde. Lemons and mandarins exhibit identical phenomena. Pears contain characteristic inclusions, and in the ripe condition acetaldehyde can always be detected, the amount increasing considerably in the mellow and over-ripe states. Ripe pears on sublimation give malic and citric acids, whilst over-ripe samples give oxalic acid in addition. In plums the acetaldehyde test is always positive, and calcium oxalate can be detected. From bananas in the ripening stage, malic acid can be sublimed, but after prolonged storage traces only of oxalic and tartaric acids can be detected. Tomatoes in the unripe state contain appreciable amounts of malic acid with traces of oxalic and tartaric acids, whilst ripe samples contain malic, citric, and tartaric acids. Acetaldehyde is present in ripe tomatoes only.

H. J. DOWDEN.

**[Colorimetric] determination of copper in green vegetables.** A. HANAK (Z. Unters. Lebensm., 1930, 59, 511—512).—The sample (10 g.) of finely-ground material is treated with 20—30 drops of sulphuric acid and ignited, and the residue is then boiled with dilute (1:5) nitric acid and filtered. The filter and its contents are ashed and the ash is dissolved in the warmed filtrate. The solution is treated with ammonia in excess followed by ammonium carbonate and is then filtered. The residue is washed, redissolved, treated further with ammonia, and filtered. The united filtrates, if highly coloured, are compared against a solution of ammoniacal copper sulphate of known concentration. If only small amounts of copper are present, a dilute solution of gum arabic containing acetic acid and a few drops of potassium

ferrocyanide solution is used. The results obtained with peas, beans, and spinach of known copper content were very satisfactory.

H. J. DOWDEN.

**Diastatic activity of honey.** L. H. LAMPITT, E. B. HUGHES, and H. S. ROOKE (Analyst, 1930, 55, 666—672).—The dextrinogen-amylase activity of honey at various  $p_H$  values does not differ from that of most enzymes, whilst the optimum acidity was found to be at  $p_H$  5.0—5.3 whatever the temperature. A reaction period of 16 hrs. is adopted as a standard for the determination of the saccharogen-amylase activity, since the increase of reducing sugars is very slow after that period and the optimum  $p_H$  is about 5.3. At a  $p_H$  value below 4.0 and above 9.0 both the enzymes are destroyed, but at  $p_H$  4—6 the dextrinogenic activity seems to be stable. Measurements of diastatic activity are not reliable for detecting adulteration or heating of honey.

D. G. HEWER.

**Detection of carrots in marmalade.** A. HANAK (Z. Unters. Lebensm., 1930, 59, 513).—A portion of marmalade is stirred with ether, and if carrots be present the liquid acquires the characteristic colour of carotene. If the supernatant liquid is decanted and allowed to evaporate spontaneously, concentric rings are formed in which the presence of carotene may be verified. Conclusive proof must be found by microscopical examination of the original materials.

H. J. DOWDEN.

**Detection and determination of *p*-hydroxybenzoic acid and its esters in foodstuffs.** F. WEISS (Z. Unters. Lebensm., 1930, 59, 472—480).—A study has been made of the methyl, ethyl, and propyl esters and their sodium derivatives, which have recently been employed as preservatives for foods. Isolation of the preservative is effected by methods previously described (B., 1928, 386). The methyl ester may be identified by Denigès' method, and the ethyl and propyl esters by alkaline hydrolysis and distillation, followed by Griebel's micro-beaker method (cf. B., 1924, 922; 1925, 470). The alkaline residue is acidified, extracted with ether, and, after removal of the solvent, *p*-hydroxybenzoic acid may be identified in the residue by its m.p. (215°), by testing with Millon's reagent, and by the formation of the crystalline blue copper derivative. Salicylic and *o*- and *p*-chlorobenzoic acids do not interfere, but vanillin must be removed by warming with semioxamide. In the presence of esters etc. the free acid is best removed as the copper salt, or, alternatively, the other materials may first be removed by carbon tetrachloride, in which *p*-hydroxybenzoic acid is sparingly soluble, or by steam distillation. The acid may be determined by Zeisel's method, vanillin, if present, being removed by semioxamide. Examples are given of the application of the methods to sugar solution, raspberry juice, margarine, and mayonnaise, 60—100% of the *p*-hydroxybenzoic acid being recovered.

H. J. DOWDEN.

**Determination of fat in chocolate.** C. E. WISEMAN (Analyst, 1930, 55, 684—685).—The Gottlieb method for determining fat in condensed milk may be adapted to chocolate or cocoa by immersing the aqueous ammonia suspension of the material in boiling water

for 5 min., and subsequently adding rather more than the usual proportion of alcohol. D. G. HEWER.

**Determination of crude fibre [cellulose] in cacao.** K. KÜRSCHNER and A. HANAK (Z. Unters. Lebensm., 1930, 59, 484—494).—The method devised previously for the determination of cellulose in wood has been modified by the substitution of acetic acid for alcohol in the mixture used for decomposing the non-cellulosic constituents (cf. Kürschner and Hoffer, B., 1930, 608). The sample of defatted cacao (0.3 g.) is intimately mixed with 15 c.c. of 80% acetic acid and 1.5 c.c. of nitric acid ( $d$  1.4) in a flask fitted with a ground-in air-condenser. The mixture is gently boiled for 15—25 min. and then filtered through a large-pored porcelain or glass filter previously moistened with acetic acid. The residue is washed successively with 7—10 c.c. of the hot mixture of nitric and acetic acids, hot water, a few drops of alcohol, 5—10 c.c. of ether, 1—2 c.c. of the acid mixture, and finally hot water, until all traces of acetic acid have been removed, the flask and walls of the filter being washed at each stage. The residue is dried at 105—108° and weighed. By this method, which gives most consistent results, white undegraded cellulose was extracted from cacao and the determination required less than 1 hr. to make.

H. J. DOWDEN.

**Analysis of chocolate. II. Adulterated chocolate.** R. LECOQ (J. Pharm. Chim., 1930, [viii], 12, 149—159).—If the sum of the total sugar and thrice the insoluble matter other than fat exceeds 100% adulteration with cacao husks and germs, gluten flour, starch, etc. should be suspected. Addition of coffee or of nut paste must be detected microscopically or in the latter case by determining the iodine value of the fat. If  $x$  is the content of cacao butter and  $y$  that of nut oil, then  $x + y$  = fat content of the sample of chocolate. Also the iodine value of cacao butter being 36, and that of the nut oil being denoted by  $I$ , then  $36x + Iy$  = iodine value of the fat of the sample  $\times$  fat content of the sample. From these equations the value of  $y$  and, the fat content of the nuts being known, the proportion of nuts in the sample may be calculated. The fat content and iodine value of various nuts and nut oils are given. With mixtures of different nuts the problem is practically insoluble. In the case of milk chocolate of known components, the milk content may be found approximately by examination of the fat or by determination of the casein, but if nuts, honey, glucose, or other admixture is present it is impossible to form reliable conclusions as to the composition.

W. J. BOYD.

**Determination of crude fibre.** FEIST and KUNTZ. —See V. "Dry-ice." KILLEFFER. —See VII. Firing of boiler furnace with coffee-pod fuel. FREISE. —See VIII. Determination of butter fat. SCHWEIZER and GROSSFELD. Tung oil seeds. Sunflower seed. Dhupa kernels. *Moringa aptera* seed. ANON. Cherry-kernel oil. JAMIESON and GERTLER. —See XII.

#### PATENTS.

**Preparing green-coloured preserved vegetables.** P. DE BACKER (B.P. 337,354, 27.7.29).—Vegetables which would otherwise turn a yellowish colour during

processing may be kept a bright green by the addition of a harmless blue dye. Methylene-blue, water-blue 6B, indigo-carmin, Lyon-blue, and indanthrene-blue are suitable. E. B. HUGHES.

**Dehydration of vegetable substances and products of organic character.** B. J. OWEN, Assr. to SUGAR BEET & CROP DRIERS, LTD. (U.S.P. 1,781,473, 11.11.30. Appl., 27.11.26, U.K., 7.12.25). —See B.P. 267,203; B., 1927, 377.

**Sterilisation of liquids** (B.P. 337,027). —See I. **Alimentary salt** (B.P. 319,203 and 336,279). —See VII. **Extraction of fatty oil** (B.P. 336,273—4). **Edible oils** (U.S.P. 1,749,976). —See XII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Analysis of Liquor aluminii acetico-tartarici and of Liquor aluminii acetici, D.A.B. VI.** R. HOLDERMANN (Pharm. Ztg., 1930, 75, 1331—1332).—The amount of aluminium acetotartrate in a sample of the former determined by evaporation ( $a$ ) at 100° (Official method); ( $b$ ) at 45° in a vacuum, or by precipitation of the aluminium: ( $c$ ) as hydroxide; ( $d$ ) as its 8-hydroxyquinoline derivative, was ( $a$ ) 44%, ( $b$ ) 48.1%, ( $c$ ) 53.3%, and ( $d$ ) 54.7%. The last result is in agreement with the method of preparation and the low values obtained in ( $a$ ) and ( $b$ ) are due to the formation of varying quantities of the basic salt,  $Al_2O(OAc)_2 \cdot C_4H_4O_6$ , during evaporation. This may also be present in the original solution. Liquor aluminii acetici is also conveniently standardised by means of 8-hydroxyquinoline.

H. E. F. NOTTON.

**Determination of potassium guaiacolsulphonate in syrups.** W. SCHÖBEL (Pharm. Ztg., 1930, 75, 1232—1233).—Previous methods (cf. D.A.B. VI; Rupp, B., 1928, 586; Rojahn and Struffmann, B., 1927, 617) are inconvenient and inaccurate. The proportions of potassium guaiacol-4-sulphonate (anhydrous), -5-sulphonate ( $+2H_2O$ ), and of the corresponding dipotassium salts ( $+2H_2O$ ) may be calculated from the alkalinity and water content of commercial samples, and official upper limits should be imposed for these values. The sulphur content, determined as sulphate after oxidation with permanganate, agrees with that obtained by the above method and remains practically constant ( $37.8 \pm 1\%$   $SO_4$ ) although the proportions of the three constituents vary widely. Syrups containing potassium guaiacolsulphonate are defecated with lead acetate and sodium phosphate, which, contrary to the statement of Rupp, do not precipitate the sulphonate, and the sulphur is determined in the above manner. The amount of drug is calculated from the mean sulphur content.

H. E. F. NOTTON.

**Determination of arsenic in pharmaceutical mixtures.** G. WEISSMANN and S. BABITSCH (Pharm. Zentr., 1930, 71, 721—724).—Several methods are reviewed (e.g., Rupp and Lehmann, A., 1912, ii, 866; Fridli, A., 1926, 591; and others) and the following, based on that of Andrews (A., 1909, ii, 637; 1914, ii, 291; 1915; ii, 279), is recommended. Liquid mixtures are evaporated to dryness, an amount containing 0.005—0.05 g. As being taken; the dry residue (or pulverised material in the case of pills etc.) is triturated with 20%



sulphuric acid, then treated dropwise with sulphuric acid and "perhydrol" until colourless. After dilution, removal of excess of hydrogen peroxide by permanganate, and decolorisation by oxalic acid, the colourless liquid is treated with 15% aqueous caustic soda and reduced with Thiele's hypophosphite solution (D.A.B. VI., 771) for 15 min., and set aside for 1 hr. The precipitated arsenic is collected, washed, and transferred to a stoppered flask; after addition of sodium carbonate, 0.1*N*-iodine is run in and the mixture shaken until the arsenic has completely dissolved, when excess of iodine is back-titrated with thiosulphate. Preparations containing iron are treated as above, except that stannous chloride is added to the mixture immediately prior to the addition of Thiele's reagent (cf. Wallrabe, B., 1928, 172). The precipitated arsenic may alternatively be oxidised by hydrogen peroxide (as above), dissolved in 25% hydrochloric acid and potassium iodide, and the free iodine in the diluted solution titrated with thiosulphate. Good agreement is shown by the two methods for such mixtures as Fowler's solution, Bland's pills, etc.

R. CHILD.

**Tinctures of cinchona. I. Preliminary report.** A. LICHTIN (Amer. J. Pharm., 1930, 102, 583—590).—The sedimentation observed in old samples of the U.S.P. X tincture also occurs to a smaller extent in the improved tinctures of Scoville (B., 1928, 139). It is not prevented by the addition of small quantities of acid, alkali, or oxidising or reducing agents, nor by refrigeration nor the use of special bottles. After a preliminary extraction of the bark with acetone, which removes tannins but practically none of the alkaloidal matter, a tincture prepared by the official method shows only a slight sediment after 3 months, whilst one obtained by percolation with 78% alcohol with subsequent addition of 7.5% of glycerin gives no sediment. H. E. F. NOTTON.

**Use of the centrifuge in determining morphine in opium.** C. STICH (Pharm. Ztg., 1930, 75, 1233—1234).—The precipitate of morphine obtained by the method of the Swiss pharmacopoeia is separated more rapidly and completely from the solution and wash waters by centrifugation than by filtration, and its greater purity renders the subsequent titration more precise.

H. E. F. NOTTON.

**Evaluation of drugs containing caffeine. II. Guarana and kola.** G. VON MIKÓ (Magyar Gyó Társ. Ert., 1930, 6, 30—42; Chem. Zentr., 1930, i, 2286).—A rapid macro- and a refractometric micro-method of evaluation, similar to those employed for tea (B., 1930, 585), are described. The micro-Kjeldahl method is applicable also.

A. A. ELDRIDGE.

**Evaluation of alkanet root: a colorimetric test.** W. A. N. MARKWELL and L. J. WALKER (Pharm. J., 1930, 125, 429).—The sample (5 g.) in No. 20 Powder is exhausted by continuous extraction in a Bolton-Revis apparatus with a mixture of 90% methylated spirit (50 c.c.) and benzene (50 c.c.). The extract is transferred to a 200-c.c. measuring flask, washings are added, and the volume is made up by addition of the mixed solvent. 2 C.c. of the resulting solution are diluted with solvent to 50 c.c. in a Nessler flask, when the colour should be equal in depth to that shown by a solution

prepared by mixing 40 c.c. of 0.01% (w./v.) aqueous potassium permanganate and 10 c.c. of 0.1% (w./v.) aqueous potassium dichromate (both of the latter being freshly made up).

R. CHILD.

**Steam-air method of treating tobacco.** P. K. DOROXOV, I. I. LOSEV, A. O. REPPICH, and A. A. SCHMUCK (U.S.S.R. State Inst. Tobacco Inv., 1930, Bull. 68, 36 pp.).—By the method described, which consists in treating the leaves with steam and air, and effects uniform moistening and sorting, the time occupied is reduced to a few minutes, all the conveying of the tobacco being by mechanical devices. At the same time the nicotine (recovered as sulphate), ammonia, and methyl alcohol are considerably diminished in amount and the flavour is improved. The process has been tested on a semi-manufacturing scale with satisfactory results.

T. H. POPE.

**Chemical composition of the [Russian] tobacco crops of 1927 and 1928.** V. BALABUCHA-POPCOVA and V. ZAPOLSKI (U.S.S.R. State Inst. Tobacco Inv., 1930, Bull. 66, 52 pp.).—A large number of data for tobaccos grown in different parts of Russia are given.

T. H. POPE.

**Orange oils from South Africa.** ANON. (Bull. Imp. Inst., 1930, 28, 282—284).—Three samples of orange oil, prepared in Pretoria, had the following respective constants:  $d_{15}^{20}$  0.848, 0.849, 0.849;  $\alpha_D^{20}$  +99.25°, +96.12°, +97.17°;  $n_D^{20}$  1.4735, 1.4735, 1.4730; non-volatile residue (at 100°) 2.3%, 3.6%, 2.9%.

E. H. SHARPLES.

**Petitgrain oil from Tanganyika.** ANON. (Bull. Imp. Inst., 1930, 28, 281—282).—A sample of the oil distilled in Tanganyika had  $d_{15}^{20}$  0.8957,  $\alpha_D^{15}$  —5.25°,  $n_D^{20}$  1.460, acid value 0.7, ester value 159.4, esters (as linalyl acetate) 55.8%, and gave a turbid solution even with 12 vols. of 80% alcohol at 15°. It compares favourably with Paraguayan oil.

E. H. SHARPLES.

**Ultra-violet window glazing.** BECKETT.—See VIII. **Cherry-kernel oil.** JAMIESON and GERTLER. **Moringa aptera seed.** ANON.—See XII. **Antirachitic activation of foods.** GUNDERSON.—See XIX.

## PATENTS.

**Manufacture of [pharmaceutical] complex iron compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,965, 5.6.29).—A polyhydroxylated mono- or di-carboxylic acid is treated with iron or an iron compound, the mixture being neutralised simultaneously or subsequently with an alkali or organic base. Complex salts are prepared from sodium gluconate and ferric acetate or chloride; from potassium hydrogen saccharate and ferric chloride or ferric ammonium sulphate, with or without quinine; from pure iron, gluconic acid, and sodium hydroxide; from diethylamine mucate, ferric chloride, and sodium hydroxide.

C. HOLLINS.

**Manufacture of non-irritant organic arsenic compound capable of being injected.** I. G. FARBENIND. A.-G. (B.P. 318,835, 9.9.29. Ger., 7.9.28).—The bis-formaldehyde-bisulphite compound of arseno-(4-amino-1-phenyl-2 : 3-dimethyl-5-pyrazolone) is

treated with an alkaline-reacting solution, *e.g.*, aqueous sodium sulphite, so that the product precipitated by addition of alcohol and ether has in 1% aqueous solution  $p_H$  above 4 (*e.g.*, 4.5). C. HOLLINS.

**Purification, and manufacture, of hormone preparations.** SCHERING-KAHLBAUM A.-G. (B.P. 336,470—1, [A] 13.11.29, [B] 14.11.29. Ger., [A] 6.12.28, [B] 1.12.28).—(A) Hormone preparations from organs, body liquids, or vegetable materials are hydrolysed with water in presence of catalysts such as Twitchell reagents, Dartring saponifier, or lipase of castor seed, thus avoiding the necessity for the removal of the large quantities of salts produced during hydrolysis with alkalis. (B) Impurities are removed from solutions of hormones by treatment with a definite amount of a water-miscible organic solvent and, after filtration, the hormone is separated by the addition of an organic liquid which is immiscible with water.

E. H. SHARPLES.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENT.

Films (B.P. 315,840 and 318,250).—See V.

## XXII.—EXPLOSIVES; MATCHES.

PATENTS.

[Preparation of] nitrocellulose [from wood pulp]. N. PICTON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,235, 27.5.29).—For the preparation of nitrocellulose of high density, wood-pulp sheets are cut into pieces about  $\frac{1}{8}$  in. square, so that the conglomeration of the fibres remains undisturbed and the product has a bulk density of 240–340 g. per litre. The mixed acid used for nitration must contain not less than 40% of nitric acid, a suitable composition to give nitrocellulose containing 11.7% N consisting of nitric acid 70%, sulphuric acid 20.5%, water etc. 9.5%.

W. J. WRIGHT.

**Continuous separation and after-separation of nitroglycerin and residuary acid.** A. SCHMID and J. MEISSNER (B.P. 336,253, 5.7.29).—An inclined separator, with cooling jacket, is employed, so that the emulsion of nitroglycerin and residuary acid, which is continuously introduced, being heavier than the nitroglycerin at the top and lighter than the residuary acid at the bottom, forms a horizontal layer at the zone of separation. As the acid flows downwards, drops of nitroglycerin separate at the top wall of the separator and move upwards, and owing to this separation and the cooling of the acid the latter gradually becomes heavier, so that circulation is ensured. Outlets are provided at the top and bottom for the nitroglycerin and residuary acid, respectively. The cooling jacket may be dispensed with if desired, or it may be replaced by an internal cooling coil and external insulation. A series of plates may be inserted in the separator in the direction of its longitudinal axis to guide the acid in its descent, and if an empty space is maintained at the upper end of these plates, a preliminary separation of the nitro-

glycerin takes place, a window at this part of the separator enabling the process to be watched.

W. J. WRIGHT.

## XXIII.—SANITATION; WATER PURIFICATION.

**Special equipment for protection against toxics in the chemical industry.** G. L. TURNER (Ind. Eng. Chem., 1930, 22, 1096–1099).—Industrial gas masks afford protection against every known gas, including carbon monoxide, for which a special catalyst is employed. Sectional drawings of the canister of the Burrell All-Service gas mask are shown. In confined spaces, where there is deficiency of oxygen, hose masks are used. Self-contained oxygen apparatus demands expert use, and is worn only in extreme emergencies. A description is given of the R.-M. indicator for detecting the percentage of inflammable gas, including the lower explosive concentration, in the atmosphere, and of a detector, designed by G. W. Jones, for rapid determinations. For determining carbon monoxide, the Hoolamite detector, involving a colorimetric test, gives rapid results; the Bell Telephone Laboratories employ palladium chloride in their device, the colour changing from canary-yellow to grey in presence of varying amounts of the gas. The M.-S.-A. continuous carbon monoxide recorder is briefly described. Rescue appliances include an inhalator, by means of which carbogen, a mixture of 93% of oxygen and 7% of carbon dioxide, is administered in cases of gassing. W. J. WRIGHT.

**Fatalities due to vitiated air produced by the oxidation of vegetable refuse.** A. R. TANKARD and D. J. T. BAGNALL (Analyst, 1930, 55, 673–676).—A case of poisoning due to carbon dioxide is reported.

D. G. HEWER.

**Soundness of concrete aggregates.** KRIEGE.—See IX.

PATENTS.

**Accelerating the action of breathing cartridges filled with peroxide.** DEUTS. GASLÜBLICHT-AUERGES.M.B.H., and HANSEATISCHE APPARATEBAU GES. (B.P. 337,170, 9.10.29. Ger., 7.11.28).—The initial rate of discharge of oxygen is accelerated by the discharge into the breathing bag of a small amount of a gasiform acid, *e.g.*, carbon dioxide, liberated from a small cylinder containing the gas under pressure. C. JEPSON.

**Insecticide.** H. V. McCLELLAN, Assr. to E. S. McCLELLAN (U.S.P. 1,758,734, 13.5.30. Appl., 24.2.27).—An insecticide containing finely-powdered ferrous carbonate (10–100 wt.-%) is claimed. It may be mixed or coated with starch and/or sugar. E. H. SHARPLES.

**Composition for repelling insects.** D. H. GRANT, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,755,178, 22.4.30. Appl., 29.12.28).—Solutions containing terpinyl esters, such as the acetate, propionate, etc., either alone or mixed with vehicles such as petrolatum, waxes, pyrethrum extract, kerosene or other mineral oils, and an emulsifying agent, are claimed.

E. H. SHARPLES.

**Cooling of air** (U.S.P. 1,749,763). **Dust filters** (B.P. 337,304).—See I. **Zeolites** (B.P. 319,746). **Glauconite** (U.S.P. 1,757,374).—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JAN. 16 and 23, 1931.\*



### L—GENERAL; PLANT; MACHINERY.

**Contribution of the physicist to the inorganic chemical industry.** P. GMELIN (Chem. Fabr., 1930, 433—434, 446—449, 457—458, 469—472).—Modern developments in inorganic chemistry, which involve especially the handling of gases, have greatly increased the importance of physical adjuncts, especially in such matters as automatic measurements and control, and the obtaining of material and energy balances. The following instruments are described: registering pressure-difference recorder for the measurement of gas flow, registering gas-density recorder for the control of nitrogen-hydrogen mixtures for ammonia synthesis, carbon monoxide recorder with alarm device operating by thermoelectric means, instruments for recording the concentration of coloured solutions optically and for mechanically titrating boiler feed-water. Amongst apparatus suitable for research work is described an automatic control for atmospheric moisture used in investigation of the effect of tropical atmospheric conditions on fertilisers. An example of the practical application of physics is given in the design of an air injector. Amongst the functions of the physicist in connexion with plant construction are the measurement of the vibrations of machinery and the oscillations of chimney stacks; the operations of magnetic analysis and spectrum analysis are also within his sphere. Several examples are given of the use of the latter method, employing ultra-violet light and a registering photometer, for the determination of small proportions of metals, e.g., 0.004% Cu in iron. A description is given also of the use of an "ultra-spectrometer," employing rock-salt prisms and a thermocouple, and using wave-lengths from  $3\ \mu$  to  $4\ \mu$  for gas analysis. By this, 0.002%  $\text{CH}_4$  in hydrogen can be detected. As every molecule produces its own characteristic lines the method is of wide range, but more investigation is needed before it can be used with the certainty of the ultra-violet ray analysis of metals.

C. IRWIN.

**[Degree of] fineness of solid substances.** A. H. M. ANDREASEN (Dansk Tidsskr. Farm., 1930, 4, 261—283).—The degree of fineness of a solid can be determined by sieving for grains down to a limit of 0.06 mm.; below that size sedimentation methods must be employed. For very small grains the pipette method should be used, and a special type of apparatus devised for this purpose is described. Using water as the liquid for sedimentation, the range of measurement attainable in this way lies between 0.03 and 0.0001 mm., but if glycerin be employed the upper limit can be extended to 0.3 mm. The degree of fineness is best expressed by means of distribution curves, using grain size ( $K$ ) as

abscissæ and the fraction of the whole quantity having grain size  $< K$  as ordinates. The influence of grain size in the employment of a number of technical materials is discussed, and the various methods available for obtaining a desired degree of subdivision are indicated.

H. F. HARWOOD.

**Bulking properties of microscopic particles.** P. S. ROLLER (Ind. Eng. Chem., 1930, 22, 1206—1208).—Powdered anhydrite, gypsum, Portland cement, and chrome-yellow pigment were sieved through 200-mesh and separated by an air-analyser into groups of homogeneous particle size. The particle diameter for each group was measured under the microscope and the surface mean diameter  $d_s$  (diam. of a sphere or cube of equal surface per g.) was calculated from the relationship  $d_s = \Sigma d^3 / \Sigma d^2$ , where  $\Sigma d$  is the sum of the diameters for all particles measured. The "bulkiness" (volume of unit weight) of each group was obtained by measuring the weight of powder filling a known volume under standard tapping conditions; the density was determined with the pycnometer. The difference between the sp. vol. and the "bulkiness" is described as the "voids per g." It is found that for all powders the same functional relationship holds between voids per g. ( $V$ ) and the surface mean diameter ( $d_s$ ). As  $d_s$  increases,  $V$  decreases until it reaches a critical value, after which it remains constant. The values of the various constants in the equation were determined experimentally, and it is therefore possible to calculate rapidly the degree of fineness of a powder from its "bulkiness." The surface mean diameter so obtained will be slightly less than the true value according to the non-homogeneity of the particle sizes. No information about the distribution of particle size is obtainable therefore. The values of the various constants throw some light on the factors governing particle packing, such as superincumbent weight of powder, electrostatic forces, shape, hardness, and nature of the surface of the grains.

J. A. SUGDEN.

**Rotary acid pumps.** F. W. ZIMMER (Chem. Fabr., 1930, 434—436).—A circulating pump is a much more efficient mixing agent for viscous liquids and the like than any form of agitator. An ebonite-lined rotary pump is described, of which it is claimed that the adherence of the lining is absolutely reliable. The design of the rotor practically prevents the corrosive liquid pumped obtaining access to the gland. A simple type of packing renders the gland tight when the pump is stationary. Curves are given indicating capacities and efficiencies. The pump is suitable for sulphuric acid except at high concentrations, nitric acid (up to 3%), and most other corrosive agents at any concentration.

C. IRWIN.

\* The remainder of this set of Abstracts will appear in next week's issue.

**Mathematical theory of filtration.** A. J. V. UNDERWOOD (World Eng. Cong., Tokio, 1929, Paper No. 278, 20 pp.).—The general filtration equation is derived, viz.,  $P = (RdV/d\theta)^{1/m} + rP^s cVdV/d\theta$ , in which  $P$  is the pressure of filtration,  $V$  the total volume of liquid filtered in time  $\theta$ ,  $R$  the resistance of the filter medium plus a thin layer of particles,  $m$  the power of the pressure to which the flow through the medium is proportional,  $c$  the thickness of cake deposited by unit volume (per unit area) of liquid, and  $rP^s$  is the sp. resistance of the sludge. When the sludge is incompressible,  $s = 0$  and the sp. resistance is independent of the pressure of filtration. The formula put forward by Walker, Lewis, and McAdams ("Principles of Chemical Engineering") for heterogeneous sludges, i.e., when filter aids are used, is criticised. D. K. MOORE.

**Cement pipe-lining.** CHAPPELL.—See IX. **Hardness tester.** DAYNES and others.—See XIV.

See also A., Dec., 1950, **Accessories for Saybolt viscosimeter** (WINNING). **Thermionic valve falling-ball viscosimeter** (MOORE and CUTHBERTSON). **Constant-temperature bath** (TREMEEARNE).

#### PATENTS.

**[Continuous muffle] furnaces.** V. A. COLLINS (B.P. 337,845, 13.8.29).—The furnace is suitable for removing iron from sand, for use in glass-making, by means of a reducing gas and chlorine. It comprises a slightly inclined tube of silica or siliceous material heated externally and provided with internal stirrers in the form of blades sliding loosely in spiders mounted on a metallic shaft which is protected from the gases by sleeves; all parts in contact with the gases are of siliceous material. B. M. VENABLES.

**Furnaces employing combustion heating and electric heating in combination.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTSGES. (B.P. 316,229, 25.7.29. Ger., 25.7.28).—In order to combine the economy of fuel and the even distribution and ease of control of electricity, both methods of heating are applied to the same surfaces of the furnace. Separate temperature-operated regulators are provided for fuel and electricity, the former of which merely reduces the supply so that the heat produced by the fuel alone cannot damage any article in the furnace, i.e., it operates at a lower temperature than any of the electric regulators. Several electric heaters are provided, each with its own regulator, so that very uniform (or graduated) heat can be obtained. B. M. VENABLES.

**Ignition and combustion process for grates with progressive movement of the fuel, and device for such purposes.** STOCKHOLMS AKTIEB. PRIVAT (B.P. 317,011, 2.8.29. Ger., 8.8.28).—A chain-grate or other form of mechanical stoker is operated at such a speed that a substantial amount of coke is delivered over the far end, at which point a classifying device is situated and the hot coke is returned by conveyors to the grate immediately in front of the new fuel. B. M. VENABLES.

**Heating device.** SOC. CHEM. IND. IN BASLE (B.P. 338,097, 29.4.30. Ger., 29.4.29).—A form of construction of a flat hot-plate (heated by an internal

fluid) is described, in which the flat faces are strongly stayed by internal ribs coupled together by rods threaded through them. B. M. VENABLES.

**Rotatable drums for cooling materials discharged from rotary furnaces.** F. KRUPP GRUSONWERK A.-G., Assees. of H. STEHMANN (B.P. 338,069, 18.1.30. Ger., 18.1.29).—The only passage for material between the cooling drum and the exterior, or between two compartments, is through an arcuate chamber formed within the end of the drum and provided with flap doors operated at the correct time by a cam. B. M. VENABLES.

**Method of insulation against heat or sound and articles produced thereby.** BRIT. CELANESE, LTD. (B.P. 315,280, 10.7.29. U.S., 10.7.28).—Fibres of organic derivatives of cellulose, e.g., cellulose acetate in very fine threads, are used as filling for heat- or sound-insulating jackets. They have the advantage over other materials that they do not absorb moisture. B. M. VENABLES.

**Gyratory crushers.** NORDBERG MANUFG. CO., Assees. of D. COLE (B.P. 319,616, 23.9.29. U.S., 24.9.28).—A gyratory crusher of the cone type is enabled to take larger pieces of feed than usual by splaying out the entrance, so that the average angle would be too great to nip the material, and having the surface of the fixed upper cone or concave stepped so that actually slip does not take place. B. M. VENABLES.

**Grinding or crushing apparatus.** II. S. REX-WORTHY (B.P. 337,911, 27.9.29).—A rod-mill of the type in which the rods are confined to segmental spaces between the spokes of driving spiders is described. B. M. VENABLES.

**Impact pulverising mills.** HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 318,145, 7.8.29. Ger., 28.8.28).—The mill is totally enclosed except for inlet and outlet pipes, and the casing and beaters are so shaped that the grinding action is partly by impact of the particles on the beater members and partly by mutual impact of the particles while flowing in eddy currents transverse to the path of the beaters. B. M. VENABLES.

**Grinding mills of the swing-hammer or beater type.** J. P. VAN GELDER (B.P. 317,446, 7.8.29. Austral., 16.8.28).—The side of the disintegrating chamber remote from the feed is open and a truncated conical screen is attached, the largest diameter of which is rather less than that of the beater chamber. Inclined blades rotating on an extension of the beater shaft are provided to deflect oversize back into the mill. B. M. VENABLES.

**Grinding pans.** J. DRYSDALE and A. E. BOTTOMLEY (B.P. 337,618, 18.11. and 5.12.29).—A method of construction of a pan of the type having edge-runners grinding on a central horizontal zone which is surrounded by a sloping screening zone, and that in turn by a vertical wall, is described. The screening segments are of such a form that when assembled they form a true truncated cone. B. M. VENABLES.

**Removal of plastic material from mills.** GOOD-YEAR TIRE & RUBBER CO., Assees. of W. E. MACMONAGLE (B.P. 337,435, 28.6.29. U.S., 30.11.28).—Material such

as rubber stock is removed continuously from a mill by means of a device comprising an inclined belt and accessories which withdraw the material in the form of a ribbon, cut it into slabs, treat it with anti-friction powder, and mark and cool it without a pause for re-rolling.

B. M. VENABLES.

**Separation of dry materials.** I. L. BRAMWELL, and BIRTLEY IRON Co., LTD. (B.P. 338,119, 6.8.29).—The initial separation is effected by an upward current of air in a vertical main flue, at an intermediate point of which the material is fed in sideways by a subsidiary strong current of air from the bottom of a feed shoot. This shoot is at such an angle that the material would just rest thereon in the absence of an air current. A secondary separation may be effected by change of direction in a subsequent part of the apparatus.

B. M. VENABLES.

**Rotary mixing machines.** O. H. FORNARO (B.P. 337,634, 27.11.29. Belg., 28.11.28).—A machine of the type having a stationary jacketed pan and rotating stirrers is described.

B. M. VENABLES.

**Mixing of paints and other semi-liquid mixtures.** I. I. BEGGROW (B.P. 337,945, 21.10.29).—A roll of metal gauze is inserted in a pipe and the mixture forced through it.

B. M. VENABLES.

**Centrifugal separators.** A. NYROP (B.P. 337,965, 1.11.29).—Separated solid matter is removed from the interior of the bowl by means of a worm formed on a correspondingly shaped interior element which is caused to rotate at a different speed by means of gearing. The fixed frame of the apparatus forms no part of the gear train and the driving shaft is not coupled directly either to the bowl or to the inner member, but the three last-mentioned parts are connected by differential gearing of the bevel or worm type. The relative motion is produced solely by lag arising from either air friction or acceleration of the entering fluid.

B. M. VENABLES.

**Centrifugal apparatus for clarifying liquids.** H. LIST (B.P. 337,628, 22.11.29).—A centrifugal bowl is described which is suitable for purifying liquids containing, simultaneously, solid substances that are lighter and heavier than the liquid.

B. M. VENABLES.

**[Centrifugal clutches for] centrifugal machines.** WATSON, LADLAW & Co., LTD., and H. WATT (B.P. 337,648 and 338,026, 9.12.29).—Centrifugal slip-couplings permitting also relative axial movement between the driving and driven parts are described.

B. M. VENABLES.

**Pressure filters.** J. HOFFMANN and T. KRIEBERNIGG (B.P. 337,848, 14.8.29).—A filter-press of the single-compartment type has the filtering period adjusted to the nature of the material by means of a nut which can be adjusted to different positions on the screwed driving shaft.

B. M. VENABLES.

**Apparatus for sieving, sifting, filtering, drying, measuring, etc.** W. D. WILLIAMS (B.P. 337,786, 2.8.29).—An apparatus for effecting the above operations on samples or small batches of material comprises a pan or water-bath, a cylindrical element, and a conical

element, arranged to fit together in various ways in conjunction with accessories such as screens and legs.

B. M. VENABLES.

**Apparatus for regulating the flow of liquids or liquefiable solids.** A. T. PRENTICE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 337,777, 7.5.29).—An apparatus of the hollow plug-cock type, which delivers the material in batches equal to the space within the plug, is rendered suitable for use with materials that are fluid only at raised temperatures by providing the body of the apparatus with a heating jacket and the plug with an axial heating tube. The amount of fluid delivered is regulated by axial adjustment of the plug, which is cylindrical in shape, or of a piston within it.

B. M. VENABLES.

**Devices for measuring the velocity and the quantity of liquids and gases.** SVENSKA ACKUMULATOR AKTIEB. JUNGNER (B.P. 317,397, 9.7.29. Swed., 15.8.28).—A pressure-head on the Pitot principle is inserted in the stream to be measured, *e.g.*, through a ship's bottom, and the differential pressure produced is transmitted to a diaphragm which is restrained by an armature between the poles of an electromagnet; the armature is connected also to the arm of a rheostat which regulates the exciting current of the magnet so that a balance is always maintained. An ammeter in the circuit serves to measure the velocity of the fluid and an ampère-hour meter will measure the quantity of fluid that has flowed through a pipe or the distance travelled by a ship.

B. M. VENABLES.

**Distillation of liquids.** R. R. COLLINS (B.P. 337,545, 2.10.29. U.S., 11.10.28).—The liquid, *e.g.*, oil for fractionation, is distilled under high vacuum and the vapour produced is rectified by horizontal sprays of liquids which are locally circulated back to the same sprays, additional reflux liquid being admitted at the top and allowed to run down.

B. M. VENABLES.

**Air filters.** C. G. VOKES (B.P. 337,966, 1.11.29).—An air filter in the form of a deeply corrugated cylinder is constructed of felt stitched to wire mesh which may be pleated before or after attaching the felt.

B. M. VENABLES.

**Separation of grit and sulphurous fumes from boiler flue gases.** J. T. BARON and J. B. CLARKE (B.P. 337,436, 1.7.29).—The gases are treated with downwardly directed fine sprays of water while flowing in a passage that is entirely unobstructed, but is of such a cross-section that the speed of the gases is reduced uniformly to 5–6 ft./sec. and has a length in feet equal to six times the speed of the gases. The passage may be rectilinear or coiled annularly or helically round the base of a chimney.

B. M. VENABLES.

**Saturating, washing, cooling, or similar treatment of gases.** J. A. REAVELL (B.P. 337,507, 22.8.29).—An electric motor is provided with a centrifugal spraying rotor on each end of its shaft and is suspended near the axis of a conduit conveying the gases, so that the sprayers produce transverse screens of liquid. The liquid feed to the sprayers may be used first to cool the motor.

B. M. VENABLES.

**Conditioning of furnace gases before purification.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 337,448

31.7.29. Ger., 9.1.29).—The gases are cooled or heated under control of a temperature-measuring device and moistened (if necessary) by injected steam under control of a moisture-measuring device, so that they enter the purifier, *e.g.*, an electrostatic precipitator, under constant conditions. B. M. VENABLES.

**Carrying out chemical and physical processes.** C. H. LANDER (B.P. 338,108, 3.5.29).—To promote reactions between a fluid and finely-divided solid, liquid, or gaseous particles, the carrier fluid is put into vortical motion and the other element is introduced without radial motion at a point approximately on the radius of equilibrium, so that the added particles will remain near that radius while the carrier fluid moves inwards, thus producing relative motion. B. M. VENABLES.

**Hygrometers.** J. D. K. TURNER, and ELDERS & FYFFES, LTD. (B.P. 337,814, 7.8.29).—A supporting stand for a wet- and dry-bulb thermometer is described which protects the dry bulb from the effects of the wet. B. M. VENABLES.

**Briquette oven.** T. NAGEL (U.S.P. 1,782,244, 18.11.30. Appl., 3.1.27).—See B.P. 283,123; B., 1929, 458.

**Separation of materials of different sp. gr.** T. M. CHANCE (Re-issue 17,873, 18.11.30, of U.S.P. 1,685,521, 25.9.28).—See B., 1929, 2.

**Crusher.** D. COLE, Assr. to NORDBERG MANUFG. CO. (U.S.P. 1,775,750, 16.9.30. Appl., 24.9.28).—See B.P. 319,616; preceding.

**Grinding mill of the swing-hammer or beater type.** J. P. VAN GELDER (U.S.P. 1,777,905, 7.10.30. Appl., 29.7.29. Austral., 16.8.28).—See B.P. 317,446; preceding.

**Sludge separator.** A. NYROP (U.S.P. 1,780,655, 4.11.30. Appl., 31.10.29. Den., 2.11.28).—See B.P. 337,965; preceding.

**Centrifugal liquid purifier.** J. W. ADAMS (U.S.P. 1,781,676, 18.11.30. Appl., 20.12.29. U.K., 4.1.29).—See B.P. 323,914; B., 1930, 269.

**Liquefaction and separation of gas mixtures.** K. E. R. MEWES (U.S.P. 1,781,693, 18.11.30. Appl., 17.11.25. Ger., 18.11.24).—See B.P. 258,773; B., 1926, 968.

**Processing of materials by means of gases.** B. F. UHL, Assr. to INDUSTRIAL SPRAY-DRYING CORP. (U.S.P. 1,782,054, 18.11.30. Appl., 1.9.27).—See B.P. 296,421; B., 1929, 80.

**Refrigeration.** ELECTROLUX, LTD., Assecs. of W. T. HEDLUND (B.P. 338,795, 13.1.30. U.S., 12.1.29).

**Refrigerating apparatus.** WESTINGHOUSE ELECTRIC & MANUF. CO. (B.P. 338,447, 31.1.30. U.S., 4.2.29).

**Drying apparatus** (B.P. 337,899).—See X. **Regulating temperature** (B.P. 337,803).—See XI. **Rubber for filtering etc.** (B.P. 313,052). **Spray-drying apparatus** (U.S.P. 1,750,753).—See XIV.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Causes of gas outbursts in coal mines.** O. RUFF (Z. angew. Chem., 1930, 43, 1038—1046).—Carbon dioxide dissolves in coal and forms a supersaturated

solution, which may give up its gas under shock just as a supersaturated aqueous solution of carbon dioxide effervesces on shaking. The fine particles of coal produced lose carbon dioxide until the external and internal pressures are equal. The power for dissolving carbon dioxide varies with the nature of the coal, the chief difference being in the rate of increase of solubility with pressure. The mineral content of the coal is not of primary importance in this connexion, and structural features are more important than is the chemical composition of the coal. Methane is considerably less soluble than carbon dioxide in coal, but the diminished danger of outbursts in the mine is compensated by its inflammability. E. S. HEDGES.

**Industrial importance of the heterogeneous character of coal seams.** F. S. SINNATT (World Eng. Congr., Tokyo, 1929, Paper No. 284, 14 pp.).—The properties of the many different layers in coal seams vary greatly. Coal seams usually fracture at layers of fusain, and since this powders easily the dust from screening plant and the duff from washery plant contain a large proportion of it. When duff is mixed with slack for coke making, the mixing must be very intimate, otherwise lines of weakness will occur in the coke. When coal is screened the decrease of ash content with increase of size of coal is not regular. Cenospheres may be produced under certain conditions when burning pulverised coal, and because of their lightness and low combustibility may be a source of annoyance. The variation of caking value and of inorganic constituents in different parts of the seam make it necessary for these to be thoroughly mixed to give the best results during carbonisation. D. K. MOORE.

**Determination of the moisture in brown coal by drying in a stream of carbon dioxide.** C. STAEMMLER (Chem.-Ztg., 1930, 54, 928).—Determinations of the moisture content of brown coal by the loss of weight when heated in a current of carbon dioxide give results which agree fairly well with those given by the xylene distillation method, but which are lower than those obtained by direct weighing of the water collected by calcium chloride. The values obtained by difference and by direct weighing show variations amounting in some cases to over 1%. The lower values given by the difference method are due to adsorption of the carbon dioxide on the surface of the coal, and it is suggested that this action may lead to changes in the coal which will vitiate the results of other determinations made on the coal dried by this method. C. RANKEN.

**Production of [artificial] coal.** E. BERL, A. SCHMIDT, and H. KOCH (Z. angew. Chem., 1930, 43, 1018—1019).—The decomposition products of lignin and of pine wood when heated with water under pressure at 350° have been investigated. The black pulverulent substance obtained from wood and cellulose yields when compressed and heated at 200—250° a material resembling hard coal. Variations of the physical properties of the "coal" are caused by differences in the content of bituminous substances: thus the cellulose "coal" contains 33% of material soluble in acetone, whereas the lignin "coal" yields hardly any extract; the former yields on distillation at 1050—1150° 4—5%

of primary tar rich in phenolic compounds, despite its aliphatic origin, but lignin "coal" yields only about 1.5% of tar. Both tars contain the compounds characteristic of coal tar, *e.g.*, benzene, naphthalene, and anthracene. If for the initial cellulose decomposition a slightly alkaline solution (*e.g.*, 0.05*N*-sodium hydroxide) be used instead of water, a "coal" is obtained which resembles natural coal in almost all respects, such as appearance, soluble constituents, nature of the primary and secondary tar, and behaviour on coking. Lignin "coal" yields always a pulverulent coke, and the employment of a higher alkali concentration results in a product resembling asphalt. It has not been found possible to convert lignite into hard coal by pressure hydrolysis, and it appears that these two substances are of different origins, hard coal being derived from vegetable material poor in lignin and free from resins and waxes, and lignite from woody materials containing lignin.

H. F. GILLBE.

**Briquetting with smokeless pulp binders.** C. J. GOODWIN (World Eng. Congr., Tokyo, 1929, Paper No. 282, 5 pp.).—Grass, straw, or other lignitic vegetable tissue is stacked and wetted, and chemicals (5%) are added to assist bacterial action. After 2–4 months the fermented mass is milled with 1–6% of soda. It is then mixed with coal or coke, briquetted, and dried at 150°. The briquettes may be waterproofed by immersing first in dilute crude soap solution, and then in dilute aluminium sulphate solution. Estimates of the production costs are given.

D. K. MOORE.

**Constituents of lignites and their behaviour on distillation at low temperatures.** I. UBALDINI (Annali Chim. Appl., 1930, 20, 461–482).—The nature of the solvent exerts considerable influence on the yield of bitumen obtained from a lignite, alcohol–benzene mixture giving a higher yield than benzene alone. The xyloid lignites are normal in this respect, the increased yield of bitumen obtained with the mixed solvent falling within the limits given in the literature (*cf.* Schneider, "Extraktion und Destillation der Braunkohle," 1922, 14). Lignite pitch, however, gives up 6.98% of bituminous substances to alcohol–benzene, and only 1.12% to benzene; of the former quantity, 65% consists of montan resin, which is readily soluble in benzene. The increased extraction by benzene under pressure is attributable to the depolymerising effect of the high temperature, but the way in which the alcohol acts is not clear. Diagrams for the distillation at low temperatures show that the first drops of distillate appear at just above 200°, and that over the range 300–325° for xyloid lignite and 350–375° for the pitchy form, rapid decomposition of the tar-forming constituents begins, the bulk of the tar formation occurring over a limited temperature range. The results obtained indicate that all the organic constituents of lignites participate, although in varying measure, in the formation of the separate products of the distillation, and that the tar undoubtedly results from the pyrogenetic decomposition both of the bitumen and of the humin substances, the former giving almost the whole of the neutral tar and the latter mainly acid tarry products. The water produced is derived mostly from the humin substances,

which are hence the constituents of lignite richest in oxygen. The tar formed from the exclusively resinous bitumen of exceptionally bituminous lignites contains no paraffin, which is hence not formed by the dry distillation of montan resin. Both the tars obtained from xyloid lignites, and also the products derived from them, contain no bases, which are, therefore, present in these tars in only very small proportion; the results with pitchy lignites are less definite, but in the main confirm this observation. Basic substances originate principally in the non-bituminous constituents. The data obtained for the substances insoluble in ether are not sufficiently clear to allow of any definite conclusions being drawn.

T. H. POPE.

**Production of oil by pressure distillation of brown coal.** R. VON WALTHER, W. BIELENBERG, and W. JENTZSCH (Z. angew. Chem., 1930, 43, 1009).—By distillation of a bituminous coal under 15–20 atm. pressure there were obtained oil 23.3%, water 20.1%, coke 40.8%, and gas etc. 15.8%; no tar was formed. The oil had  $d_4^{20}$  0.828 and contained creosote 3.9%, paraffins 0.8%, and unsaturated hydrocarbons 33.5%; 48% of the oil distilled below 200°, and 96% below 300°. The coke was denser than that obtained by the ordinary distillation process, and the gas contained 23.4% ( $\text{H}_2\text{S} + \text{CO}_2$ ), 3.7% CO, 4.3%  $\text{H}_2$ , and 57.8% of paraffins.

H. F. GILLBE.

**Formation of carbon disulphide from hydrogen sulphide and coke.** T. J. DRAKELEY and W. J. BAKER (J.S.C.I., 1930, 49, 475–478 *r*).—When hydrogen sulphide was passed over heated wood charcoal or coke, an extended delay occurred before the evolution of gas and ultimate formation of carbon disulphide. This delay was due to the adsorption of the hydrogen sulphide by the carbon. Carbon disulphide was formed in small amounts (up to 0.02%) by the action of hydrogen sulphide on wood charcoal between 700° and 1000°, or on coke obtained from coal carbonised in the laboratory between 600° and 950°. To obtain the maximum yield of carbon disulphide the rate of travel of hydrogen sulphide over the reacting carbon surface must be adjusted to suit particular experimental conditions. In the present experiments 2.4 litres (at N.T.P.) per hour gave the highest yields. Carbon disulphide was not formed by the action of hydrogen sulphide on coke obtained by carbonising coal above 950°, nor from hydrogen sulphide and carbon when the reaction temperature was below 500° or above 975°. For cokes obtained from coals carbonised at definite temperatures the maximum yields of carbon disulphide were obtained when the reaction temperature was 800°. The highest yields of carbon disulphide for a definite reaction temperature were obtained from coke produced from coals carbonised between 750° and 850°. Carbon disulphide decomposed to the extent of more than 99% when passed over wood charcoal or coke between 850° and 1000°.

**Some properties of carbon black.** D. PARKINSON (Trans. Inst. Rubber Ind., 1930, 5, 263–283).—The particle size and shape, degree of blackness, adsorptive capacity, volatile constituents, and adsorbed gases of gas black, acetylene black, lampblack, and thermatomic black are discussed particularly from the point of view



of the use of these materials with rubber. Acetylene black is intermediate in fineness between gas black and lampblack, the particles ranging in size from those of lampblack to those of gas black; by centrifugal separation of the larger particles it is possible to obtain an aqueous suspension showing on dilution the amber tint characteristic of gas black. Using a testing machine of the pendulum type it is shown that at low concentrations lampblack and acetylene black give rubber mixtures of approximately equal resilience and greater than gas black, but with higher proportions lampblack gives the highest resilience; this result is not due merely to increased internal friction, because zinc oxide and magnesium carbonate up to relatively large proportions increase the resilience; the degree of hardness of mixtures with rubber, however, shows little relation with the particle size of the blacks. The theory that the dispersion of gas black in rubber is dependent on the adsorption of a unimolecular film of fatty acid (cf. Blake, B., 1928, 867; 1929, 828) is discountenanced by the fact that gas black can be dispersed well in acetone-extracted rubber by the ordinary mixing process. D. F. TWISS.

**Determination of the asphaltene content [of asphalt].** F. J. NELLENSTEYN and N. M. ROODENBURG (Chem.-Ztg., 1930, 54, 819).—Ether is recommended as more suitable for the extraction than light petroleum. A number of experimental results are given. S. I. LEVY.

**By-product ammonia—an economic aspect.** P. PARRISH (World Eng. Congr., Tokyo, 1929, Paper No. 279, 18 pp.).—Full details are given of the cost of manufacturing concentrated ammonia liquor from gas liquor. The desirability of gasworks undertaking this concentration depends on local conditions, which are fully considered. D. K. MOORE.

**Plant resins as the parent substance of the optically active constituents of petroleum.** M. A. RAKUSIN (Petroleum, 1930, 26, 1055—1056).—From a consideration of the work of Zelinski and Semiganovski (A., 1929, 1293) on the action of aluminium chloride on plant resins it is concluded that the optically active constituents of some natural petroleum may have been derived from plant resins by natural processes having the same resultant effect as the action of aluminium chloride. A. B. MANNING.

**Treatment of oil shale.** T. M. DAVIDSON (World Eng. Congr., Tokyo, 1929, Paper No. 283, 12 pp.).—Vertical-shaft, conveyor, and rotating-tube types of retorts, each of which may be adapted for either external heating by hot gases or internal heating by hot producer gas, superheated steam, and gas given off by the shale after reheating, are used for distilling oil shale; the application of an internally heated tunnel kiln is indicated. External heating especially with the rotating tube retort is preferred. The admission of steam into the retort minimises the decomposition of the oil, but not more than 60 lb. per ton of shale should be used. The best oil yields are obtained by keeping the temperature as near as possible to that required to decompose the bitumen, by providing ample passages for the evacuation of the vapours, and by adopting a heating gradient so that the oil vapours are removed at 360—460°.

D. K. MOORE.

**Cracking of hydrocarbons.** E. BERL and R. LIND (Petroleum, 1930, 26, 1027—1042, 1057—1070).—A description is given of experiments carried out on the cracking of oils under atmospheric pressure in a laboratory apparatus wherein the vapour of the oil is passed through an electrically heated tube, containing the catalyst if one is used, the gases and lighter oils produced being led through a condenser, an adsorption tube packed with activated charcoal, and a gas-meter, while the heavier oils are returned for further treatment. A Messel shale oil cracked at 440—480° in the presence of activated charcoal yielded after 7 hrs.' treatment 21.8% of benzene, of which 75% was soluble in liquid sulphur dioxide (therefore aromatic and unsaturated hydrocarbons), and 11.5% of gas. At 530—560° the same oil yielded after 3 hrs. 24.5% of benzene (olefines 24%, aromatics 55%, naphthenes 7%, and saturated hydrocarbons 14%, determined by Egloff and Morrell's method) and 25.9% of gas. By cracking in an empty tube above 600° lower yields (16—21%) of benzene of high unsaturated and aromatic content were obtained. Little cracking occurred below 600° when pumice was used as catalyst; at 600—700° 17.6% of benzene and high gas yields were obtained. A paraffin wax, m.p. 52—54°, cracked at 470—570° and at 570—600° yielded 53.3% and 34.8%, respectively, of benzene; the former product contained 25% of aromatic and 65% of paraffin hydrocarbons, the latter 60% of aromatic and 27% of unsaturated hydrocarbons. Somewhat higher yields were obtained when the iron tube was replaced by one of monel metal. Naphthalene was not decomposed at 550° in the presence of activated charcoal, but tetralin was readily reduced to naphthalene at 550—580°.

A. B. MANNING.

**Hydrogenation of residues from Boryslaw crude oil.** J. EHRLICH and A. SZAYNA (Petroleum, 1930, 26, 1043—1046).—By the repeated hydrogenation of a fraction obtained by removing the constituents boiling below 220° from Boryslaw crude oil the following successive yields (total 47.4%) of benzene were obtained: 26.6%, 9.4%, 7.3%, and 4.1% (calc. on the original oil). There were also produced 26.2% of oil and asphalt, and 26.4% of gas (and loss). The autoclave was maintained at 430—450° and the time of hydrogenation was varied from 4 to 8 hrs. With a higher temperature or longer reaction time a higher yield of benzene was obtained. The fractions of the benzene had a lower sp. gr. and a lower content of aromatic and naphthene hydrocarbons than the corresponding fractions of the straight-run benzene from the same crude. The combined fractions boiling below 165° had  $d_{4}^{15}$  0.714, saturated hydrocarbons 92%, unsaturated hydrocarbons 3%, aromatic hydrocarbons 5%.

A. B. MANNING.

**Compressibility and thermal expansion of petroleum oils in the range 0° to 300°.** R. S. JESSUP (Bur. Stand. J. Res., 1930, 5, 985—1039).—Measurements have been made of the compressibilities and coefficients of thermal expansion of 14 samples of petroleum oils, including 4 gas oils and 6 lubricating oils, from different sources, over the ranges 0—300° and 0—50 kg./cm.<sup>2</sup>; the results are tabulated and compared with those of previous observers. Although there is, in general, no direct relationship between

these properties, both are closely related to the viscosity of the oil and are approximately constant for oils of the same density and viscosity, although for oils having different viscosities the variation may amount to 30%. Tables are given showing the relative volumes of oils of viscosities from 0.020 to 5.000 c.g.s. units (at 100° F.) and  $d$  0.80–0.95, and of viscosities from 0.042 to 1.100 c.g.s. units (at 210° F.) and  $d$  0.90–1.00, under 0 and 50 kg./cm.<sup>2</sup> pressure, for the temperature range 0–400°.

H. F. GILLBE.

**Table for the calculation of the dynamic viscosity in centipoises from Engler degrees.** M. FREUND (Petroleum, 1930, 26; Motorenbetrieb, 1930, 3, No. 10, 3–1).—The table gives the dynamic viscosity corresponding with Engler degrees from 1.00 to 8.00, in steps of 0.05, and with sp. gr. at the temperature of measurement from 0.80 to 1.00, in steps of 0.01. For higher values than 8° E. the relationship is given by  $\eta = 7.6Ed$ .

A. B. MANNING.

**Burette for determination of unsaturated and aromatic hydrocarbons in oils.** J. FORMÁNEK (Chem.-Ztg., 1930, 54, 908).—The burette is provided with two bulbs near its lower end, which is closed by a tap; at the upper end is a cylindrical enlargement of about 25 c.c. capacity, closure at this end being made by a hollow stopper with a perforation which in one position coincides with a hole in the neck of the burette. The lower bulb is filled with sulphuric acid to the zero point between the two without wetting the upper portion, into which are then introduced 25 c.c. of the liquid under test. The perforated stopper and neck permit occasional release of excess internal pressure caused by the heat of reaction; the enlargement facilitates effective agitation. After the completion of the action the burette is cooled and the acid level is then reduced to the zero point; the volume of residual hydrocarbon liquid is indicated by the level of the upper surface in the graduated portion.

D. F. TWISS.

**Alcohol as motor fuel.** P. W. UHLMANN (Chem.-Ztg., 1930, 54, 818–819).—A fuel containing absolute alcohol 75, ether 15, petroleum 10 pts. by wt. was employed in Brazil during the war. Alcohol of 99.8% purity was obtained by dehydrating the product fermented from sugar-cane liquor first with freshly burnt lime, then with calcium carbide, and distilling. The fuel gave the same mileage as petrol, and caused no engine trouble or corrosion.

S. I. LEVY.

**Physics and the chemical industry.** GMELIN.—See I. **Hydrolysis of sodium chloride by steam.** BERL and STAUDINGER.—See VII. **Wood carbolineum as insecticide.** JENČIČ.—See XVI. **Fuel gas by fermentation.** BUSWELL.—See XVIII. **Mineral oil emulsions.** KRANTZ.—See XX.

See also A., Dec., 1513, **Adsorption of methane and hydrogen on charcoal** (FROLICH and WHITE). 1531, **Hydrocarbon-forming action of iron catalyst** (KODAMA). Reaction  $\text{CO}_2 + \text{H}_2 \rightarrow \text{H}_2\text{O} + \text{CO}$  on tungsten and thoriated tungsten (SRIKANTAN). 1532, **Action of high-speed electrons on methane, and on oxygen-carbon monoxide** (McLENNAN and GLASS).

1553, **Formation of diisobutylene from acetone** (PETROV and IVANOV).

## PATENTS.

**Low-temperature carbonisation of fuel.** F. L. DUFFIELD (B.P. 335,613, 5.7.29).—The powdered fuel is passed through a horizontal retort, steam being used as the carrying medium. The steam is admitted tangentially at a number of jets arranged along the lower edge of the retort, and is further heated as it enters by being caused to pass over a part of the retort wall which is heated externally to a high temperature. A number of spiral baffles fixed within the retort cause the steam and fuel to take a helical path therein. The retort is preferably divided by means of bulkheads into three zones of successively higher temperature. The steam and volatile products of carbonisation are passed through dust-precipitating chambers to heat inter-changers and/or condensers. The heat recovered from the steam and hot vapours is utilised for the production of steam for use in the process. The treated fuel is delivered into a hopper at the end of the retort communicating with a rotary discharge valve.

A. B. MANNING.

**Manufacture of liquid products from solid distillable carbonaceous materials by destructive hydrogenation.** I. G. FARBENIND, A.-G. (B.P. 315,764, 15.7.29. Ger., 17.7.28).—Carbonaceous material such as brown coal is subjected to a mild hydrogenation under pressure so that the formation of benzene and consequent precipitation of asphaltic matter is avoided. The material is passed through the hydrogenation apparatus, countercurrent to the hydrogen, at about 450° and 200 atm. From the gas a light oil is condensed which on further hydrogenation gives benzene. The solid or semi-solid residue is extracted with benzol under a pressure of about 75 atm. The extracted materials are of high mol. wt. and can be worked up into lubricating oils.

T. A. SMITH.

**Manufacture of combustible gas.** HUMPHREYS & GLASGOW, LTD., ASSEES. of W. H. FULWEILER (B.P. 335,869, 27.1.30. U.S., 2.5.29).—A non-poisonous combustible gas suitable for domestic and industrial consumption is produced by passing a portion of blue water-gas over a suitable heated catalyst for the conversion of its carbon monoxide and hydrogen into methane, carbon dioxide, and water vapour, heating another portion of blue water-gas with steam in the presence of a suitable catalyst for the conversion of its carbon monoxide into hydrogen and carbon dioxide, mixing the two gases so produced together, and removing the carbon dioxide and water vapour from the mixture.

A. B. MANNING.

**Apparatus for treatment of hydrocarbons.** G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,751,729, 25.3.30. Appl., 1.9.20. Renewed 21.11.27).—Charging stock is pumped through a heating coil mounted in a furnace and, while still liquid, is passed through a valve into the side of an expansion chamber provided with a residue draw-off and vapour outlet, both controlled by valves. The vapour outlet is connected to a horizontal dephlegmator provided with alternately disposed baffles and an

inclined bottom, the lowermost part of the latter being connected to a pipe leading back to the charging line. The uncondensed gases pass on through a valve to a water-cooled condensing coil connected to a receiver with a draw-off pipe and gas outlet. The whole apparatus is maintained under a suitable differential pressure.

H. S. GARLICK.

**Apparatus for cracking oil.** G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,751,730, 25.3.30. Appl., 11.2.21. Renewed 21.2.29).—The apparatus consists of a cracking coil mounted in a furnace and an expansion chamber fitted with a dephlegmator. The temperature of the dephlegmator is controlled by means of an air blower, the air, preheated in the dephlegmator, being used in firing the furnace.

T. A. SMITH.

**Conversion of hydrocarbons into hydrocarbons of lower b.p.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,046, 26.7.29).—Hydrocarbons of high b.p. are thermally decomposed at 300–600° (preferably at 350–450°) in the presence of catalysts which comprise hydrocarbon oil-soluble metal compounds of  $\beta\delta$ -diketones *e.g.*, the acetylacetonates or homologues thereof, the metallic constituent preferably belonging to groups III–VII.

H. S. GARLICK.

**Manufacture of hydrocarbons.** I. G. FARBENIND. A.-G. (B.P. 313,123, 15.5.29. Ger., 7.6.28).—Mineral oils or tars are subjected first to a condensation process and then to hydrogenation in the presence of a catalyst such as molybdenum, zinc, and magnesia, at 450° under high pressure. The condensation may be carried out at high or ordinary pressures and at raised temperatures in the presence of catalysts. Gases containing or supplying oxygen, such as steam, are used as condensing agents. Under these conditions lubricating oils are obtained.

T. A. SMITH.

**Treatment of light hydrocarbons and kerosene.** D. A. HOWES, R. SCOTT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,515, 22.7.29).—Light distillates are more stable as regards colour and gum formation if distilled at temperatures below 300° in the presence of gaseous ammonia. The ammonia may be removed from the distillate by washing, but it is best not to remove it immediately, as its presence inhibits the formation of gum.

T. A. SMITH.

**Apparatus for condensing hydrocarbon vapours.** J. E. BELL, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,757,108, 6.5.30. Appl., 22.1.25).—Oil vapours from a reflux tower or vapour space of a still are passed through an evaporative cooler and/or a condenser arranged to discharge into a separator where the fixed gases are separated from the condensed distillate, which is passed to a tubular heat exchanger. Cooling water is conducted first through the cooler and then through the condenser and, if desired, the evaporator.

H. S. GARLICK.

**Purification of hydrocarbons.** J. C. BLACK, ASSR. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,749,240, 4.3.30. Appl., 10.3.28).—Cracked gasoline is treated with caustic soda solution and then with caustic soda solution containing manganese dioxide, whereby it is

rendered sweet to the "doctor" test. The spirit is then treated with absorbent clay at a temperature of at least 120° and under sufficient pressure to prevent vaporisation, and is finally distilled to remove polymerides.

T. A. SMITH.

**Refining of hydrocarbons.** N.V. DE BATAAFSCHE PETROLEUM MAATS., ASSEES. of J. A. BUCHEL and R. N. J. SAAL (B.P. 316,137, 10.7.29. Holl., 23.7.28).—Liquid hydrocarbons are once treated with sulphur dioxide, acetone, or furfuraldehyde, preferably on the counter-current principle, to remove the major portion of the aromatic hydrocarbon content. Benzol or other low-boiling aromatic hydrocarbon is then added to the oil before again treating, in order to increase the ratio of the high-boiling aromatic hydrocarbon concentration in the extract layer to that in the raffinate layer.

H. S. GARLICK.

**Bleaching and deodorising products of high-molecular hydrocarbons (*e.g.*, paraffin wax).** H. FRANZEN and M. LUTHER, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,757,455, 6.5.30. Appl., 5.1.29. Ger., 4.2.28).—The oxidation products of paraffin wax are treated while hot with a mixture of a metal (*e.g.*, zinc) and an acid furnishing nascent hydrogen. The product is finally washed.

H. S. GARLICK.

**Oxidation of hydrocarbons in the gaseous phase.** H. HARTER (B.P. 337,407, 24.6.29).—Air or other mixture of nitrogen and oxygen is introduced into a lower chamber and passes through slots into an externally cooled pipe through which a high-tension electric arc is passed. The pipe is preferably made of quartz, zirconia, thoria, boron nitride, or other substance catalysing the formation of nitrogen oxides, and leads to a double-walled tube, surrounded by a cooler, to which are supplied through a number of passages hydrocarbon gases or vapours, especially methane; the tube ends in a chamber in which the hydrocarbon gases come into direct contact with the activated gases leaving the flaming arc. The oxidation products are at once withdrawn and strongly cooled. The formation of formaldehyde is promoted by the presence in the reaction chamber of catalysts such as copper, silver, cobalt, and manganese, preferably deposited or silica gel or activated carbon.

H. S. GARLICK.

**Cracking of hydrocarbon oils.** W. W. TRIGGS. From PANHANDLE REFINING Co. (B.P. 337,380, 29.6.29).—Oil is heated in a primary heating zone under a pressure proportionate to the latent heat of vaporisation of the oil under treatment and is then admitted to a flashing or expansion zone wherein a pressure below 14 lb. abs. is maintained. The resulting vapours are passed to a cracking zone maintained at 540–650° and under 6–14 lb. abs., and the products are passed through a number of heat-exchanging and fractionating zones countercurrent to make-up oil. The final low-boiling products are cooled and collected, while the make-up oil, preheated to the temperature of initial vaporisation, is introduced into the system as vapour at a point preceding the flashing zone.

H. S. GARLICK.

**Treatment of hydrocarbon oil.** G. EGLOFF, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,751,731, 25.3.30. Appl., 22.10.23. Renewed 12.1.29).—Petrol-

cum emulsions are heated to cracking temperature, under pressure, in a coil and passed to a dephlegmator. Fractions are taken from the dephlegmator and the vapours of light oil and water are led to a condenser where the bulk of the water is condensed under pressure. The remaining vapours are condensed separately.

T. A. SMITH.

**Treatment of hydrocarbon oil.** KOPPERS Co., Assecs. of J. A. SHAW (B.P. 336,636, 15.4.29. U.S., 6.11.28).—A previously acid-washed hydrocarbon oil is treated with a washing solution containing free alkali and alkali phenoxide in such amount that the free alkali neutralises most of the acid content of the oil while leaving a portion thereof to liberate phenols from the alkali phenoxide which dissolve in the oil. The washing solution and products of neutralisation are drawn off from the oil, which is then treated with a further quantity of washing solution sufficient to complete the neutralisation of any remaining acid substances originally present as well as the dissolved liberated phenols. This washing solution is withdrawn and contains substantial quantities of alkali phenoxides and not more than limited amounts of the neutralisation products of the original acid-treatment of the oil.

H. S. GARLICK.

**Refining of hydrocarbon oils.** E. C. HERTHEL, H. L. PELZER, and J. G. STAFFORD, ASSES. to SINCLAIR REFINING Co. (U.S.P. 1,756,155, 29.4.30. Appl., 16.6.28).—A stream of lubricating oil stock is passed through a heating zone, where it is heated to a relatively low vaporising temperature, into a vapour-separating zone into which is introduced superheated steam and where the vapours, including the lubricating oil constituents, are bubbled through a hot liquid oil bath maintained alkaline by supplying hot caustic alkali thereto. Vapours from this operation are subjected to fractionation and the higher-boiling constituents condensed. A lubricating oil fraction or a series of such fractions is then condensed from the remaining vapours, and while at 260–315° each is brought in contact with a solid absorbent such as fuller's earth, in the presence of a non-oxidising or preferably a reducing atmosphere.

H. S. GARLICK.

**Refining of light hydrocarbons, especially petrol.** R. SCOTT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,885, 31.5.29).—The vapours from the destructive hydrogenation of coal are passed at above 10 atm. and at 200–250° through a solid purifying agent (bauxite, fuller's earth), which is revived by treatment with air preheated to 400–500°.

T. A. SMITH.

**Continuous distillation of heavy hydrocarbon oils.** R. E. WILSON, ASSR. to STANDARD OIL Co. (U.S.P. 1,751,182, 18.3.30. Appl., 3.4.25).—Crude oil or other hydrocarbon is subjected to distillation by heat and steam in a continuous battery of stills, from the last of which the residual oil passes through a pipe to a mixing device formed with a central expanding nozzle, for the introduction of the oil, surrounded by a casing which forms a chamber into which superheated steam at over 538° is introduced. The nozzle has openings on its expanding side directed in the direction of flow of oil, the steam expanding on passing through these openings and intermingling with the oil. The mixer opens into

a conduit where thermal equilibrium is obtained, and which leads to a separator consisting of a central conduit the end portion of which is partly surrounded by an outer casing having a draw-off valve. Within the casing the conduit is provided near its exit end with a horizontal slit on its lower side. An Archimedean screw or series of spiral vanes extends partly through the conduit and imparts to the mixture of vapours and unvaporised liquid a rapid rotating movement, thereby throwing out upon the walls of the conduit the unvaporised liquid, which drains through the opening into the outer casing and is withdrawn. Gases and vapours pass from the separator to a condenser and, if desired, to a source of vacuum.

H. S. GARLICK.

**Decolorisation of mineral oils and oil distillates.** N.V. MIJNBOW- EN CULTUURMAATS. "BOETON" (B.P. 335,952, 5.4.29. Holl., 3.11.28).—Discoloured oils, particularly cracked distillates, are improved without loss of antiknock value by treatment with caustic soda or potash dissolved in alcohols or ketones. With intensely coloured oils the action is enhanced by the addition of small quantities of reducing agents such as aldehydes or zinc powder. The treated oil is distilled, the impurities being obtained as a resinified residue.

T. A. SMITH.

**Preventing the formation of coke in the cracking of mineral oils.** S. SEELIG (B.P. 336,822, 6.11.29. Ger., 7.11.28).—The products of cracking leaving the reaction zone are brought into immediate contact with inert gases or vapours, *e.g.*, nitrogen, hydrogen, steam, or carbon dioxide, in order to prevent further reaction.

H. S. GARLICK.

**Elimination of colloidal asphaltenes and carbonenes from fuel oil.** A. JURRISEN, ASSR. to SIMPLEX REFINING Co. (U.S.P. 1,756,981, 6.5.30. Appl., 24.1.25).—Cracking residues are mixed with a light petroleum distillate and kept at a raised temperature not above the b.p. of the solvent for sufficient time for most of the asphaltenes and carbonenes to be separated from the free carbon and dissolved in the balance of the cracking residue, from which the solvent is recovered by distillation before passing first through a filter to remove the solid granular carbon and thence to storage.

H. S. GARLICK.

**Treatment of commercial gasoline for internal-combustion engines to remove and prevent the formation of carbon.** M. J. FESSLER (U.S.P. 1,749,244, 4.3.30. Appl., 24.12.25).—About 0.2% of tetrahydronaphthalene is added to the gasoline.

H. S. GARLICK.

**Treatment of waste products from petroleum refining.** I. G. FARBENIND. A.-G. (B.P. 316,274, 26.7.29. Ger., 26.7.28. Addn. to B.P. 299,086; B., 1928, 919).—Viscous oils, suitable as lubricants, are prepared by treating an extract obtained in the refining of petroleum oils by means of liquid sulphur dioxide with a lower olefine or gaseous mixture containing lower olefines, in the presence of a catalyst of the Friedel-Crafts type, *e.g.*, aluminium, ferric, or zinc chloride, preferably under increased pressure.

H. S. GARLICK.

**Heat-treatment of carbonaceous materials to obtain oils of low b.p.** STANDARD OIL DEVELOPMENT Co., Assecs. of R. T. HASLAM (B.P. 314,871, 2.7.29. U.S.,

3.7.28).—Fluid carbonaceous material is circulated through a cracking and hydrogenation cycle, low-boiling constituents being removed at each stage and the residue recirculated. Oil is pumped through a cracking coil to a reaction chamber and expanded into a vaporiser. Light products are removed and the lighter portion of the residue is returned for further cracking. The heavy residue and some of the intermediate fractions are mixed with powdered coal and passed through a heating coil with hydrogen to a stirred reaction chamber where further treatment with hydrogen is given. The vapours from this reaction chamber may be passed through catalyst and are then condensed, the heavy portion being returned to the cracking coil. T. A. SMITH.

**Manufacture of lubricating oil.** G. H. TABER, JUN., ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,749,896, 11.3.30, Appl., 26.5.28).—Lubricating oil stock is distilled in steam, and the vapours are passed upwards through a treating tower fitted with baffles. Soda solution is sprayed into the top of the treating tower and removed at the bottom with a certain amount of condensed oil. Vapours from the treating tower are either fractionally condensed out of contact with soda or given a further treatment. T. A. SMITH.

**[Highly viscous] lubricating and other technical oils.** I. G. FARBENIND. A.-G. (B.P. 337,639, 28.11.29, Ger., 29.11.28).—An increase in viscosity, and in some instances a viscosity that remains approximately constant over a wide range of temperature, is acquired by dissolving in such oils one or more esters, ethers, mixed esters, or ether-esters of polymeric carbohydrates (especially of starch or cellulose). H. S. GARLICK.

**Lubricant.** E. A. RUDIGIER, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,750,134, 11.3.30, Appl., 23.4.24).—A heavy residual oil containing sodium naphthenate is compounded with a mineral lubricating distillate having viscosity 65–200 sec. (Saybolt) at 38°; the product has a viscosity (Saybolt, 100°) above 125 sec., and a pour test not above 3°.

H. S. GARLICK.

**De-waxing of oils.** OLIVER UNITED FILTERS, INC., ASSECS. of J. W. WEIR (B.P. 316,271, 26.7.29, U.S., 26.7.28).—A flocculent wax-absorbing medium, *e.g.*, fuller's earth, kieselguhr, filter cel, etc., is added to a chilled, diluted, wax-bearing oil, and the mixture is passed through a filter to separate the oil from the wax and flocculent medium; the latter are separated subsequently by use of a suitable solvent.

H. S. GARLICK.

**Extraction of products from montan wax by means of solvents.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,929, 1.7.29).—The yield of wax acids and esters from crude montan wax by extraction with solvents is increased and the quality of the product improved if the extraction is carried out under pressure. Extraction at 150° under 12 atm. with a 4:1 mixture of alcohol and benzol gives, on cooling, a mass of crystals of pale brown wax, the resins remaining in solution. T. A. SMITH.

**[Gas] generator or producer.** E. SCHUMACHER

(U.S.P. 1,781,765, 18.11.30, Appl., 31.1.28, Ger., 8.2.27).—See B.P. 285,007; B., 1928, 806.

**Recovery of sulphur dioxide from [mineral] oils containing same.** G. CATTANEO, ASSR. to ALLGEM. GES. F. CHEM. IND. M.B.H. (U.S.P. 1,758,595, 13.5.30, Appl., 27.5.26, Ger., 5.3.26).—See B.P. 267,071; B., 1927, 386.

**Doors of horizontal ovens for production of gas, coke, etc.** E. WOLFF (B.P. 338,450, 6.2.30, Ger., 3.10.29, Addn. to B.P. 314,281).

**Burners for pulverulent materials.** H. NIELSEN and B. LAING (B.P. 338,542, 21.5.29).

**Pulverised fuel burner system.** A. W. FIELD (B.P. 338,362, 15.10.29).

**Apparatus for burning pulverised fuel.** GASIFIED PULVERISED FUEL (G.P.F.), LTD., H. D. TOLLEMACHE, and O. MARGETSON (B.P. 338,243, 27.8.29).

**Gas burners for gas-fired installations.** A. C. BECKER (B.P. 338,122 and 338,128, 8.8.29, Ger., 13.4.29).

**Wicks for hydrocarbon burners.** C. and E. SCOTT-SNELL, and SCOTT-SNELL LABS., LTD. (B.P. 338,277, 23.9.29).

**Fuel-distributing device for gas producers and the like.** A. JADOUL (B.P. 338,141, 11.7.29, Belg., 11.7.28).

**Combustion process for grates** (B.P. 317,011). **Distillation of liquids** (B.P. 337,545).—See I. **Acetylene** (B.P. 338,003).—See VII. **Paving compositions** (B.P. 334,588).—See IX. **Rubber composition** (U.S.P. 1,751,848). **Working up rubber etc.** (B.P. 337,095).—See XIV. **Anhydrous alcohol** (U.S.P. 1,751,211).—See XVIII.

### III.—ORGANIC INTERMEDIATES.

**Detection and determination of pyridine.** S. B. TALLANTYRE (J.S.C.I., 1930, 49, 466–468 t.).—Methods for the determination of pyridine and "pyridine bases" are reviewed and suggestions are made with regard to improved procedure. Previous to distillation of the bases it is recommended to make the solution just pink to phenolphthalein indicator, thus freeing the bases, but preventing any appreciable amount of ammonia distilling over. Benzene can be used as a useful solvent for the extraction of "pyridine bases" from alkaline liquors, especially after the addition of sodium chloride. Phosphotungstic and silicotungstic acids are sensitive reagents for pyridine and the "pyridine bases," and are applicable, with certain limitations, to their determination in small quantities. Tests based on the formation of copper pyridine thiocyanate and 2-aminodihydropyridinium-phenyl bromide have been examined. The use of a solution prepared from aqueous 5% potassium cyanide and bromine water, instead of solid cyanogen bromide, is recommended as a convenient process for the determination of traces of pyridine such as may occur in ammonia and methylated spirits.

**Bromo-potentiometric titration of  $\beta$ -naphthol-sulphonic acids in presence of each other.** I. Schaeffer acid, R acid, and G acid. II. Mixtures

containing crocein acid. S. D. FORRESTER and D. BAIN (J.S.C.I., 1930, 49, 410—412 t, 423—425 t).—I. Schaeffer and R acids are monobrominated quantitatively in sulphuric acid solution at the ordinary temperature, whereas G acid is not. To 50 c.c. of a solution containing any or all of the three acids or their salts 20 c.c. of concentrated (36*N*) sulphuric acid are added and the solution is titrated potentiometrically at the ordinary temperature with a standard solution containing 5.567 g. of potassium bromate and 30 g. of potassium bromide per litre. This high concentration of acid is necessary in order to obtain a good potential jump. A piece of platinum gauze about 1 cm. square was used as indicator electrode, and an interval of 1 min. was allowed to elapse before taking a reading.

II. When crocein acid is present, the procedure is modified by adding 10 c.c. of a 20% solution of potassium bromide to the sulphuric acid solution and titrating with a standard solution containing 5.567 g. of potassium bromate per litre at 30°. The above method gives only the Schaeffer or/and R acid content of a mixture. Results are within 1%.

**Manufacture of vanillin.** J. SCHWYZER (Chem.-Ztg., 1930, 54, 817—818, 839—840).—Four commercial methods of synthesis are described, with details of manipulation. (1) Pyrocatechol is prepared by chlorination of phenol at 160—170° and 9 atm. followed by hydrolysis of the *o*-chlorophenol with baryta: this is treated with formaldehyde in presence of hydrochloric acid, iron filings, and *m*-nitrobenzenesulphonic acid, the last-named being reduced to the hydroxylamino-compound, which condenses with the protocatechuic alcohol; the condensation product is hydrolysed by means of acid to protocatechualdehyde, which is methylated by treatment with methyl sulphate in presence of sodium ethoxide. (2) Piperonal is treated with phosphorus pentachloride at 110°, phosphorus oxychloride and trichloride are distilled off, and the residue is hydrolysed to protocatechualdehyde by boiling with water. (3) From guaiacol, which is condensed with (*a*) hydroxylaminobenzenesulphonic acid, the product being hydrolysed as in (1); or (*b*) with nitrosodimethylaniline, whereby *p*-aminodimethylaniline is obtained as a by-product. Guaiacol may also be condensed with alloxan, which is prepared from guano by extraction of uric acid and treatment of this with chlorine; the condensation is effected by use of sulphuric acid. The product is hydrolysed by means of sodium hydroxide at 90°, and vanilloylcarboxylic acid obtained by treatment with sulphur dioxide: this is separated as the toluidine salt, which is decomposed by means of sodium hydroxide after elimination of carbon dioxide by boiling. The successive removal of isovanillin, resins, and colouring matters present in vanillin prepared from guaiacol is described, also the final purification by recrystallisation and drying. (4) *iso*Eugenol is prepared from potassium *isoeugenoxide* obtained by heating clove oil with alcoholic potash under pressure, the potassium compound after removal of alcohol by distillation being decomposed by 3% sulphuric acid below 3°; after careful purification, *isoeugenol* is acetylated, the acetyl derivative oxidised with ozone in pure xylene solution, and the vanillin extracted as the bisulphite

compound. *iso*Eugenol may be oxidised directly to vanillin by treatment of a fine suspension in water with ozone at low temperature. S. I. LEVY.

**Carbon disulphide.** DRAKELEY and BAKER.—See II. **Aniline sulphate and vulcanisation.** ZEITLIN and WILLIAMS.—See XIV. **Choline from beet molasses.** STANEK.—See XVII. **Industrial fermentations.** MAY and HERRICK. **Ethyl alcohol.** BACKHAUS. **Butyl alcohol and acetone by fermentation.** GABRIEL and CRAWFORD. **Lactic acid.** GARRETT.—See XVIII.

See also A., Dec., 1532, **Electrochemical preparation of formates from carbonic acid** (RABINOVITSCH and MASCHOWETZ). 1553, **Catalysts for formation of alcohols from carbon monoxide and hydrogen** (FROLICH and CRYDER). 1556, **Preparation and purification of ethyl acetate** (ROSHDESTVENSKI and others). 1563, **Preparation of glycine** (BOUTWELL and KUICK). **Electrolytic separation of arginine and alanine** (HENDRY and JOHNSON). **Preparation of prolylalanine and prolylpeptides** (FRANKEL and KUK). 1566, **Preparation of  $\psi$ -cumene** (SMITH and LUND). 1570, **Catalytic hydrogenation of aromatic nitro-compounds** (KOMATSU and AMATATSU). 1571, **Condensations of aromatic halogeno-nitro-compounds** (GALLAS and ALONSO). 1581, also 1587, **Perylene and its derivatives** (PONGRATZ: also ZINKE and others). 1621, **Acetone fermentation** (ZACHAROV).

#### PATENTS.

**Preparation of acetylides [of alkali metals].** DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 336,516, 23.1.30. Ger., 7.2.29).—A molten alkali metal is treated with purified acetylene, preferably diluted with inert gas, in a rotating tube, whereby fresh surfaces of metal are continually exposed to the gas. At 170—180° sodium gives the monosodium compound, at 210—220° the disodium compound. C. HOLLINS.

**Production of anhydrous alcohol.** O. LEONORI (U.S.P. 1,751,211, 18.3.30. Appl. 9.6.27. It., 26.6.26).—Anhydrous alcohol, suitable as fuel or solvent, is produced by saturating the aqueous alcohol with ammonia gas and adding a mixture of metal carbides decomposable by water together with a small amount of aluminium and calcium hydrides. After decantation of the anhydrous alcohol a small amount of alcohol is recovered from the residuary deposit by distillation and passing the alcohol vapour over layers of carbide. C. RANKEN.

**Production of urea.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,394, 29.7.29).—In distilling the mixture from the interaction of ammonia and carbon dioxide at high temperature and under a pressure of about 100 atm., fresh ammonia is pumped in during the process to maintain an excess, so as to avoid decomposition of the urea. W. J. WRIGHT.

**Condensation products [from benzoin, alcohols, and arylsulphonic acids] and their application [as dispersing and wetting agents].** SOC. CHEM. IND. IN BASLE (B.P. 319,249, 18.9.29. Switz., 18.9.28).—A benzene- or naphthalene-sulphonic acid is condensed, in any order, with benzoin and an alcohol in presence of an acid condensing agent (sulphuric acid, oleum, chlorosulphonic acid); an aromatic hydrocarbon may be

sulphonated and condensed with benzoin and an alcohol in the same or successive operations. Examples are: naphthalene, benzoin, and isopropyl, butyl, or ethyl alcohol. The products are wetting and dispersing agents, and may be used in preparation of lakes or of dye pastes. C. HOLLINS.

**Preparation of monoalkylxylenesulphonamide [xylenesulphonalkylamides].** IMPERIAL CHEM. INDUSTRIES, LTD., N. BENNETT, H. DODD, and W. C. SPRENT (B.P. 336,512, 17.1.30).—Xylenesulphonamide (from commercial xylene) is alkylated, *e.g.*, by heating in an autoclave with methyl or ethyl chloride and alkali. C. HOLLINS.

**Manufacture of arylides [arylamides] of aromatic hydroxycarboxylic [arylamino]salicylic acids.** W. W. GROVES. FROM I. G. FARBENIND. A.-G. (B.P. 336,428, 14.10.29).—The 4-arylamino]salicylic acids of B.P. 333,783 (B., 1930, 1016) are condensed with arylamines by the usual methods, *e.g.*, in presence of phosphorus trichloride or thionyl chloride and a solvent (toluene, xylene), to give arylamides having good affinity for the fibre. The following are described: 4-anilinosalicylic anilide, m.p. 186°,  $\beta$ -naphthylamide, m.p. 208°, dianisidide, m.p. above 340°, 1:5-naphthylene-diamide, m.p. 293°, *p*-nitroanilide, m.p. 211–212°, *p*-toluidide, m.p. 196°, *o*-anisidide, m.p. 123–125°; 4-*p*-chloroanilinosalicylic *m*-hydroxyanilide, m.p. 210–212°, 5-hydroxy-*o*-toluidide, *m*-nitroanilide, m.p. 203°, *p*-nitroanilide, m.p. 229°, *o*-chloro-*p*-nitroanilide, m.p. 240°, anilide, m.p. 222°, *o*-toluidide, m.p. 201°, *p*-toluidide, m.p. 223°, *o*-chloroanilide, m.p. 198°, *p*-chloroanilide, m.p. 188–190°, 2:5-dichloroanilide, m.p. 220–221°, 4-chloro-*o*-toluidide, m.p. 197°, 5-chloro-*o*-toluidide, m.p. 183°, *p*-hydroxyanilide, m.p. 165–166°, *o*-anisidide, m.p. 142°, 5-chloro-*o*-anisidide, m.p. 178–180°, *p*-anisidide, m.p. 178–179°, *p*-phenetide, m.p. 165°, 5-nitro-*o*-toluidide, m.p. 203–204°, 4-nitro-*o*-anisidide, m.p. 239–240°,  $\beta$ -naphthylamide, m.p. 237–239°,  $\alpha$ -anthraquinonylamide, m.p. 240–243°,  $\beta$ -anthraquinonylamide, m.p. 298–299°, 5-amino- $\alpha$ -anthraquinonylamide, m.p. above 330°, and 3-carbazolylamide, m.p. 219°; anilides, nitroanilides,  $\beta$ -naphthylamides, etc. of 2', 3', 4'-methyl, 2'- and 3'-chloro-, 2'- and 4'-methoxy-, 4'-phenoxy-, 2': 4'- and 2': 5'-dimethyl-, 4'- and 5'-chloro-2'-methyl-, 4'-chloro-3'-methyl-, 3'-chloro-4'-methyl-, 4'-methoxy-3'-methyl-, 2': 4'- and 3': 4'-dichloro-, and 3': 4'-dichloro-6'-methyl derivatives of 4-anilinosalicylic acid are also described, with m.p. C. HOLLINS.

**Manufacture of arylaminophenolcarboxylic acid [5-arylamino]salicylic acids].** O. Y. IMRAY. FROM I. G. FARBENIND. A.-G. (B.P. 336,394, 27.9.29).—An alkali salt of a *p*-arylamino]phenol is treated with carbon dioxide under pressure (10 atm.) at 150–170°. Examples are 5-arylamino]salicylic acids from 4-hydroxydiphenylamine (m.p. 153°) and its 4'-methyl (m.p. 165–166°), 4'-methoxy- (m.p. 144–145°), 3-methyl (m.p. 195–197°), 3:4'-dimethyl (m.p. 207–210°), 2'-methyl (m.p. 139°), and 3'-methyl (m.p. 212°) derivatives. C. HOLLINS.

**Manufacture of 1-phenyl-2-methylamino-1-propanol [ $\beta$ -methylamino- $\alpha$ -phenylpropyl alcohol].** KNOLL A.-G. CHEM. FABR., and W. KLAVERN (B.P. 336,412, 4.10.29. Ger., 30.7.29).—Benzoylacetyl is

reduced (*e.g.*, with aluminium amalgam and water) in presence of methylamine in ether to give "diephedrine,"  $\text{OH} \cdot \text{CHPh} \cdot \text{CHMe} \cdot \text{NHMe}$ , m.p. 185–186°.

C. HOLLINS.

**Manufacture of aryl [naphthyl]alkyl ethers which are alkylated in the nucleus and of their sulphonic acids [wetting-out agents].** M. KAHN and A. THAUSS, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,781,981, 18.11.30. Appl., 21.6.26. Ger., 27.6.25).—See B.P. 277,098; B., 1927, 860.

**Preparation of carbazole-indophenol compounds.** F. BALLAUF, F. MUTH, and A. SCHMELZER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,781,966, 18.11.30. Appl., 6.11.26. Ger., 24.1.24).—See B.P. 243,557; B., 1926, 121.

**Oxidation of hydrocarbons (B.P. 337,407).**—See II. Acetylene (B.P. 338,003). Reactivation of catalysts (B.P. 334,251).—See VII. Sulphonated fatty acids etc. (B.P. 312,283).—See XII. Quebrachitol from rubber latex serum (U.S.P. 1,758,616).—See XIV. Acetone and butyl alcohol by fermentation (B.P. 315,002).—See XVIII.

#### IV.—DYESTUFFS.

**Azo dye in vulcanisation.** DRAKELEY and COULSON. —See XIV.

See also A., Dec., 1498, Adsorption spectra of organic dyes (AUSCHKAP). 1571, Condensations of aromatic halogeno-nitro-compounds (GALLAS and ALONSO). 1573, Coupling of diazonium compounds with 2-methoxy-3-naphthoic acid (JAMBUSERWALA and MASON). 1581, Perylene and its derivatives (PONGRATZ). Fungus dyes (KÖGL and others). 1587, Perylene and its derivatives (ZINKE and others). 1589, 1-Hydroxyphenanthraquinone (BRASS and others). 1593, Preparation of thioindigo dyes (DZIEWOŃSKI and others).

#### PATENTS.

**Production of dibenzanthrone derivatives [vat dyes].** I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 336,268, 9.5.29).—3:3'-Dibenzanthronyls carrying as substituents phenoxy-, nitro-, or simple amino-groups, but having 2- and 2'-positions free, are fused with alkali to give vat dyes. Examples are: phenoxydibenzanthronyl (from 3:3'-dibenzanthronyl, chlorinated with sulphuryl chloride and treated with phenol and alkali), which gives a reddish-violet vat dye; the mono- and di-nitro-compounds, prepared from 3:3'-dibenzanthronyl and mixed acid, give, respectively, a greenish-blue and a greenish-black; the monoamino-compound gives a bluish-red.

C. HOLLINS.

**Manufacture of fast [acid] azo dyes [for wool].** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 335,893, 30.4.29).—The diamine,  $\text{P}[\cdot\text{ArNH}_2]_2$ , obtained according to B.P. 313,421 (B., 1929, 746) by condensing a cyclohexanone with an arylamine is tetrazotised and coupled with a coupling component, particularly 2:8-dihydroxynaphthalene-6-sulphonic acid or its monoaryl-sulphonate, or a naphtholdisulphonic acid containing as substituents an amino-, acylamino-, or second hydroxyl



group (the last-named may be esterified) with an arylazo group. Examples are: product from *cyclohexanone* and *o*-toluidine with 2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid (bluish-red, fast to light, washing, milling, and perspiration), or with 1 mol. of *p*-nitroaniline  $\rightarrow$  H-acid and 1 mol. of salicylic acid (bluish-green, yellower by after-chroming), or with 1 mol. of  $\gamma$ -acid and 1 mol. of NW-acid, diazotised,  $\rightarrow$   $\beta$ -naphthol (intense red); product from *cyclohexanone* and aniline with 1 mol. of 1:3:6-naphtholdisulphonic acid and 1 mol. of NW-acid (bluish-red), or with 1 mol. of phenyl- $\gamma$ -acid and 1 mol. of *m*-phenylenediamine-4-sulphonic acid (brown); product from *cyclohexanone* and *o*-anisidine with 1 mol. of H-acid, diazotised, and coupled with 2 mols. of 4-nitro-*m*-phenylenediamine (brown). C. HOLLINS.

**Manufacture of acid dyes of the phenonaphthosafranin series.** P. LAEUGER, Assr. to J. R. GEIGY Soc. ANON. (U.S.P. 1,782,356, 18.11.30. Appl., 25.10.28. Ger., 14.11.27).—See B.P. 300,549; B., 1930, 364.

**Dispersing agents** (B.P. 319,249).—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Crude fibre: investigation of the "Dutch" process.** S. GOLDBERG (Quart. J. Pharm., 1930, 3, 375—389).—From experiments with *Podophyllum* rhizome and other materials, it is shown that the so-called "Dutch" process, as modified by Wallis (Pharm. J., 1919, 103, 76), can be applied to obtain consistent quantitative results if the time of digestion with nitric acid be lengthened from 30 sec. to 15 min., and careful attention be paid to regulation of temperature and rate of stirring during the digestion. The difficulty of filtering the fibre after alkali treatment is overcome by adding a third treatment with 1% sulphuric acid, which does not alter the weight of the fibre; 1% nitric acid is unsuitable, since it acts on the cellulosic constituents which have been altered by the action of alkali, with formation of mucic acids, high results being consequently obtained. R. CHILD.

**Lustre of textile fibres and a method of measurement.** M. O. PELTON (Trans. Optical Soc., 1930, 31, 184—200).—The distribution of light from a lustrous surface has been investigated with a specially constructed photometer. Lustre is due to the concentration, in certain directions, of reflected light from a unidirectional beam. In calculation, it is assumed that an ideal filament is a perfectly polished cylinder of a perfectly transparent homogeneous and isotropic medium.

C. W. GIBBY.

**Nitrated cellulose.** MILES and others.—See XXII.

See also A., Dec., 1500, **Fluorescence of cellulose acetate and nitrate in ultra-violet light** (McNALLY and VANSELOW). 1610, **Waxy substance of cocoon silk fibres** (MASAMI).

### PATENTS.

**Production of waterproof fabrics.** BRIT. CELANESE, LTD. (B.P. 338,065, 15.1.30. U.S., 15.1.29).—A suitable fabric base is coated on one or both sides with a solution or paste containing rubber, and, after vulcanising the latter, is further treated with a solution containing a cellulose derivative and a synthetic resin compatible

therewith, e.g., of the diphenylolpropane-formaldehyde type, together with plasticisers and effect materials.

F. R. ENNOS.

### Manufacture of artificial products from wood.

A. CARPMÆL. From I. G. FARBENIND. A.-G. (B.P. 337,791, 7.8.29).—Hard wood, which has been disintegrated and degummed, is treated below 30° with acetic anhydride with or without glacial acetic acid, and in the presence of 8—14% (calc. on wt. of wood) of sulphuric acid, or the equivalent amount of another acid catalyst; when the swollen mass no longer shows the fibrous structure, it is worked up in an analogous manner to that of the manufacture of cellulose acetate.

F. R. ENNOS.

**Production and treatment of textile and other materials made of or containing cellulose.** H. DREYFUS (B.P. 335,894, 31.5.29).—In place of mineral acids, carboxysulphonic acids and other organo-mineral acids containing carboxyl groups are used for precipitation of viscose. The best results are obtained with 60% aqueous sulphoacetic acid. C. HOLLINS.

### Manufacture of artificial filaments and the like.

BRIT. CELANESE, LTD. (B.P. 318,630, 5.9.29. U.S., 8.9.28).—In the manufacture of artificial horsehair, bristles, straw, etc. of denier 20—2000 from organic derivatives of cellulose by the evaporative method, the heavy filaments in the heated meter casing, while still containing solvent, are supported by passing over or round one or more heated feed devices, which rotate each with a linear speed greater than the one preceding.

F. R. ENNOS.

**[Photomechanical] production of paper showing the grain of wood.** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 319,000, 23.8.29. Ger., 15.9.28).

**Treatment of paper and like surfaces [for modifying the light reflected by them].** W. W. TRIGGS. From NAT. PAPER PROCESS CO., INC. (B.P. 338,168, 14.8.29).

**Heat- and sound-proof articles** (B.P. 315,280).—See I. **Treatment of waste liquors** (B.P. 337,533).—See VII. **Building blocks** (U.S.P. 1,760,446). **Uniting wood and paper** (B.P. 319,001).—See IX. **Cable insulation** (B.P. 337,052).—See XI. **Rubber dispersions** (U.S.P. 1,749,607—8).—See XIV. **Tanning substances** (B.P. 336,984).—See XV. **Artificial manure** (B.P. 311,405).—See XVI. **Nicotine from paper** (B.P. 337,311).—See XX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Weatherproof braidings.** KINDSCHER.—See XI.

### PATENTS.

**Printing with the aid of stable reduction compounds of [thioindigoid] vat dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,907, 11.5.29).—Printing pastes containing the stable reduction products of 6:6'-dihalogeno-4:4'-dimethyl- or 6:6'-diethoxythioindigo prepared according to B.P. 334,878, 334,919—20 (B., 1930, 1144), with or without a reducing agent, give full-strength shades when after-treated in the usual manner. C. HOLLINS.

Fluid treatment of textile yarns and threads in the form of compact wound packages. J. BRANDWOOD (B.P. 338,131, 7.8.29).

Wetting agents (B.P. 319,249).—See III. Colouring of rubber (B.P. 331,688 and 336,446).—See XIV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Reconcentration of sulphuric acid-nitric acid baths. A. ALBERTO (Ann. Acad. Brasil. Sci., 1930, 2, 113—129).—The method of calculating the quantities of nitric and sulphuric acids of any given concentrations required to be added to a spent nitrating bath in order to bring it to a specified composition is described.

H. F. GILLBE.

B.-p. and distillation curves of hydrochloric acid-water mixtures. E. BERL and H. STAUDINGER (Z. angew. Chem., 1930, 43, 1019—1022).—These have been determined for concentrations up to 62.73% HCl. The temperature-vapour composition curve falls to 100% HCl at  $-14^{\circ}$ , whereat the liquid phase contains 62% HCl. The curve showing the relationship between the composition of the liquid and that of the vapour differs appreciably from that obtained by other workers: the smooth curve shows no indication of the formation of a decalhydrate, whilst the vapour in contact with the 42% acid contains 2.1% of water. The constant-boiling mixture has b.p.  $110^{\circ}$ , and contains 20.17% HCl.

H. F. GILLBE.

Cold lixiviation [of caliche] at Oficina "N." G. LEIMBACH and M. WARNECKE (Caliche, 1930, 11, 439—454).—Analyses are given of a number of samples of caliche worked at Oficina "N." and the methods available for the extraction of the nitrate are described. Owing to the nature of the raw material, the direct process, in which spent liquor is returned to the dilute liquor obtained by cold lixiviation, is impracticable, and it appears to be preferable to use part at least of the spent liquor for hot lixiviation.

H. F. GILLBE.

Caliche of Taltal and Oficina "N," with special reference to the cold lixiviation process practised at Taltal. G. LEIMBACH (Caliche, 1930, 11, 448—457).—Details are given of the lixiviation of two widely differing types of caliche.

H. F. GILLBE.

Lixiviation with dilute liquors at various temperatures, with special reference to the treatment of "fines" rich in sodium chloride. G. LEIMBACH and M. WARNECKE (Caliche, 1930, 11, 500—506).—Details are given of the treatment of dilute liquors obtained by lixiviation of caliche at  $25^{\circ}$ ,  $50^{\circ}$ , and  $82^{\circ}$ , and the economic aspect of the processes is discussed.

H. F. GILLBE.

Possibilities of obtaining saturated solutions from a given caliche, deduced from system  $\text{NaNO}_3$ - $\text{NaCl}$ - $\text{Na}_2\text{SO}_4$ - $\text{H}_2\text{O}$  at  $25^{\circ}$ . G. LEIMBACH (Caliche, 1930, 11, 458—464).—An account is given of the method of determining, by application of the phase relationships in the above system, the nature of the saturated solutions obtainable from a given type of caliche.

H. F. GILLBE.

Influence of extraneous salts on the hygroscopic properties of commercial sodium nitrate. A.

PFEIFFENBERGER (Caliche, 1929, 11, 353—355).—The influences of certain salts on the rate of absorption of moisture from air by sodium nitrate have been investigated by determining the increase of weight of samples of the material when exposed to moist air for periods up to 52 days. Sodium chloride increases the quantity of water absorbed in a given time to a much greater extent than does sodium sulphate, and the two salts when simultaneously present cause an even greater increase. Magnesium nitrate has a greater influence than the sodium salts, whilst a mixture of magnesium nitrate and chloride causes a six-fold increase of the quantity of water absorbed, as compared with the original salt.

H. F. GILLBE.

Conversion of Chile saltpetre into potassium nitrate. KÜPPER (Caliche, 1930, 11, 466—486).—A general account is given from the viewpoint of the phase rule of the conversion of natural sodium nitrate into potassium nitrate by means of potassium chloride or sulphate, the relevant data being tabulated. The technical procedure and economics of the chloride process are discussed. The potash deposits of Pintados, although relatively rich in potassium salts, cannot economically be employed alone for nitrate production, but the Taltal sulphate deposits lend themselves to satisfactory working, with recovery of all valuable by-products, without requiring any considerable quantity of added heat, since all the reactions involved take place either at the ordinary or at but slightly raised temperatures.

H. F. GILLBE.

Hydrolysis of sodium chloride by steam in presence of silicic acid. E. BERL and H. STAUDINGER (Z. angew. Chem., 1930, 43, 1006—1009).—Reasonable yields of silicate are obtained by the action of steam on sodium chloride-silica mixtures only at temperatures above  $1000^{\circ}$ , and the evolution of hydrochloric acid is aided by addition of wood charcoal to the charge and by using a 2:1 mixture of steam and air. At  $1250^{\circ}$  about 90% of the chloride is converted into silicate within 9 hrs., and a further 3 hrs. are necessary for a 97% conversion; loss of sodium chloride by volatilisation may be reduced by working at a slightly lower temperature. The possible technical development of the process, with simultaneous generation of water-gas, is described, the main difficulty being the economic concentration of the very dilute hydrochloric acid (1—2%) which is produced.

H. F. GILLBE.

Analysis of phosphates. V. VINCENT (Ann. Falsif., 1930, 23, 475—481).—The phosphate (0.75 g.) is extracted with ammonium citrate for 1 hr. at  $50^{\circ}$ . An aliquot portion of the solution is then precipitated with ammonium molybdate. The precipitate is collected, washed, and dissolved in a known volume of standard caustic soda, the excess of soda being determined by titration with standard acid, using phenolphthalein as indicator. From the volume of soda required for the dissolution of the phosphomolybdate the %  $\text{P}_2\text{O}_5$  is calculated.

B. W. TOWN.

Rapid determination of sulphur dioxide in air. K. ZEPF and F. VETTER (Mikrochem., Emich Festschr., 1930, 280—288).—Sulphur dioxide in air at dilutions of  $1:10^5$  to  $1:10^7$  is absorbed in iodine-potassium iodide-

starch solution in a specially designed apparatus: details are given of the colorimetric determination of the liberated iodine. The apparatus is portable and a determination may be completed within 2--6 min.

H. F. GILLBE.

**Physics and the chemical industry.** GMELIN. **Bulking of particles.** ROLLER. **Acid pumps.** ZIMMER.—See I. **Kjeldahl decompositions. Determination of phosphoric acid.** LEPPER.—See XIX. **Phosphorus solutions.** SITSSEN. **Determination of iron in scale etc.** FERREY.—See XX. **Light filters.** LUTHER.—See XXI.

See also A., Dec., 1532, **Electrochemical preparation of sodium hyposulphite** (RABINOVITSCH and FOKIN). 1550, **Alumina as desiccant** (BARNITT and others).

#### PATENTS.

**Manufacture of sulphuric and nitric acids.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,406, 13.6.29. Addn. to B.P. 301,232; B., 1929, 431).—The gas mixture containing oxides of sulphur and of nitrogen, obtained as described in the prior patent, is passed into absorption towers fed with sulphuric or nitrosylsulphuric acid in presence of so much nitrogen oxide that all the sulphur dioxide is converted, with the production of sulphuric or nitrosylsulphuric acid, or a mixture of sulphuric and nitric acids. The nitrogen oxides are recovered from the residual gas in the form of nitric acid. [Stat. ref.]

W. J. WRIGHT.

**Manufacture of concentrated nitric acid.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 336,638, 17.4.29).—Gases containing nitrogen oxides, such as are produced in the arc process or in the oxidation of ammonia, are led under pressure into the bottom of a tower, each section of which comprises a base plate with a liquid-sealed central opening and an overflow cup communicating with the plate below. Layers of nitric acid, gradually decreasing in concentration towards the top of the tower, are maintained on the plates, the uppermost plate being supplied with water. As the gases bubble through the acid, nitrogen dioxide is absorbed, the nitric oxide produced being oxidised to dioxide, which again is absorbed in the next section. Substantially all of the nitrogen oxides are thus removed from the gases.

W. J. WRIGHT.

**Manufacture of finely-crystalline soda [sodium carbonate].** W. MANN (B.P. 337,401, 30.7.29).—Finely-ground Solvay soda is mixed in an edge-runner mill with water, this being added as fast as it is absorbed.

W. J. WRIGHT.

**Leaching of phosphate rock.** KUNSTDÜNGER-PATENT-VERWERTUNGS A.-G. (B.P. 338,079, 5.2.30. Swed., 7.3.29).—After treatment of phosphate rock with nitric acid and precipitation of the lime with potassium sulphate, the solution, containing potassium nitrate and phosphoric acid, is partly freed from the nitrate by cooling and crystallisation. It is then neutralised with ammonia, evaporated, and cooled to cause crystallisation of the ammonium phosphate, the mother-liquor being used as a diluent in subsequent lime precipitations. The formation of insoluble double salts is thus avoided.

W. J. WRIGHT.

**Manufacture of superphosphate and the like products.** D. WELLSCH (B.P. 338,075, 31.1.30).—A reaction chamber for the continuous production of superphosphate comprises a cylindrical vessel, which can rotate on a vertical axis round a stationary inner hollow cylinder, attached to the cover carrying the mixer. In the intervening space is a transverse partition, attached to the cover and to a wall of either the outer chamber or the inner cylinder, and communicating with this inner cylinder are a discharge device, consisting of movable knives, and a conveyor. Superphosphate in a fluid condition, admitted from the mixer into the outer vessel while this is stationary, forms a layer on the bottom extending from the partition to the discharge device. When operation commences, this layer prevents liquid superphosphate from prematurely reaching the discharge and the conveyor, the solidified and dissociated material being gradually broken up by the discharging knives and carried through a gap in the base of the inner cylinder to the conveyor. The reaction chamber may be of spherical form, or may consist of a cylindrical vessel rotatable on a horizontal axis, in which latter case the inner cylinder serves as the barrier, and the dried material is ultimately discharged through a longitudinal slot in the cylinder.

W. J. WRIGHT.

**Production of monocalcium phosphate and mixtures of mono- and di-calcium phosphate.** F. C. and F. PALAZZO (B.P. 316,550, 19.7.29. It., 30.7.28).—Superphosphate is lixiviated at ordinary temperature with about half its weight of water so as to give a concentrated extract with a high proportion of monocalcium phosphate and free phosphoric acid. This is separated from the insoluble residue, the latter being treated with water, and the phosphoric acid in the extract precipitated with milk of lime. The di-calcium phosphate obtained is caused to react with the first extract, the pasty monocalcium phosphate produced being finally dried in a vacuum dryer.

W. J. WRIGHT.

**Working up of crude potash salts.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 316,548, 15.6.29. Ger., 30.7.28).—In an application of the process described in B.P. 311,226 (B., 1930, 1150) to crude potash salts of any composition, the salts are treated with the mother-liquor, consisting of ammoniacal ammonium chloride solution, subsequent treatment depending on the original composition of the crude salts.

W. J. WRIGHT.

**Treatment of waste liquors [containing alkali sulphide].** COURTAULDS, LTD., R. S. JONES, and S. PEARSON (B.P. 337,533, 23.9.29 and 11.2.30).—The liquors are treated with a compound containing the  $\text{SO}_2$  radical (e.g., a sulphite, bisulphite, or the gas itself), a compound of zinc or magnesium, and sufficient acid to give a neutral or slightly acid reaction. Precipitated sulphur is allowed to settle, and caustic alkali is added until the mixture is alkaline, the liquor being finally passed through tanks in which the precipitated zinc hydroxide and the remainder of the sulphur settle out.

W. J. WRIGHT.

**Manufacture of lime-nitrogen [calcium cyanamide].** ELEKTROCHEM. GES.M.B.H., and H. GROHMANN

(B.P. 336,677, 20.7.29).—Channels are formed in the mass of calcium carbide by inserting cardboard tubes into the powdered carbide and leaving them until the reaction temperature (800°) is reached. As fusion occurs before this point, the channels persist after carbonisation of the tubes. The content of free carbide in the resultant cyanamide is appreciably reduced by this method. W. J. WRIGHT.

**Manufacture of metal carbamates.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,749, 19.9.29).—Metal carbamates are prepared without the use of solvents by causing ammonium carbamate to react with finely-divided, dry, metal oxides at a temperature not above 100°, and at normal, reduced, or increased pressure. The reaction may be carried out in a current of ammonia, air, or nitrogen. W. J. WRIGHT.

**Preparation of barium carbide and application to the manufacture of acetylene and barium hydrate in continuous working.** INTERNAT. INDUSTRIAL & CHEM. CO., LTD. (B.P. 338,003, 18.11.29. Fr., 21.11.28).—In the reaction of barium carbonate with carbon, an inert substance such as dibarium silicate is added, thus enabling the mixture to be heated to 1600° without fusion or loss of porosity. The mixture is preferably heated in a current of hydrogen or methane or a mixture of carbon monoxide and hydrogen. If the barium carbide is used to produce acetylene, the residue may be treated to remove the barium hydroxide and freed from excess of silica, and, after adding more carbon and barium carbonate, returned to the carbide furnace. W. J. WRIGHT.

**Manufacture of tungsten and like carbides.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF F. C. KELLEY (B.P. 319,698, 25.9.29. U.S., 26.9.28).—A powdered mixture of 94% of tungsten and 6% of carbon is placed in a porous carbon receptacle closed by a carbon plug. The receptacle is heated for about 3 hrs. at 1500° in an electric furnace, a current of hydrogen being caused to flow over it at such a rate that no hydrogen entering the receptacle remains uncombined with carbon. The receptacle is then pushed into a water-jacketed chamber and allowed to cool to room temperature. W. J. WRIGHT.

**Production of zinc salt solutions.** METALLGES. A.-G. (B.P. 334,458, 29.11.29. Ger., 7.12.28. Addn. to B.P. 302,924; B., 1930, 508).—The zinciferous residues obtained in the prior process are leached with a large excess of acid. The solution is then treated with hydrogen sulphide or metal sulphides (preferably zinc sulphide, or, when much arsenic and antimony are present, ferrous sulphide). The liquid is added, if desired, to the liquor obtained as described previously, and is freed from iron. The latter may be precipitated, preferably after oxidation to the ferric state, *e.g.*, by neutralising the liquid with zinc oxide, or with raw zinciferous material in accordance with the former process, in which cases the resulting liquor may be added to the liquor obtained according to the main patent. The ferrous or zinc sulphide employed may be recovered by suitable treatment of the by-products. S. K. TWEEDY.

**Manufacture of artificial zeolites.** REYMERSHOLMS

GAMLA INDUSTRI AKTIEB. (B.P. 313,522, 11.6.29. Swed., 13.6.28).—Natural silicates (*e.g.*, kaolin) or more or less refractory clays are decomposed with acid (*e.g.* mineral acid, such as sulphuric) and/or acid alkali salt. The residue is boiled under pressure with oxides, hydroxides, or carbonates of the alkali and alkaline-earth metals in admixture, the proportions being such that porous solid silicates (artificial zeolites) are formed. Hydration of the product occurs during the boiling. The artificial zeolite, prior to employment for base-exchange purposes, may be washed with an alkali salt solution until lime-free reaction is obtained. S. K. TWEEDY.

**Preparation of base-exchanging materials.** W. W. TRIGGS. From GEN. ZEOLITE CO. (B.P. 337,768, 27.7.29).—One portion of the aluminium in the final product is derived from aluminium hydroxide in a non-alkaline, and the other from the hydroxide in an alkaline, combination, *e.g.*, a solution of aluminium sulphate containing sulphuric acid is mixed with sodium silicate solution, and to the mixture is added sodium aluminate solution. After the gel has formed it is dried only to a point at which it assumes a rigid structure, and the soluble salts are then removed by washing, so that there is no substantial alteration of the original structure. [Stat. ref.] W. J. WRIGHT.

**Production of catalytic material [containing vanadium].** IMPERIAL CHEM. INDUSTRIES, LTD., and J. A. WEIL (B.P. 334,268, 1.6.29).—The material, particularly adapted for the large-scale catalytic oxidation of sulphur dioxide, comprises a physical mixture of a vanadium compound with a silicious diluent (*e.g.*, kieselguhr, sand) and a silicious hydrogel. The mixture may be formed into pellets or masses, which are then dried and treated with burner gas at 450–500°. The vanadium compound, which is preferably a soluble vanadate, vanadite, or vanadyl compound (*e.g.*, potassium metavanadate, ammonium vanadyl oxalate), may also be mixed with a silicious material of which a part at least is in the form of a wet hydrogel. S. K. TWEEDY.

**[Reactivation] treatment of catalyst masses.** BRIT. CELANESE, LTD., W. BADER, and E. B. THOMAS (B.P. 334,251, 31.5.29).—Catalysts (*e.g.*, oxides such as zinc oxide, alone or admixed with metals or metal oxides) used for synthesising methyl alcohol or other oxygenated organic compounds from mixtures containing carbon monoxide and hydrogen, or carbon monoxide and aliphatic alcohols, esters, or ethers, are reactivated by treatment, at temperatures not much above those employed in the catalytic reaction, with hydrogen or a gas mixture rich in hydrogen (*e.g.*, richer than the mixture employed in the synthesis), preferably under pressure (100 atm.) S. K. TWEEDY.

**Manufacture of chromium oxides suitable as pigments and the like.** A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 336,671, 19.7.29).—An alkali chromate or dichromate, *e.g.*, the crude lye from chromate manufacture, freed from iron, is reduced at 105–120° by the action of sulphur which is present in solution or which is partly in suspension initially, but subsequently passes into solution by the action of sodium hydroxide, carbonate, sulphide, or polysulphide. The

chromium hydroxide thus formed is ignited, giving different shades of chromic oxide according to the temperature of reduction. The spent lyes after reduction are worked up for further use, *e.g.*, by crystallising out sodium thiosulphate, treatment with sulphuric acid, etc. S. S. WOOLF.

**Working up of oxides of nitrogen.** N. CARO and A. R. FRANK (B.P. 337,847, 13.8.29).—In the conversion of nitrogen oxides in gas mixtures, such as those obtained from the oxidation of ammonia, part of the water vapour is first removed without oxidation of the nitrogen oxides, this being effected at a high discharge temperature. After oxidation of the nitric oxide, promoted by lowering the temperature, the residual water vapour is separated as aqueous nitric acid, and on further cooling the gas mixture nitrogen tetroxide is produced. The condensates obtained in the second and third phases may be combined to give a mixture which is suitable for producing highly concentrated nitric acid. W. J. WRIGHT.

**Production of pure sulphur.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,395, 29.7.29).—The decomposition of ammonium polysulphide solutions at temperatures above the m.p. of sulphur, as described in B.P. 269,546 (B., 1928, 91), is carried out in presence of adsorbent substances, *e.g.*, silica gel, the sulphur thus obtained having a pure yellow colour. W. J. WRIGHT.

**Manufacture of sulphuric acid.** H. PETERSEN (U.S.P. 1,781,954, 18.11.30. Appl., 24.2.27. Ger., 20.3.26).—See B.P. 267,885; B., 1928, 332.

**Conversion of salts into globular or similar shaped bodies.** B. HAAK, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,782,038, 18.11.30. Appl., 19.11.28. Ger., 13.12.27).—See B.P. 313,652; B., 1929, 680.

**Gas separation process. [Manufacture of hydrogen by liquefaction.]** G. CLAUDE, Assr. to Soc. L'AIR LIQUEFIE, Soc. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,782,287, 18.11.30. Appl., 8.5.25. Fr., 3.6.24).—See B.P. 235,129; B., 1925, 672.

**Porous masses** (B.P. 336,318).—See VIII. **Artificial ice** (B.P. 336,218).—See IX. **Lithopone** (B.P. 336,839).—See XIII. **Fertilisers** (B.P. 336,692).—See XVI.

## VIII.—GLASS; CERAMICS.

**Air-conditioning in ceramics.** II. B. MATZEN and C. E. PARMELEE (J. Amer. Ceram. Soc., 1930, 13, 856—864).—The general factors involved in air-conditioning in complete drying of ware; partial drying of material for working and forming; influencing the atmosphere in the casting and decoration shops; improving the conditions of health, are outlined. J. A. SUGDEN.

**Rapid-fire tunnel kiln designed for small production.** A. L. DONNENWIRTH (J. Amer. Ceram. Soc., 1930, 13, 865—870).—A miniature oil-fired tunnel kiln designed to fire 10,000 spark-plug porcelains daily, to cone 17, with a firing time of about 2 hrs. is described, and details of its efficiency and method of operation are given. J. A. SUGDEN.

**Aluminium fluoride in cast-iron enamels.** A. L. GORMLY (J. Amer. Ceram. Soc., 1930, 13, 900—902).—Aluminium fluoride can be substituted for cryolite if the fluxes are increased proportionately with the additional alumina introduced by the fluoride. Fluorspar can, in turn, replace the aluminium fluoride. J. A. SUGDEN.

**Boric acid in the glaze of the sealed vases of Arezzo.** R. NASINI (Nature, 1930, 126, 877—878).—Analyses now show that boric acid is a constituent of this glaze. L. S. THEOBALD.

**Vapour-glazing and colour-flashing of pre-Cambrian shales.** H. L. LONGENECKER (J. Amer. Ceram. Soc., 1930, 13, 794—804).—Success of vapour-glazing with zinc depends on vitrifying the brick in an oxidising atmosphere and the use of pure metal on clean fires at a maximum temperature. Scrap zinc gives a yellowish-green glaze. Moss zinc is recommended as being pure and in an easily volatilised form. The kiln should be sealed until the zinc vapour has been completely absorbed. Salt is not necessary to produce a green colour, but does give a glossy surface. The quantity of zinc required depends on the particular shale. Also in colour-flashing, the brick should be vitrified in an oxidising atmosphere and the mixture of coal tar and slack should be thrown on clean white-hot fires. The addition of salt to the tar-slack mixture gives a glossy black instead of a purplish-black. In both cases maximum colour is developed by cooling under reducing conditions. Pre-Cambrian, but not Triassic, shales give good colours by both vapour-glazing and colour-flashing. J. A. SUGDEN.

**Determination of the firing range of vitreous enamels used in wet-process enamelling.** C. J. KINZIE (J. Amer. Ceram. Soc., 1930, 13, 896—899).—Trial plates are held in the furnace for a given length of time, either alone or along with a standard enamel. The temperature is raised or lowered until the enamel ceases to be stable to heat or fails to mature. In this way it is found that enamels vary greatly in behaviour. Two enamels may mature to equal lustre in the same time at 900°, but one may fail to mature at 800° and the other may mature at as low as 700°. J. A. SUGDEN.

**Causes for inconsistent results in commercial fineness tests of porcelain enamels.** H. G. WOLFRAM (J. Amer. Ceram. Soc., 1930, 13, 884—886).—To obtain consistent results in sieve tests it is necessary to use selected wire cloths, avoid distortion during soldering to frames, and to retain a standard sieve and sample for calibration of new sieves. J. A. SUGDEN.

**Cause and prevention of certain brown stains on semi-vitreous tableware.** G. A. LOOMIS (J. Amer. Ceram. Soc., 1930, 13, 871—873).—Brown stains which appeared on the ware when embedded in a certain sand and fired in a reducing atmosphere were traced to the adsorption of iron from the sand. The trouble was prevented by firing with an excess of air, in saggars with holes to permit better circulation of air round the ware during oxidation. With closed saggars more time must be allowed at the oxidation temperatures. J. A. SUGDEN.

**Simple consistency pipette for use in the adjustment and control of enamel slips.** C. J. KINZIE and C. H. COMMONS (J. Amer. Ceram. Soc., 1930, **13**, 894—895).—An easily reproducible pipette with metal body and tip and a glass mouthpiece is described.

J. A. SUGDEN.

**Determining the tensile strength of unfired clays.** S. M. PHELPS and V. CARTWRIGHT (J. Amer. Ceram. Soc., 1930, **13**, 845—849).—The cylindrical test piece has a collar of low-melting alloy cast round each end. This eliminates the usual gripping difficulty and greatly increases the accuracy.

J. A. SUGDEN.

**Transverse strength of ball clay-sand and ball clay-flint mixtures.** L. P. COLLIN (J. Amer. Ceram. Soc., 1930, **13**, 876—883).—In comparing the plasticities of clays by measuring the transverse strengths of mixtures with non-plastic materials, it was found that fine potter's flint gave much better results than coarse silica sand. It is suggested that further consideration be given to the method of forming test-bars and to the rate of loading.

J. A. SUGDEN.

**Carbon and sulphur content of certain Ohio common clays used in heavyware industries.** G. M. MACHWART and J. R. WITHROW (Ind. Eng. Chem., 1930, **22**, 1224—1225).—A chemical survey of 27 clays shows that manufacturers select clays low in sulphur (av. 0.12%) and carbon (av. 0.91%), in order to be free from firing troubles. Analytical methods are given.

J. A. SUGDEN.

**Correcting the tenderness of a shale by chemical treatment.** H. E. SIMPSON (J. Amer. Ceram. Soc., 1930, **13**, 817—822).—The effect of the addition of various acids, alkalis, and organic binders on the properties of a shale were studied. Although such properties as rate of settling, water of plasticity, strength, and adsorption could be greatly modified, it was found that the most practical expedient to increase the strength and machining properties was finer grinding and ageing.

J. A. SUGDEN.

**Altering the properties of heavy clays by the use of electrolytes.** T. HUSAIN (J. Amer. Ceram. Soc., 1930, **13**, 805—816).—The effects on the properties of three clays of the addition of hydrochloric and tannic acids, sodium carbonate, and calcium chloride were studied both in the laboratory and on the plant. Measurements of settling time, slaking time, viscosity of slips, amount of pug water required, drying rate and behaviour, drying shrinkage, and porosity were made. Results indicate that although the properties of a clay can be altered over a wide range, no laboratory test can decide what addition is necessary. The effects are specific to both the clay and the electrolyte, some clays being more sensitive than others. The plant application of a 1% solution of hydrochloric acid has reduced the drying losses with a certain clay from 13% to 3%.

J. A. SUGDEN.

**Casting slip.** L. SAMPLE (J. Amer. Ceram. Soc., 1930, **13**, 874—875).—Viscosimetry is not a reliable control of consistency. In the cup test an actual casting is made in a small mould kept at constant moisture content. After draining, the thickness of the piece is measured and this value gives the rate at which the slip

will "set up." Working the piece in the fingers detects small changes in the "hardness" of the slip. It was found better to vary the ratio of sodium silicate and "sal soda" than to vary the amount of a standard electrolyte containing the two. Increase in sodium silicate thickens the slip.

J. A. SUGDEN.

**Unit analysis of feldspar.** E. E. PRESSLER (J. Amer. Ceram. Soc., 1930, **13**, 850—855).—All the major oxides are determined on one sample. The sample (0.5 g.) is sintered with 0.5 g. of sodium carbonate at 850° for 2 hrs., and the amount of alkali added as flux is subtracted from the total amount determined. Full routine schedule is described. The principle of the method is applicable, in general, to aluminous silicates.

J. A. SUGDEN.

**Forming pressure of dry-pressed refractories.** II. **Effect of pressure variations on fired properties.** R. E. BIRCH (J. Amer. Ceram. Soc., 1930, **13**, 831—844; cf. B., 1930, 613).—Increase in forming pressure produces an increase in firing shrinkage (this apparent anomaly is due to the increased vitrification caused by closer contact between the particles), fired density, and crushing strength. Improvement is also found in load-bearing capacity and in resistance to slag attack and abrasion. Resistance to thermal spalling is decreased. The magnitude of these changes is decreased by the addition of flint clay. It is pointed out that the improvements to be obtained by higher forming pressures are, as yet, limited by "pressure cracking." The evacuation of the mould appears to be a possible solution of this problem.

J. A. SUGDEN.

See also A., Dec., 1511, **Reactions in the melting of glass batches** (TAMMANN and OEISEN).

## PATENTS.

**Apparatus for melting glass and the like.** W. A. MORTON, Assr. to AMCO INC. (U.S.P. 1,760,371, 27.5.30. Appl., 19.11.26).—Material is fed to a melting chamber isolated from the atmosphere, passed by a submerged opening into an "ebullition" chamber, and thence is fed in a thin stream over a weir shaft, preferably circular. Gas from a burner playing along the shaft "supermelts" the glass, driving the reactions to completion and eliminating bubbles. At the bottom of the shaft the glass collects in a chamber communicating by a submerged opening with the working chamber, which is heated indirectly by the waste gases from the supermelting zone. Means are provided for fusing the batch in the melting chamber electrically.

M. PARKIN.

**Smelting of quartz glass.** W. GLEICHMANN (B.P. 334,720, 20.9.29).—Pieces of rock crystal, preheated to about 500° by passing through a charging device in countercurrent to a blowpipe flame, drop into an oxy-hydrogen flame in a muffle furnace and are forced by the blast on to a molten lump of quartz on the opposite wall of the furnace. The molten mass is stabbed by a tool from time to time and rolled up into a lump for further treatment.

L. A. COLES.

**Manufacture of porous [silicate] masses.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,318, 19.7.29).—The silicious residue obtained as a

by-product in the treatment of clay with hydrochloric acid for the recovery of alumina is heated with 20% sodium hydroxide solution under pressure until a solid mass is obtained. Further quantities of the silicious residue are incorporated with the mass and the mixture is heated at 300–500°, whereby a porous product is obtained. Alternatively, only part of the swelling may be produced in this operation; the product is then ground and formed into shapes which are again fired to form porous bricks for electrical or thermal insulation purposes.

A. R. POWELL.

**Laminated glass.** F. B. DEHN. From LIBBEY-OWENS GLASS Co. (B.P. 336,732, 30.8.29).—A sheet of non-brittle material is sprayed with a bonding medium comprising a mixture of butyl phthalate with polyglycol derivatives, *e.g.*, diethylene glycol monoethyl (and/or monobutyl) ether. The sheet is then placed between coated glass plates and united thereto by heat and pressure.

L. A. COLES.

**Manufacture of sheets of silica [for use as window panes].** BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of J. H. SULLIVAN (B.P. 336,923, 13.2.30. U.S., 14.2.29).—Silica fused *in vacuo*, *e.g.*, in a furnace similar to that described in B.P. 210,489 (B., 1926, 667), is allowed to flow by gravity under raised gaseous pressure into sheets  $\frac{3}{8}$ – $\frac{3}{4}$  in. thick.

L. A. COLES.

**Preventing the weathering of lamp-bulb glass.** R. C. MATLOCK, JUN., ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,127, 27.5.30. Appl., 17.10.28).—The reduction in strength noticeable when internally-frosted bulbs are stored before making up is greatly prevented by washing after the fortifying etch with a concentrated solution of a substance inert to sodium silicate, but reactive or having strong solvent action towards the silica fluorides formed in the etching process. Suitable substances are inorganic and some organic ammonium salts, in particular a saturated solution of the chloride at 60–110°. The bulbs are afterwards washed and dried as usual.

M. PARKIN.

**Treatment of clay [for production of refractories].** W. G. BUCKLES, ASSR. to PARCO DEVELOPMENT Co. (U.S.P. 1,760,191, 27.5.30. Appl., 27.4.28).—In the production of high refractories from a crystalline mineral base (*e.g.*, quartzite or "baukite") with clay or finely-ground shale (*e.g.*, Dakota shale) as binder, the clay or shale is ground with sufficient water to form a flowable cream which is stored (*e.g.*, for 24 hrs., to permit microscopic penetration of the particles) before pugging with the base.

L. A. COLES.

**Refractory and its manufacture.** H. C. HARRISON, ASSR. to McLANAHAN-WATKINS Co. (U.S.P. 1,760,133, 27.5.30. Appl., 22.12.24).—Mixtures comprising more than 50% of a finely-ground mineral of the cyanite group and a highly aluminous bond (*e.g.*, Georgia bauxitic clay) and substantially free from alkali or alkaline-earth fluxes, are dry-pressed and fired.

L. A. COLES.

**Refractory product.** H. P. HOOD, ASSR. to CORNING GLASS WORKS (U.S.P. 1,760,360, 27.5.30. Appl., 27.4.23. Renewed 18.6.28. Cf. U.S.P. 1,527,874; B., 1925, 318).—A paste composed of finely-ground crystalline aluminium silicate (sillimanite) and a hydrosol or hydrogel

comprising equimolecular proportions of silicic acid and aluminium hydroxide is moulded, dried, and fired.

L. A. COLES.

**Abrasive articles.** CARBORUNDUM Co., LTD., ASSEES. of H. C. MARTIN (B.P. 316,189, 23.7.29. U.S., 24.7.28).—The mixing of binding material comprising synthetic resins with abrasive grains previously wetted with an aldehyde of b.p. above 100° (furfuraldehyde) is effected below 21° in an atmosphere the humidity of which is kept automatically below saturation; the mixtures are subsequently moulded under pressure and heated.

L. A. COLES.

**Manufacture of abrasive refractory or like articles.** CARBORUNDUM Co., LTD., ASSEES. of P. H. WALKER and S. S. KENYON (B.P. 310,811, 15.4.29. U.S., 1.5.28).—Abrasive grains and a binder, particularly those used for truing abrasive wheels, are impregnated with solutions containing inorganic material capable on subsequent treatment of depositing insoluble material in the pores, *e.g.*, with aluminium and calcium chloride solution followed by treatment with ammonia, or with aluminium sulphate solution, and are subsequently heated strongly.

L. A. COLES.

**Preventing the weathering (especially clouding, tarnishing, and lustering) of the surface of glass.** K. KAMITA, ASSR. to ASAHI GLASS Co., LTD. (U.S.P. 1,782,169, 18.11.30. Appl., 9.9.27. Jap., 21.10.26).—See B.P. 294,391; B., 1928, 711.

(A) Moulds for manufacture of articles in silica glass. (B) Working of fused pure silica, or other vitreous material fusible with difficulty. QUARTZ & SILICE (B.P. 338,456-7, 13.2.30. Fr., [A] 16.4.29, [B] 17.4.29).

**Muffle furnace** (B.P. 337,845).—See I. **Refractory materials** (B.P. 336,440).—See IX.

## IX.—BUILDING MATERIALS.

**Relation of Brinell hardness and transverse strength to the compressive strength of building brick.** J. W. MCBURNEY (J. Amer. Ceram. Soc., 1930, 13, 823–830).—Tests on 25 makes of bricks show that although there is somewhat better correlation between Brinell numbers and flat compressive strength than exists between the transverse and flat compressive strengths, yet the relationship is not sufficiently exact for the compressive strength to be calculated from the Brinell number. The deviations are due to lack of homogeneity in the brick.

J. A. SUGDEN.

**Chemical characteristics of cement pipe-lining.** E. L. CHAPPELL (Ind. Eng. Chem., 1930, 22, 1203–1206).—In linings which have been exposed to cold water for 5–60 years or to hot water for 1–2 years the soluble lime salts have usually been largely replaced by ferric hydroxide. This does not disintegrate the lining, but serves to bind it together still more firmly. Tests on steel plates covered by silica sand show that, on exposure to water, the layer of sand in time becomes cemented together by the precipitated ferric hydroxide to form a protective layer. Cement lining is therefore especially protective in cases where iron tends to be precipitated.



A cement lining based on these principles (high in iron and silica, low in lime) has been developed.

J. A. SUGDEN.

**Soundproofing.** R. MOENS (*Natuurwetensch. Tijds.*, 1930, 12, 200—206).—A method and apparatus for comparing the conductivities for sound of different materials are described.

S. I. LEVY.

**Bulking of particles.** ROLLER.—See I. **Moisture-proof wood.** HUNT.—See XIII.

#### PATENTS.

**Manufacture of bituminous paving compositions.** E. C. WALLACE (B.P. 334,588, 6.6.29).—Coarse mineral aggregate (diam.  $>\frac{1}{4}$  in.) is stirred with bituminous cement in quantity slightly in excess of that required to coat the particles; finer aggregate (diam.  $<\frac{1}{8}$  in.) and more bituminous cement are then stirred in, and a quantity of mineral dust (all passing 50-mesh and at least 60% passing 200-mesh) equal in volume to one half of the cement is finally added.

L. A. COLES.

**Manufacture of building blocks.** M. SKOLNIK, Assr. to INSOLO HOLDING CO. (U.S.P. 1,760,446, 27.5.30. Appl., 19.11.28).—Fibrous material, *e.g.*, straw, rendered pliable by treatment with alkali solutions, reduced mechanically to pieces of  $\frac{3}{8}$ —1 in. in length, and subsequently treated with lime, is pressed into shape, dried, and treated with water-glass. The articles may be sprayed with hot asphalt into which crushed marble, granite, etc. is pressed.

L. A. COLES.

**Treatment of asbestos and other composition sheets or material.** R. C. NEVILLE (B.P. 336,731, 29.8.29).—Sheets, mouldings, etc., constructed, *e.g.*, of cement, clay, or asbestos, are successively impregnated with tar, tar oils, or pitch, dried at about 250°, dipped in a solution of formaldehyde and phenol, and heated gradually to and maintained for 3—12 hrs. at about 230°.

L. A. COLES.

**Production of artificial ice rinks for skating and other purposes.** H. REESER (B.P. 336,218, 4.6.29).—Cast blocks comprising a mixture of, *e.g.*, about 70% of sodium thiosulphate, 30% of borax, and a little sodium sulphate and alum, are laid, the moulded surfaces uppermost, on a foundation of laths overlaid with wire netting embedded in the mixture used for casting the blocks, and are cemented with the same mixture. After it has been smoothed, the surface is covered with a preparation of soap and stearine.

L. A. COLES.

**Heat- and sound-insulating materials, and refractory materials.** M. CASES (B.P. 336,410, 18.10.29. It., 24.10.28).—A porous insulating material comprises a mixture of 5—10% of asbestos, 20—40% of silicate wool, and 50—75% of the residue obtained by the combustion of rice chaff bonded with small percentages of clay. The mixture may be formed into bricks containing a proportion of granulated cork and the bricks burned to remove the cork and leave a light porous insulating material.

A. R. POWELL.

**Wood preservation.** H. S. McQUAID, Assr. to H. W. WALKER (U.S.P. 1,758,958, 20.5.30. Appl., 25.7.24. Renewed 8.4.27).—The wood, especially that exposed to

the action of marine borers, is impregnated with petroleum oil containing in solution (preferably 1% of) an aromatic arsenic compound having a solubility of 0.7—60 pts. per 10<sup>5</sup> pts. of sea water at 20°. Such compounds as phenyldichloroarsine, phenylarsenious oxide or its nitro-derivative, diphenylchloroarsine or its amino-derivative, diphenyl- or dichlorodiviny-arsenious oxides, etc. are mentioned.

L. A. COLES.

**Uniting surfaces of wood or paper.** I. G. FARBININD. A.-G. (B.P. 319,001, 28.8.29. Ger., 14.9.28).—Adhesive sheets which soften at raised temperatures or pressures and comprising one or more carbohydrate esters of saturated or unsaturated higher fatty acids or of cyclic substituted fatty acids or of hydroaromatic carboxylic acids, *e.g.*, cellulose naphthenate-oleate, with the addition, if desired, of resins, softening agents, fillers, etc., are interposed between the surfaces and the masses are united by heat and/or pressure.

L. A. COLES.

**Rotary cement-burning kiln.** A. V. JENSEN, Assr. to F. L. SMITH & CO. (U.S.P. 1,781,845, 18.11.30. Appl., 25.4.28. U.K., 28.11.27).—See B.P. 306,613; B., 1929, 325.

**Bricks, blocks, tiles, etc., and walls and roofs made therefrom.** A. F. BERRY (B.P. 338,147 and 338,162, 8.8.29).

**Waterproof plates or sheets [of corrugated cardboard] for building purposes.** E. RUDIN and H. KOLLBRUNNER (B.P. 338,403, 13.12.29. Switz., 13.12.28).

**Porous masses** (B.P. 336,318).—See VIII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Heat- and acid-resisting cast irons with high chromium and carbon contents.** E. VALENTA (*Iron & Steel Inst. Carnegie Schol. Mem.*, 1930, 19, 79—165).—If alloys are to be really proof against scaling, the chromium content should be not less than 15%, the upper limit being determined by economy and by the fact that the heat-resistance remains constant above 35% Cr. The carbides are heat-resistant, but the massive crystals have a destructive effect on the homogeneity and strength of castings. The iron-chromium-carbon system was investigated with graphical representation. A marked increase of hardness after air-cooling corresponds to the formation of those alloys in which the ratio Cr : C is almost exactly 5, and a distinct crest which extends over the whole zone follows a line corresponding to a Cr : C ratio of 10. Self-hardening was entirely suppressed in all homogeneous alloys containing 2.3—2.5% Si; there is, however, no precise practical limit for the minimum silicon content in an alloy, as it is influenced by the position of the alloy in the system. In high-carbon alloys with a comparatively low eutectoid temperature (about 800°) it would be necessary to add about 4% Si in order to raise the critical points and the alloy would become brittle. Aluminium exerts a much stronger influence than silicon in the same direction, as small a quantity as 0.5% being sufficient to suppress entirely the self-hardening of alloys, and the effect of aluminium on grain growth was similar to that of silicon. The carbon

content proved to have little influence on the liability to oxidation. The volumes of chromium alloys are essentially permanent and no changes such as occur in alloys of the Fe-Fe<sub>3</sub>C system due to graphitisation, oxidation of silico-ferrite, etc. were observed.

C. A. KING.

**Malleability of rolled soft steel and a new method for its improvement.** J. VIETORISZ (Iron & Steel Inst. Carnegie Schol. Mem., 1930, 19, 167—213).—Results of a detailed study of the properties of soft steel rolled at different temperatures with different degrees of deformation over the range 1300—20° showed that both the uniform and total elongations have their highest values at 900° and the lowest at 300°. Marked changes occurred at 700—850°, in which range all the mechanical properties have remarkably low values. The yield point, traction hardness, and tensile strength reach a maximum value when steel is rolled at 200—400°, with low values at 700—850°, at about which latter temperatures the values for tenacity are highest, falling rapidly with decrease in temperature to 300°, which indicates that any deformation due to "cold" rolling below 700° results in the exhaustion of the resistance of the material to an external load. Changes in malleability as a function of the temperature were not so distinct. It has been proved that any rolled material, even if rolled at a high temperature, becomes oriented and that such treatment cannot produce homogeneity. All-round "best" material was produced at 850°, but for special qualities the appropriate temperature must be selected with a careful avoidance of the critical temperature range. The structure under the different treatments is discussed.

C. A. KING.

**Effect of nickel and silicon on the properties of whiteheart malleable cast iron (with special reference to "thin-walled malleable").** I. S. J. E. DANGERFIELD, F. JOHNSON, and E. R. TAYLOR (Iron & Steel Inst. Carnegie Schol. Mem., 1930, 19, 1—18).—The presence of nickel improves the fluidity of "white" iron, which remains white when cast even with 3.66% Ni. The tensile strength is increased to a maximum at 2.5% Ni and then falls rapidly to 6.4 tons/in.<sup>2</sup> at 3.66% Ni. Increase in nickel causes a progressive fall in elongation and 0.7% Ni gave the best results from bend tests. Diminution in the size of temper-carbon nodules is attributed to the effect of nickel and also, up to 2.8% Ni, to a retardation in the migration rate of carbon during annealing, which therefore impairs the toughness and ductility of the cast bars. With 2.5% Ni or more, carbon aggregates at the crystal boundaries with the result that when 3.8% Ni is present only 0.085% C is left in the annealed bars, which causes excessive oxidation and "burning."

C. A. KING.

**"Over-reduced" condition in molten steel.** J. V. McCRAE, R. L. DOWDELL, and L. JORDAN (Bur. Stand. J. Res., 1930, 5, 1123—1149; cf. McCrae and Dowdell, B., 1930, 1156).—A study of four charges of acid steel made in the electric furnace, two being normal and two over-reduced, indicates that the difficulty of pouring thin castings of the over-reduced metal is not due to differences of temperature, rate of cooling, tensile properties, or microstructure. The fluidity of the over-reduced steels in the later stages is lower than that of

normal steel; when this stage is reached all the oxygen present is combined as silica, and there remains an excess of free silicon which, by oxidising on the surface of the metal, causes the apparent reduction of fluidity and may lead to an incorrect estimate of the temperature of the steel on account of the high emissivity of the silica film. The surface film may also increase the rate of loss of heat by radiation and possibly retard the flow of the metal during casting, causing the steel to wet the sand of the mould. Deoxidation of the steels with aluminium lowers the percentage elongation and reduction of area; aluminium retained by the metal causes a reduction of ductility.

H. F. GILLBE.

**Mechanism of the recovery of martensite.** P. CHEVENARD and A. PORTEVIN (Compt. rend., 1930, 191, 1059—1062).—The authors' method of isothermal recovery (B., 1930, 1069) has been applied to tempered steels containing various proportions of martensite and austenite. The recovery of martensite in hypereutectoid steels follows the reaction martensite (solid solution of  $\alpha$ -iron and carbon)  $\rightarrow$  (cementite + less carburised martensite). Irregularities in the curves obtained by plotting mechanical properties (*e.g.*, change in hardness and relative extension) against the recovery temperature may be interpreted by superposition of the two recovery reactions of austenite, viz., (a) austenite  $\rightarrow$  (cementite + less carburised austenite), and (b) austenite  $\rightarrow$  (cementite + martensite).

J. GRANT.

**Transformation and constitution of high-chromium steels.** T. MURAKAMI, K. OKA, and S. NISHIGORI (Tech. Rep. Tôhoku, 1930, 9, 405—445).—The general diagram of steels containing 10—30% Cr and less than 1.1% C has been constructed and the microstructure is illustrated. The A<sub>2</sub> point descends with increasing chromium content and ascends as carbon increases, and in the same alloy the lowered transformation increases in magnitude as the maximum heating temperature rises or the cooling rate increases, though in high-chromium steels in which no  $\gamma$ -phase is formed no transformation is observable. The A<sub>2</sub> transformation is lowered with increasing chromium more readily in medium-carbon steels.

C. A. KING.

**Micro-analysis of steel.** J. KASSLER (Mikrochem., Emich Festschr., 1930, 170—174).—Carbon may be determined by combustion of 0.05 g. of the steel in a current of oxygen in presence of lead peroxide at 1200° and absorption of the carbon dioxide evolved by soda-lime; manganese is determined by Heike's modification of P. Smith's method, and nickel either gravimetrically with dimethylglyoxime or volumetrically with potassium cyanide. For the determination of chromium the steel is dissolved in dilute (1:5) sulphuric acid containing sodium phosphate (60 g./litre) to retain tungsten in solution, and oxidised with nitric acid, and, after boiling the solution, the chromium is oxidised by an excess of potassium permanganate. The solution is heated with hydrochloric acid to dissolve precipitated manganese dioxide and destroy the excess of permanganate, and the chromate is finally titrated with ferrous ammonium sulphate. If the steel dissolves with difficulty in sulphuric acid it may be fused with sodium peroxide, the chromate being extracted with water.

H. F. GILLBE.

**Determination of iron in aluminium.** G. AGAMENONE (Giorn. Chim. Ind. Appl., 1930, 12, 486—487).—To 5 g. of the metal are added 75 c.c. of water and, in portions, 50 c.c. of concentrated hydrochloric acid, and, after boiling gently to ensure complete dissolution, the solution is diluted to 250 c.c. and filtered. To 100 c.c. of the filtrate are added a few c.c. of hydrogen peroxide solution, and, after boiling for 15 min., the solution is cooled and neutralised with sodium bicarbonate. The quantity of iron present is determined by titrating the iodine liberated on adding potassium iodide with 0.05*N*-sodium thiosulphate. The procedure is more rapid than are gravimetric methods, and is more convenient than a method involving the titration of small quantities of ferrous iron, especially in presence of titanium.

H. F. GILLBE.

**Analysis of certain metallic alloys.** E. DI NOLA (Annali Chim. Appl., 1930, 20, 457—461).—Rapid methods for the analysis of copper-zinc and copper-zinc-nickel alloys are given. Copper is determined by dissolving the metal in nitric acid and making up to a definite volume, almost neutralising an aliquot part of the solution with sodium carbonate, adding sodium hydrogen sulphite and excess of 0.1*N*-ammonium thiocyanate, making up to known volume, and titrating the excess of thiocyanate with 0.1*N*-silver nitrate. Under the conditions used, alloys with 65 and 72% Cu showed 64.92 and 71.88%. Nickel may be determined either by Belasio and Marchionneschi's method or by the dimethylglyoxime method, after removal of the copper from the nitric acid solution by means of powdered tin; this procedure serves also for the determination of nickel in argentan, nickel-bronze, and nickel or chromium-nickel steels. Zinc in copper-zinc nickel alloys is determined by difference.

T. H. POPE.

**The logarithmic wedge sector and its use in quantitative spectrum analysis [of alloys].** F. TWYMAN and F. SIMEON (Trans. Optical Soc., 1930, 31, 169—183).—A logarithmic wedge sector similar to that used by Scheibe and Neubäusser (B., 1928, 930) is described, and its use in the quantitative analysis of alloys is discussed. It can be used whenever the intensity of a spectral line of one constituent of an alloy relative to that of a second increases with the percentage of the first constituent relative to that of the second, the differences between the lengths of selected lines of the two constituents, obtained under stated conditions, being plotted first for a series of alloys of known composition. Numerous precautions are discussed.

C. W. GIBBY.

**Method of making ball-hardness tests of metals; plotting table for the simplification of "Meyer (hardness) analysis."** H. O'NEILL (Iron & Steel Inst. Carnegie Schol. Mem., 1930, 19, 19—38).—Following a critical survey of the principles of hardness and of the existing appliances for testing hardness, an attempt is made to fulfil most of the demands of theoretical hardness, and a machine is described which uses an "unrecovered" indentation, gives truly comparable hardness numbers, and can be used for "Meyer" analysis. In principle a hemispherical diamond indenter is mounted on a steel shank, and the specimen is pressed

up against the indenter against a recording weight indicator. A steel cap fits over the diamond and standardises the penetration, for when the cap contacts with the specimen an electrical relay reverses the movement. By exposing more or less of the indenter by adjustment of the cap different-sized indentations may be made and interpretations of hardness by the Meyer method become possible. A plotting table designed to facilitate the determination of Meyer constants by the graphical method is described.

C. A. KING.

**Gravimetric electrodeposition of metals and its application to pharmaceutical chemicals. II.** S. G. LIVERSEDE (Quart. J. Pharm., 1930, 3, 482—487; cf. B., 1929, 855).—Bismuth, in "nitrate mother-liquors," in pharmaceutical mixtures (*e.g.*, Liq. Bism. et Ammon. Cit.), and in such compounds as xeroform, may be conveniently determined electrolytically. The known methods for determining cadmium in presence of zinc, tin, lead (particularly traces in tartar emetic), and of nickel and cobalt are discussed with reference to the best conditions for the electrolysis in each case.

R. CHILDS.

**Physics and the chemical industry.** (MELIN.—See I. Cast-iron enamels. GORMLY.—See VIII. Determination of mercury in pharmaceuticals. REIMERS.—See XX.

See also A., Dec., 1503, **X-Ray study of tungsten magnet-steel residues** (WOOD). 1509, **System lead-silver** (YOLDI and JIMENEZ). **Systems silver-strontium and silver-barium** (WEIBKE). **Copper-arsenic alloys** (KATOH). 1510, **Electrolysis of alloys** (KREMANX). 1527, **Passivity and corrosion of iron** (CHITTUM). **Anodic passivation of metals** (SHUTT and STIRUP). 1537, **Purification of mercury** (JONES). 1546, **Detection of impurities in metals** (REDEKER and LEIGHTON). 1547, **Colour reaction for cobalt** (EGRIGWE).

PATENTS.

**Concentration of fluorspar values from fluorspar ores.** ALUMINIUM, LTD., Asses. of J. C. WILLIAMS and O. W. GREENMAN (B.P. 319,685, 19.8.29. U.S., 26.9.28).—After desliming the ground ore, the pulp is frothed with a mixture of oleic and cresylic or phosphocresylic acids.

W. J. WRIGHT.

**Apparatus for the distillation of volatile metals.** E.M.S. INDUSTRIAL PROCESSES, LTD., R. A. STOKES, and E. G. L. ROBERTS (B.P. 337,471, 3.8.29).—The hearth or bottom of the distillation chamber is formed of a number of semi-circular troughs arranged abreast, and the material to be treated is conveyed transversely across the troughs by means of scrapers rotating about horizontal axes.

B. M. VENABLES.

**[Case]-hardening process [for iron and steel].** A. B. KINZEL, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,747,549, 18.2.30. Appl., 27.6.28).—See B.P. 314,423; B., 1930, 1033.

**Magnetic material [alloys] and appliance.** G. W. ELMEN, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,768,443, 24.6.30. Appl., 12.7.26).—See B.P. 274,136; B., 1928, 97.

**Aluminium alloy.** H. C. HALL and T. F. BRADBURY, ASSRS. to ROLLS-ROYCE, LTD. (U.S.P. 1,782,300, 18.11.30. Appl., 23.10.29. U.K., 18.12.28).—See B.P. 323,353; B., 1930, 198.

**Pickling compound [for metals].** P. J. COLE and C. BANTA, ASSRS. to BARRETT Co. (U.S.P. 1,759,840, 27.5.30. Appl., 31.10.26).—See B.P. 334,418; B., 1930, 1034.

**Tuyères for blast furnaces.** E. POHL, A. WAGNER, and GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 338,130, 10.5.29).

**Drying apparatus [for small metallic articles etc.].** J. N. COLLINS (B.P. 337,899, 16.9.29).

**Electric furnaces** (U.S.P. 1,751,856 and 1,751,912—3).—See XI. Non-corrosive alcoholic solutions (U.S.P. 1,752,145). Varnished metals (B.P. 337,192).—See XIII.

## XI.—ELECTROTECHNICS.

**Weatherproof impregnated braidings of electric conductors.** E. KINDSCHER (Gummi-Ztg., 1930, 45, 333—336).—In the use of minium and linseed oil for the impregnation of the braiding of electric conductors, the quality of minium used is of importance as it affects the degree of formation of lead soap, and this has an important bearing on the induced water resistance; not only is the fineness of the minium of importance, but so also is its content of free lead oxide, because this latter is the main factor in the formation of the organic lead salt. Litharge alone, on the other hand, reacts too extensively and results in the formation of too hard a reaction product. It is consequently necessary to select such minium as will result in the optimum degree of formation of organic lead compounds.

D. F. TWISS.

**Physics and the chemical industry.** GMELIN.—See I. Electrodeposition of metals. LIVERSEDGE.—See X. Insulation tests on rubber. CHURCH and DAYNES.—See XIV.

See also A., Dec., 1503, X-Ray study of tungsten magnet-steel residues (WOOD). 1510, Electrolysis of alloys (KREMAN). 1527, Electrochemical behaviour of metals (CHITTUM). Anodic passivation of metals (SHUTT and STIRRUP). 1532, Electrochemical preparation of sodium hyposulphite, and of formates from carbonic acid (RABINOVITSCH and others). Action of high-speed electrons on methane, and on oxygen-carbon monoxide (McLENNAN and GLASS). 1537, Purification of mercury (JONES). 1546, Detection of impurities in metals (REDEKER and LEIGHTON). 1550, Thermionic valve falling-ball viscosimeter (MOORE and CUTHBERTSON). 1563, Electrolytic separation of arginine and alanine (HENDRY and JOHNSON).

## PATENTS.

**Electric induction furnace process. Electric induction furnace. Electric [arc] furnace.** A. E. GREENE (U.S.P. 1,751,856, 1,751,912—3, 25.3.30. Appl., [A] 25.3.21, [B] 30.8.20, [C] 8.2.27. Renewed [A] 12.5.23).—(A) In an electric induction furnace, part of a horizontal channel serving a secondary circuit forms a duct of relatively small cross-sectional area and is

closed except at its ends, where it joins and opens into the other part of the channel so that the current density in the duct is increased sufficiently to cause arcing and to move metal through the duct. Means are provided for rocking the furnace body and for tilting it to pour the charge. (B) Tubular channels connect two furnace hearths, and current is induced in metal contained both in the channels and the hearths. An electrode is arranged in each hearth chamber so that current may be passed through metal in the connecting channels from one chamber to the other. (C) An electrode-holding mechanism is described. J. S. G. THOMAS.

**Electric furnace [resistor support].** C. L. IPSEN, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,751,408, 18.3.30. Appl., 23.10.24).—A resistor is supported directly on ring-like metallic members having very small area of contact with the resistor. J. S. G. THOMAS.

**[Electrical] apparatus for regulating temperature.** PILKINGTON BROTHERS, LTD., and P. M. HOGG (B.P. 337,803, 2. and 26.8.29).—An *E.M.F.* is applied to electrodes placed in liquid heated in a furnace so that current passes substantially through the whole of the liquid, variations of the *E.M.F.* and/or current due to changes in the electrical conductivity of the liquid being caused to control the heating means. Apparatus for effecting this control comprises two solenoids and relays energised, respectively, proportionately to the *E.M.F.* applied to, and the current flowing in, the liquid. J. S. G. THOMAS.

**Electric current rectifier.** S. RUBEN, ASSR. to RUBEN RECTIFIER CORP. (U.S.P. 1,751,361, 18.3.30. Appl., 1.6.26).—A dry-surface rectifying device comprising an electropositive electrode, *e.g.*, of zinc, and an electronegative electrode composed of a metal compounded with an element of group VI, *e.g.*, a compound of copper and tellurium, is claimed. J. S. G. THOMAS.

**Metallic oxide rectifiers.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 336,926, 24.2.30. Ger., 27.2.29).—A conductor connected to a source of current is soft-soldered to the metal back supporting a layer of oxide, *e.g.*, cuprous oxide. Preferably the parts to be soldered together are smeared with acid-free soft solder containing bismuth and sprayed with liquid metal, *e.g.*, zinc. J. S. G. THOMAS.

**Rectifier.** J. SLEPIAN, ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING Co. (U.S.P. 1,749,549, 4.3.30. Appl., 19.1.27).—A metal, *e.g.*, copper, is coated with a compound of the metal, *e.g.*, cuprous oxide, which is further coated with a layer produced by electrolytic reduction of the compound, so that good electrical contact may be made between the oxide layer and a source of current. J. S. G. THOMAS.

**Thermionic cathodes.** E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 336,710, 14.8.29. Addn. to B.P. 320,866).—Filaments coated by being drawn through a sludge consisting of an insulator and an agglutinant are passed through a die which limits the amount of coating, before the coated filament is dried, baked, or sintered. Preferably the coating and moulding is carried out in one operation by an

extrusion process, the sludge being pressed into intimate contact with the filament. J. S. G. THOMAS.

**Manufacture of filaments for electron-discharge tubes.** ETABL. INDUSTRIELS DE E. C. GRAMMONT ET DE A. GRAMMONT (B.P. 337,971, 5.11.29. Fr., 10.12.28).—An alkaline-earth cyanide, deposited upon a refractory filament, *e.g.*, of tungsten, is converted by chemical or physical treatment within the tube, *e.g.*, by treatment with steam, into the oxide. Alternatively, a mixture of carbon and a carbonate of an alkaline-earth metal deposited upon the filament is heated in a stream of nitrogen. J. S. G. THOMAS.

**Gas-filled electric-discharge illuminating tubes.** F. ROTHER (B.P. 315,794, 17.7.29. Ger., 18.7.28).—The cathode in a glow-discharge gas-filled tube is bombarded with  $\alpha$ -particles from a radioactive anode, *e.g.*, of thorium or uranium. J. S. G. THOMAS.

**[Reducing the space charge in] electron-discharge devices.** BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of J. L. ZEHNER and M. A. ACHESON (B.P. 318,526, 4.9.29. U.S., 4.9.28).—A considerable area of the device, other than the cathode, *e.g.*, the anode, is covered with a substance, *e.g.*, an amalgam, which, on heating, produces a vapour which on collision with electrons forms positive ions. J. S. G. THOMAS.

**[Removing deposits from the inner surface of the bulbs of] electric lamps etc.** BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of D. K. WRIGHT (B.P. 337,782, 4.7.29. U.S., 18.2.29).—The internal surface of the bulb is scoured with finely-powdered tungsten contained in the bulb. J. S. G. THOMAS.

**Insulation of electric wires and cables.** BRIT. INSULATED CABLES, LTD., and F. J. BRISLEE (B.P. 337,052, 27.7.29).—A wrapping of esterified cellulose containing a plastifier, enclosed in tape or lead sheath, is heated at about 100°, so that the layers of wrapping unite to form a homogeneous covering. A suitable composition consists of (pts. by wt.): cellulose acetate 100, toluenesulphonethylamide 50, toluenesulphonanilide 30, tolyl phosphate 30. J. S. G. THOMAS.

**Insulating material.** J. E. GROSS, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,750,305, 11.3.30. Appl., 19.11.27).—Sheets of magnetic material forming a core are coated with a film composed of water 60 pts., sodium silicate 45 pts., sugar or other carbohydrate 10 pts., and a filler, *e.g.*, mica, and annealed at a temperature sufficiently high to decompose the carbohydrate so that the separate sheets do not adhere. J. S. G. THOMAS.

**Insulating material.** H. G. WALKER, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,749,008, 4.3.30. Appl., 31.8.27).—A waterproof insulating material composed of about 50–55% of rubber, 18–23% of hard rubber dust, 15–5% of silica gel, 7.75% of zinc oxide, 6.25% of litharge, 2% of sulphur, and 1% of paraffin is claimed. J. S. G. THOMAS.

**Electrically detecting, measuring, and recording changes in physical conditions or properties of materials etc.** J. J. DOWLING and K. S. G. SOMMERVILLE (B.P. 337,785, 30.7.29).—A condenser, the capacity of which is influenced by the material to be

tested, is included in an electrical circuit comprising a current-indicating device subjected to rapid current impulses, the change of reading of the device indicating a change in the property of the material under test.

J. S. G. THOMAS.

**Electric resistance furnace for hardening [of metals] by nitrogenisation.** A. FRY, Assr. to NITRALLOY CORP. (Re-issue 17,874, 18.11.30, of U.S.P. 1,661,694, 6.3.28. Ger., 4.9.25).—See B., 1928, 306.

**X-Ray tubes.** H. SEABROOK. From C. H. F. MÜLLER A.-G. (B.P. 336,726, 27.8.29).

**Generation of intense photoelectric currents.** G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 336,574, 12.7.29).

**Furnaces (B.P. 316,229). Purification of gases (B.P. 337,448). Measuring the velocity etc. of liquids and gases (B.P. 317,397).—See I. Oxidation of hydrocarbons (B.P. 337,407).—See II. Preventing weathering of lamp bulbs (U.S.P. 1,760,427). Porous masses (B.P. 336,318).—See VIII. Treatment of balata etc. (B.P. 335,966). Rubber sheets from latex (U.S.P. 1,750,177). Rubber articles (B.P. 334,581 and 336,659). Porous rubber [diaphragms] (B.P. 313,052).—See XIV.**

## XII.—FATS; OILS; WAXES.

**Variation in colour-test value of commercial samples of cod-liver oil.** F. J. DYER and F. WOKES (Quart. J. Pharm., 1930, 3, 417–426).—Twenty-four retail samples have been examined by the following modified antimony trichloride method (cf. Carr and Price, A., 1926, 870): 0.2 c.c. of a 20% (vol./vol.) solution of the oil in chloroform and 2 c.c. of a saturated solution of antimony trichloride in the same solvent (*d* 1.626–1.627; cf. Wokes and Barr, B., 1927, 569, who give *d* 1.592–1.618) are mixed in a cylinder 1 cm. diam. and the blue colour produced is matched in a Lovibond tintometer, the reading being taken after exactly 30 sec. The "colour-test values" ranged from 3.0 to 9.0 (av. 5.7). These figures are compared with those given by a series of 16 manufacturers' samples found, by feeding experiments, to be of satisfactory vitamin-A potency (4.5–13.0; av. 8.2). Without claiming any definite relation between "colour-test values" and "biological units," it is suggested that the value 4.0 for the former should be regarded as the minimum for a satisfactory oil. It is recommended also that the oil be stored in completely filled containers of amber glass, since storage in white, blue, or green bottles, especially in direct sunlight, causes a rapid loss of activity (cf. Drummond and Morton, A., 1929, 1202).

R. CHILD.

**Para rubber-seed oil.** G. S. JAMIESON and W. F. BAUGHMAN (Oil & Fat Ind., 1930, 7, 419–421, 437).—Kernels from *Hevea brasiliensis* contained moisture 5.04% and oil 42.53%. The clear dark-red expressed oil had:  $d_{20}^{25}$  0.9185,  $n_D^{20}$  1.4737, acid value 40.9 (42.4 after 9 months' keeping), saponif. value 191.8, unsaponifiable matter 0.8%, Reichert–Meissl value 0.3, Polenske value 0.2, iodine value (Hanus) 135.2, thiocyanogen-iodine value 88.8, insol. (acid) hexabromide value 15.7 ( $\equiv$  5.5% of linolenic acid), saturated acids

(lead salt-ether method) 16.0% (corr.), unsaturated acids 78.4% (corr.), iodine value 163.8. The composition of the oil, determined by thiocyanometric analysis and fractionation of the methyl esters of the saturated acids, is given as: acids in the oil—linolenic (total) 19.6%, linoleic 31.5%, oleic 27.3%, palmitic 7.0%, stearic 8.74%, and arachidic acid 0.26%. Large amounts of free fatty acids appear to be formed in the oil while still in the seed; if this could be prevented the oil should be useful in the paint industry.

E. LEWKOWITSCH.

**Fatty acids in rubber compounds.** GARNER.—See XIV. **Fat of asses' milk.** LAXA.—See XIX.

See also A., Dec., 1557, **Preparation of pure palmitic and stearic acids** (DUBOVITZ). **Purification of higher fatty acids** (HOLDE AND BLEYBERG). 1610, **Waxy substance of cocoon silk fibres** (MASAMI). 1623, **Decomposition of fat by tubercle bacilli** (SEDYKH). 1625, **Vitamins and refining of olive oil** (MARGAILLAN). 1627, **Vegetable oils** (McNAIR). 1628, **Digitalis seed** (CULTER). **Aegle marmelos** (DIKSHIT AND DUTT).

#### PATENTS.

**Rendering process [for animal fats].** S. HILLER (U.S.P. 1,760,059, 27.5.30. Appl., 24.11.24).—Plant for a continuous process is described, in which the comminuted fatty material is disintegrated under the action of indirect heat (temperatures between 50° and 100°) and mechanical impact (cf. U.S.P. 1,518,926; B., 1925, 78) and dehydrated until the moisture content is reduced to below 20%; the solid dehydrated residue leaving the inclined rendering cylinder is continuously expressed in order to separate the oil.

E. LEWKOWITSCH.

**Rendering process [for animal fats].** W. LAABS, ASSR. to ALBRIGHT-NEIL Co. (U.S.P. 1,761,480, 3.6.30. Appl., 20.3.26. Cf. U.S.P. 1,578,245; B., 1926, 449).—Animal fats are rendered in a jacketed cooker at temperatures above 100°, the material being disintegrated by steam at pressures of 20—60 lb./in.<sup>2</sup>, developed from the naturally contained moisture. The excess of steam and vapours are removed, while maintaining the pressure, until the material attains a moisture content of 17—35%, which is insufficient to cause emulsification, but enough to prevent absorption of nitrogenous material by the fat. After disintegration is completed the pressure is reduced to atmospheric, and moisture is evaporated until the water content of the residue is reduced sufficiently (below 17%) to allow clear fat to be expressed from it.

E. LEWKOWITSCH.

**[Continuous] extraction of oils and fats by liquid solvents.** C. SCHLOTTERHOSE and H. BRANDT (SCHLOTTERHOSE & Co.) (B.P. 316,881, 1.8.29. Ger., 4.8.28).—The dried material is carried by screw conveyors in a counter-current direction upwards through a battery of inclined extractor chambers; the upper part of each extractor is tapered and fitted so that the bulk of the solvent is pressed out of the material as it passes to the next extractor. Apparatus is provided for the continuous removal of solvent from the meal and extract.

E. LEWKOWITSCH.

**Extraction of oils and fats by liquid solvents.** C. SCHLOTTERHOSE and H. BRANDT (SCHLOTTERHOSE & Co.) (B.P. 337,079, 2.8.29. Ger., 28.3.29. Addn. to B.P. 316,881; preceding).—Instead of, or in addition to, treatment with liquid solvent, as in the process described previously, the material is treated with the vapour of the solvent, supplied con- or counter-current-wise.

E. LEWKOWITSCH.

**Manufacture of soaps.** HENKEL & Co., G.M.B.H. (B.P. 316,935, 17.7.29. Ger., 6.8.28).—Technical fatty acids are continuously mixed with the theoretical amount of alkali carbonate solution and conveyed through a heating system (wherein saponification is completed and the carbon dioxide and excess water are removed) into a mixing device in which the hydrolysis of the neutral fat present is effected by continuously admitting theoretical amounts of caustic alkali lye. Apparatus suitable for a continuous process is described.

E. LEWKOWITSCH.

**Sea-salt soap.** L. C. LAURENT, ASSR. to H. J. LENICHECK (U.S.P. 1,760,759, 27.5.30. Appl., 18.4.27).—A large proportion (over 15%) of sea salt can be incorporated in a bath soap in the presence of a gluten binder (e.g., 10%). Glycerin (e.g., 1%) may be added.

E. LEWKOWITSCH.

**Sulphonation of higher fatty acids and fatty acid derivatives.** N.V. CHEM. FABR. "SERVO," and M. D. ROZENBROEK (B.P. 312,283, 23.5.29. Holl., 23.5.28).—Salts of acetic acid or its lower homologues, or of the sulfo-, chloro-, hydroxy-, sulphochloro-, or sulphohydroxy-substitution products of these acids, are added to sulphuric acid or oleum for use in the sulphonations. Sulphates or bisulphates crystallising out may be separated before using the mixtures, and the spent acid may be neutralised to recover the added salts for re-use. Phosphoric or phosphorous acid, or their anhydrides, chlorides, or oxychlorides, may also be added to the mixtures (cf. B.P. 293,690; B., 1930, 203).

L. A. COLES.

**Apparatus for distilling fatty acids.** H. BOLL-MANN (U.S.P. 1,781,745, 18.11.30. Appl., 16.12.25. Ger., 2.2.25).—See B.P. 246,803; B., 1926, 713.

**Covering materials [linoleum etc.] and their decoration.** ARMSTRONG CORK Co., Assees. of H. W. PRENTIS (B.P. 338,205, 16.8.29. U.S., 9.10.28).

**Viscous oils** (B.P. 337,639).—See II.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Paste paints.** W. DROSTE (Z. angew. Chem., 1930, 43, 1002—1006, 1022—1025).—Methods of determining the principal properties of paste paints are described, including tests of the viscosity (within 5%) and elasticity by measurement of the depth of penetration of a falling cone, and of the adhesive properties by measurement of the length of thread which may be drawn from the paint by slow withdrawal of the cone. Satisfactory paints are smooth and have a viscosity of 80—180 cm.-g./cm.<sup>3</sup>, and should yield threads 5—20 mm. long; elastic properties are of little importance. The oil number, derived from the quantity of oil required to convert a pigment into a paste paint, has little relationship to the viscosity. The behaviour of a paste during mixing is

dependent on both the pigment and the nature of the oil used. The influence on the viscosity and ropiness of adding stand oil, fatty acids, and aluminium stearate to poor pastes is discussed, and the greater improvement produced by adding certain wetting agents is described; the action of such substances is not due merely to depression of the surface tension of the oil, but is concerned also with their behaviour as protective colloids.

H. F. GILLBE.

**Application of microchemical methods to investigation of the pigments in paintings.** H. HETTERICH (Mikrochem., *Enrich Festschr.*, 1930, 152—162).—Particles of pigment may easily be removed from oil paintings by means of a fine needle moistened with glycerin, without causing any visible injury; details are given of micro-analytical methods suitable for the examination of the pigments.

H. F. GILLBE.

**Effectiveness of moisture-excluding coatings on wood.** G. M. HUNT (U.S. Dept. Agric., 1930, Circ. No. 128).—Among numerous coatings examined, those containing aluminium leaf between two coatings of other materials were the most effective.

A. G. POLLARD.

**Permeability to air and water of films of "fillers" [for automobile finishing].** W. TOELDT (Farben-Ztg., 1930, 36, 318—321, 364—366, 409—410. Cf. B., 1930, 570).—An apparatus by which the transmission of air at pressures up to 12 atm. through "filler" films may be measured is described and illustrated. Air-permeability is defined as the number of c.c. of air passing in 1 sec. through 1 cm.<sup>2</sup> of a film 1 cm. thick, with a pressure difference of 1 atm.; permeability values for films of 24 fillers, before and after irradiation with ultra-violet light for 24 hrs., are quoted together with their compositions. The results are discussed in detail with reference to dependence of permeability on the nature and proportions of pigment and vehicle. The permeability to water, measured by Husse's method, in which penetration by water is shown by its dissolving methylene-blue with which the underside of the film has been coated, was also determined on the same series of fillers, and the results are compared with the air-permeabilities. In general, parallelism is found as anticipated, but a filler containing red lead gave anomalous results.

S. S. WOOLF.

**Accelerated testing of cellulose lacquers.** O. MERZ (Farben-Ztg., 1930, 36, 316—318, 362—364).—A range of lacquers containing increasing proportions of ester gum was tested by an accelerated weathering cycle, by normal exposure, and by a bending test after artificial ageing. The results are tabulated and discussed, and the difficulty of forecasting durability from such accelerated tests is indicated. It is considered that photochemical methods offer possibilities in this direction, the work of Stutz and others being summarised. Light-absorption values of various films before and after drying are given.

S. S. WOOLF.

**Bulking of particles.** ROLLER.—See I. **Plant resins and petroleum.** RAKUSIN.—See II. **Weather-proof braidings.** KINDSCHER.—See XI. **Butyl alcohol and acetone.** GABRIEL and CRAWFORD.—See XVIII.

See also A., Dec., 1578, **Resin of *Garcinia Mangostana*** (DRAGENDORFF).

## PATENTS.

**Manufacture of [weather-resistant] paints.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,198, 2.5.29).—Basic pigments, with the exception of precipitated zinc oxide which has been dried at temperatures not above 500° (cf. B.P. 335,626; B., 1930, 1164), are incorporated with coating materials in quantity sufficient to combine into soaps all free or combined fatty acids present in the binding agent.

S. S. WOOLF.

**Preparation of weather-resisting lithopone.** "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND. (B.P. 335,839, 15.11.29. Ger., 5.12.28).—Freshly prepared and calcined lithopone, nearly always containing excess of soluble barium salts, e.g., uncombined barium sulphide, is quenched in water containing added soluble sulphates, e.g., sodium or magnesium sulphate. If desired, 1—5% of soluble barium compounds may be added to the lithopone before calcination. Increased durability of the pigments is claimed.

S. S. WOOLF.

**Printing inks.** I. G. FARBENIND. A.-G. (B.P. 337,199, 31.10.29. Addn. to B.P. 307,877; B., 1930, 26).—Inks or pastes comprising finely-divided colouring materials, unhardened soluble condensation products of polybasic carboxylic acids (e.g., phthalic or succinic acid), polyhydric alcohols (e.g., glycerol or glycols), and one or more monobasic carboxylic acids containing at least 7 carbon atoms, e.g., fatty acids of linseed, tung, or castor oils, and, if desired, cellulose derivatives, oils, resins, plasticisers, and organic solvents are claimed.

S. S. WOOLF.

**Manufacture of nitrocellulose solutions or compositions.** W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,615, 11.4.29).—Water-wet nitrocellulose (containing 5—60% of water) is dissolved in a solvent which evaporates in air at normal temperatures and has b.p. at atmospheric pressure of not less than 120°, e.g., butyl acetate, with the addition of a totally or partly water-miscible solvent or diluent, e.g., industrial alcohol, in amounts sufficient to hold the water in solution. A higher-boiling solvent, e.g., methyl cyclohexanone, and water-wet pigments may also be incorporated.

S. S. WOOLF.

**Coating compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,344, 3.4.29. U.S., 26.5.28).—Drying oils of iodine value above 100 (preferably blown, iodine value above 140) are added to one or more polymerised inorganic or organic vinyl esters, e.g., acetone-soluble vinyl chloride polymeride or vinyl acetate polymeride; plasticisers, solvents, and pigments are incorporated. [Stat. ref.]

S. S. WOOLF.

**Lacquer [of flat transparent finish].** D. J. TIGHT, Assr. to R. N. NASON & Co. (U.S.P. 1,756,528, 29.4.30. Appl., 3.6.24).—Transparent or semi-transparent pigment material, e.g., magnesium carbonate or china clay, is incorporated in low-viscosity nitrocellulose lacquers.

S. S. WOOLF.

**Preparation of [non-penetrative] varnish or lacquer.** H. F. WAKEFIELD, Assr. to BAKELITE CORP.



(U.S.P. 1,756,267, 29.4.30. Appl., 24.2.27. Renewed 19.5.28).—A reactive resin of the phenol-aldehyde type is partly transformed into the insoluble resinoid modification by heat, a solvent for the reactive component is admixed, and the mixture is colloiddally dispersed.

S. S. WOOLF.

**Application of varnishes, lacquers, or like coatings to metal surfaces.** "HEROLD" A.-G. (B.P. 337,192, 29.10.29. Ger., 30.4.29).—Phenol-formaldehyde resin varnishes are applied to metal surfaces which have been preheated to a temperature above the b.p. of the solvent present, and the film is then hardened by further heating, bubbling being thus avoided. The resin should preferably remain soft in the initial stages of drying to ensure that any bubbles formed will rise to the surface; to this end, small amounts of suitable retarders, *e.g.*, acetic acid, are added or the resin is diluted with cresol, glycerin, etc. or solid inert fillers, *e.g.*, felspar or kieselguhr.

S. S. WOOLF.

**Non-corrosive alcoholic solutions.** W. S. CALCOTT and H. W. WALKER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,752,145, 25.3.30. Appl., 22.10.28).—A solution which is non-corrosive to iron is prepared by adding a small amount of a higher fatty acid which is substantially insoluble in water to an aqueous solution of a dihydric or trihydric alcohol.

C. RANKEN.

**Manufacture of resinous compounds.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. HILL (B.P. 336,698, 3.8.29).—Non-hydroxylated fatty oils, especially drying oils, *e.g.*, linseed or tung oil, are introduced into synthetic resins of the "glyptal" type, by using the product of interaction of a polyhydric alcohol (glycerol) and a lower aliphatic aldehyde (paraformaldehyde) as part or the whole of the polyhydric alcohol component of the "glyptal."

S. S. WOOLF.

**Manufacture of neutral polymerisation products from vinyl esters.** I. G. FARBERIND. A.-G. (B.P. 336,237, 27.6.29. Ger., 16.11.28).—Before, during, or after polymerisation there is added to the vinyl ester a small amount of a basic substance soluble in organic solvents, *e.g.*, 0.5% of diphenylguanidine, 1.2% of cyclohexylamine, 0.8% of hexamethylenetetramine, 0.4% of dimethylaniline and 2.0% of a soluble urea-formaldehyde product, 0.5% of phenylhydrazine and 0.4% of benzoyl peroxide. The treated resin is more stable and less liable to discoloration.

C. HOLLINS.

**Synthetic resinous materials and processes for hardening and moulding them.** TOLEDO SCALE MANUFACT. Co. (B.P. 312,343, 3.4.29. U.S., 26.5.28).—In the manufacture of resins of the urea-formaldehyde type, a fusible or partly fusible intermediate reaction product or condensate, before or after drying, is treated with an agent the acidity of which is increased by heating, *e.g.*,  $\beta$ -bromohydrocinnamic acid, isodibromosuccinic anhydride. The mixture is dried and ground to produce a moulding powder which when subjected to heat develops sufficient acidity to overcome its residual alkalinity and is converted into glassy final products.

S. S. WOOLF.

**Moulding mixture. Making ornamented moulded articles.** H. WADE. From BAKELITE CORP. (B.P. 336,264—5, 10.7.29).—(A) A mixture of a potentially re-

active resin binder, fillers (*e.g.*, wood flour) and colouring materials is extruded as a sheet in which the coloured particles are distributed so as to simulate grain and cloud effects etc. (B) Depressed lines or areas of a suitable die are filled with a composition comprising a pigment and a reactive phenol resin; the composition is transferred to a phenol resin composition in partly cured, substantially non-flowing, and slightly plastic state, and the whole is further "cured," whereby integral union is secured without blurring.

S. S. WOOLF.

**Mixing of paints (B.P. 337,945).**—See I. **Dispersing agents (B.P. 319,249).**—See III. **Chromium oxide (B.P. 336,671).**—See VII. **Laminated glass (B.P. 336,732).** **Abrasive articles (B.P. 316,189).**—See VIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Some fundamental rubber problems.** G. S. WHITBY (Trans. Inst. Rubber Ind., 1929, 5, 184—195; 1930, 6, 40—62).—In a discussion of the chemical constitution of rubber it is assumed that the polymerisation of isoprene to caoutchouc is similar in mechanism to the polymerisation of indene, and several methods of formulation of caoutchouc with an open-chain structure are indicated; the additional ethylenic linking in the total of  $(n + 1)$  per  $n$  units of isoprene would be experimentally distinguishable only with difficulty if  $n$  were large. When unvulcanised rubber is kept in an organic solvent for years, the whole, excepting the protein, ultimately dissolves; the swelling behaviour of vulcanised rubber also fails to give evidence of the presence of two sharply defined fractions. The theory that rubber consists of two portions, a sol (or liquid) phase and a gel (or solid) phase, is consequently inadequate. Certain artificial polymerides such as polystyrene and polyvinyl acetate show marked analogies with rubber in physical characteristics. The structure of the globule in rubber latex therefore is not essential to the elastic behaviour of rubber. The elastic artificial products show a high degree of heterogeneity. Rubber likewise must consist, not of two modifications, but of a mixture of an unbroken series representing a wide range of polymerisation, and so-called "diffusion rubber" or "sol" rubber is far from homogeneous; this, indeed, is evident from the fact that "sol" rubber when stretched exhibits X-ray diffraction phenomena. The view that the elasticity of rubber is associated with a helical molecular structure also is discountenanced by the wide range of artificial products possessing elastic extensibility, and particularly by the fact that the elastic character may become apparent only on warming or swelling. It is believed that for the production of artificial products with good elastic properties an optimal degree of polymerisation is necessary; vulcanisation of rubber improves the qualities of rubber by exercising a further polymerisation effect and accelerators act as catalysts in this polymerisation. The behaviour of vegetable oils towards heat and towards sulphur is adduced in support of this view.

D. F. TWISS.

**Applications and analysis of [rubber] latex.** W. H. STEVENS (Trans. Inst. Rubber Ind., 1930, 5, 362—374).—A review, covering the nature of *Hevea* latex and its application to manufactures by such processes as

dipping, spreading, electrophoresis, spraying, extruding, etc. For determination of the dry rubber content of latex it is recommended to weigh 10–15 c.c. into a dish and coagulate by adding acetic acid of 0.5–1% concentration with stirring. After heating for  $\frac{1}{2}$  hr. on a water-bath, the coherent coagulum is removed, washed, and sheeted thinly on washing rollers, dried at 70°, and weighed. D. F. TWISS.

**Effect of keeping coagulum in serum on the plasticity of smoked sheet [rubber].** ANON. (Trop. Agric., 1929, 73, 7–9).—Acetic acid gave variable, and acetic acid with *p*-nitrophenol uniform, results in mastication tests after the coagulum had remained in the serum for 4.5–55.5 hrs. Hardening tests after 6 months' storage at 5° or 15° with ordinary humidity showed little change, whilst samples kept over water or calcium chloride hardened considerably, those prepared from coagulum kept for the shortest period in the serum hardening least. A mixture with 10% S, vulcanised at 148°, showed a progressive decrease in the time of vulcanisation with the length of time during which the coagulum remained in the serum. *p*-Nitrophenol had no effect on the tensile strength. CHEMICAL ABSTRACTS.

**Smoke production for the rubber industry.** D. F. TWISS (Trans. Inst. Rubber Ind., 1930, 5, 407–416).—A discussion of the reinforcement of rubber from the point of view of the production of reinforcing agents of small particle size, and their improved dispersion, or possible formation, in rubber. D. F. TWISS.

**Developments in the physical testing of rubber.** A. A. SOMERVILLE (Trans. Inst. Rubber Ind., 1930, 6, 130–169).—The ordinary tensile, elongation, and friction tests give no definite indication of the probable service life of a rubber article. Stress-strain curves at low elongations are more enlightening, and a large moulded ring test piece (mean circumference 20 in., cross-section  $0.25 \times 0.25$  in.) has several advantages. Test pieces cut along the mill-grain of "pure" and compounded stock are slightly stiffer than those cut lengthwise with the grain. Compounded stock shows a much reduced resistance to stretching at higher temperatures, whereas "pure" vulcanised stock gives substantially the same behaviour at 0° and 100°. The effect of temperature, compounding, anti-oxidants, and artificial ageing on resistance to fatigue, flex-cracking, and cutting is also investigated. Deterioration in air at 70°, for a wide range of compositions, is 30% greater when the samples are stretched 100%, but stretch does not affect the results in bomb ageing. In ozone and in sunlight a stretch of 10% has a maximum influence in intensifying ageing. D. F. TWISS.

**Double refraction and structure in rubber.** B. W. ROWLAND (Ind. Eng. Chem., 1930, 22, 1182–1184).—"Diffusion rubber" and benzene-insoluble rubber resemble ordinary rubber in exhibiting anisotropy and double refraction when distorted, as also does synthetic rubber produced from isoprene. As a thin ribbon of rubber which has been soaked in chlorobenzene (which has approximately the same refractive index as rubber) or other organic solvents still exhibits double refraction when stretched, the micelles in distorted rubber must themselves be anisotropic with a

positive rodlet type of double refraction. On stretching, the micelles become oriented with their long axes in the direction of stretch. The double refraction observed with rubber under compression (of which colour photographs are reproduced) is mainly the result of stretch in a direction oblique to the line of compression; compression between lubricated surfaces results in much feebler anisotropy. It is believed that stretching causes an intimate distribution of the soluble "diffusion rubber" within the gel phase, tending towards optical homogeneity of the mass and loss of distinction between the two phases. D. F. TWISS.

**Röntgenographic investigation of gutta-percha and balata.** H. HOPFF and G. VON SUSICH (Kautschuk, 1930, 6, 234–237).—Contrary to the results of Clark (A., 1927, 816), the hydrocarbon constituent of balatā is röntgenographically identical with that of gutta-percha, but that of india-rubber is quite distinct. The gutta-percha hydrocarbon exists in two modifications, one of which is stable up to 60° and is crystalline in the unstretched condition; the other, stable above 60°, resembles rubber in that it is amorphous and becomes crystalline only when stretched. Although conversion of the normally stable  $\alpha$ -form into the  $\beta$ -variety occurs rapidly and spontaneously on heating above the transformation temperature, the inverse change is often delayed, but may be induced by separation from a solvent below the transformation point. The temperature of transformation of the two forms is considerably influenced by impurities. D. F. TWISS.

**Instrument for testing hardness and permanent set of rubber and for measuring thickness of test pieces.** H. A. DAYNES, E. B. JOHNSON, and J. R. SCOTT (Trans. Inst. Rubber Ind., 1930, 6, 63–81).—A simple, inexpensive instrument is described by which the hardness of rubber is determined in one operation by a measurement of the indentation caused in the specimen by the rounded end of the gauge stem, the upper end of which carries a loaded platform, relative to the unloaded surface of the rubber. By means of an attachment the instrument can be adapted to the measurement of the thickness of test pieces, e.g., tensile-test rings, the mean of three thicknesses being read at one operation. D. F. TWISS.

**High-speed tensile experiments with vulcanised rubber.** A. VAN ROSSEM and H. J. BEVERDAM (Kautschuk, 1930, 6, 224–229).—Tensile tests were made on vulcanised rubber samples of various compositions by breaking a ring test piece, supported at two points, by a blow from a pendulum; the stronger the ring the greater was the reduction of the amplitude of the pendulum. This form of apparatus is far from ideal, but the results show that this type of test is of great promise for technical purposes and calls for development. Maximum tensile strength, measured in this way, for a mixture of rubber 100, sulphur  $7\frac{1}{2}$ , diphenylguanidine 1, is attained at a vulcanisation coefficient of 2.4, whereas with the ordinary slow test the tensile optimum is observed at a coefficient of 4.3. In other words, with a high-speed tensile test the degree of vulcanisation giving the maximum strength is identical with the "technical optimum." D. F. TWISS.

**[Rubber] curing [vulcanising] conditions.** E. F. POWELL (Trans. Inst. Rubber Ind., 1930, 6, 184—201; cf. Perks, *ibid.*, 1928, 4, 123).—An experimental consideration of causes of and methods for obviating variations in the temperature of articles during vulcanisation. The effect of the presence of air in an open-steam tyre-vulcanising pan, the advantage of additional internal heating by an "air bag" containing steam and air, and the rate of penetration of heat into the rubber mixture are illustrated by actual records.

D. F. TWISS.

**Vulcanisation experiments with aniline sulphate.** A. ZEITLIN and L. H. WILLIAMS (Trans. Inst. Rubber Ind., 1930, 5, 173—178).—Aniline sulphate is without accelerative influence in a rubber-sulphur mixture; in a mixture containing also litharge it actually retards vulcanisation. Even in the presence of a large proportion of litharge the decomposition of aniline sulphate into its constituent base and acid (or lead sulphate) is not complete.

D. F. TWISS.

**Accelerating influence of a basic azo dye in vulcanisation.** T. J. DRAKELEY and A. G. COULSON (Trans. Inst. Rubber Ind., 1930, 5, 179—183; cf. Drakeley, Zeitlin, and Williams, B., 1924, 683).—In otherwise unaccelerated rubber mixtures, Bismarck-brown has a distinct accelerating influence, but in the presence of diphenylguanidine its effect is masked considerably.

D. F. TWISS.

**Volume increase in vulcanisation of rubber.** F. KIRCHHOFF (Chem.-Ztg., 1930, 54, 906—908).—The statements of Ditmar and Preusse (B., 1930, 781) are based on misconception and errors of observation. The sp. gr. of a mixture of rubber and sulphur or selenium is greater after vulcanisation. The overflow from a mould during the vulcanisation process is the effect of mechanical pressure and of thermal expansion.

D. F. TWISS.

**Comparison of the effect of pigment reinforcement of crude and reclaimed rubber.** J. STOPFORTH (Trans. Inst. Rubber Ind., 1930, 5, 356—361).—The results of Winkelmann and Croakman (B., 1928, 238) are analysed on the lines adopted by Lunn (B., 1929, 445). It is found that the increases in resilient energy effected by the incorporation of various fillers and reinforcing agents are notably less with reclaimed than with new rubber.

D. F. TWISS.

**Dispersion of gas black and the physical properties of rubber mixtures.** E. A. GRENQUIST (Kautschuk, 1930, 6, 229—233).—By microscopical examination of raw and vulcanised samples, using vertical illumination, it is possible to estimate the degree of dispersion of gas black in highly compounded rubber mixtures. The degree of dispersion is improved by progressive mastication only as long as the plasticity of the mixture does not fall below a certain value. For any gas black mixing there is an optimal degree of working which will lead to maximal physical properties on vulcanisation.

D. F. TWISS.

**Ageing of rubber compounded with china clay.** T. J. DRAKELEY and P. G. PACKMAN (Trans. Inst. Rubber Ind., 1930, 5, 426—441; cf. Drakeley and Pollett, B.,

1929, 444).—Ageing (at 70°) of vulcanised mixtures of rubber (100 pts.), zinc oxide (5), sulphur (4), diphenylguanidine (1), and various proportions of three clays of different degrees of fineness resulted at first in a displacement of the stress-strain curve towards the stress axis with subsequent movement in the reverse direction. Increase in the proportion of clay up to 35 pts. lessened the rate of decay even in the presence of 1 pt. of an antioxidant, but above 35 pts. the rate of ageing was approximately constant. In all cases ageing resulted in an increase of the "slope" of the stress-strain curve.

D. F. TWISS.

**Alternating behaviour of fatty acids in rubber compounds.** T. L. GARNER (Trans. Inst. Rubber Ind., 1930, 5, 403—406).—Vulcanisation tests with mixtures of rubber (100 pts.), sulphur (10), zinc oxide (5), and fatty acid ( $\frac{1}{100}$  mol.), using fatty acids containing from 1 to 18 carbon atoms, showed an alternating effect on the tensile strength of the product, particularly if the rubber had previously been extracted with acetone. No corresponding alternating effect was observed on the viscosity of solutions of the unvulcanised rubber.

D. F. TWISS.

**Effect of adding sodium bisulphite to latex on the plasticity of the crêpe.** G. MARTIN and L. E. ELLIOTT (Trop. Agriculturist, 1930, 75, 213—215).—Addition of sodium bisulphite to latex (up to 1:133) renders the crêpe harder and more difficult to masticate in proportion to the amount added.

A. G. POLLARD.

**Oxidation of rubber mixings.** W. C. DAVEY (Trans. Inst. Rubber Ind., 1930, 5, 386—402; cf. B., 1929, 611).—When kept at 100° vulcanised rubber oxidises rapidly, but the ultimate increase in weight is not greatly different from that at 70°; there is, however, less difference observable between the behaviour of the various samples at 100°, and the effect of prior extraction with acetone is less marked. In every case the proportion of acetone-extractable matter attains a maximum and then decreases owing to further conversion of the soluble matter into an insoluble form. The presence of organic accelerators, especially of the aldehyde-amine class, improves the behaviour of the rubber, as also do antioxidants, their influence being exerted essentially on the oxidation of the chloroform-extractable matter to acetone-soluble matter. "White substitute" also reduces the tendency to oxidation, but coarse fillers are particularly undesirable.

D. F. TWISS.

**Oxidation of rubber at atmospheric temperatures.** W. C. DAVEY (Trans. Inst. Rubber Ind., 1930, 6, 202—213).—Samples of finely-divided vulcanised rubber became oxidised slowly in the dark, but previous extraction with acetone expedited the rate of oxidation, particularly of unaccelerated mixtures. The final increases in weight were greater than for similar samples at 70°, and the proportion of acetone extract was higher. A moist atmosphere retarded the oxidation, but the degree of this effect varied with the nature of the sample. The proportion of acetone-soluble matter, on account of the further conversion of this into an insoluble product, gradually attained a maximum, but the tendency in this direction was small at the ordinary temperature. Exposure to sunlight led to a lower proportion of

acetone-extractable matter than did storage indoors; the tendency of the acetone-soluble matter to become insoluble again was less than at 70°. D. F. TWISS.

**Antioxidants [for rubber].** W. J. S. NAUNTON (Trans. Inst. Rubber Ind., 1930, 5, 317—335).—Antioxidants may be classified as (a) natural, (b) physical, such as waxes and dyes, and (c) chemical, such as amines, phenols, and aldehyde-amine condensation products. Apart from ordinary chemical examination, they should be tested as to their dispersability in rubber and as to their preservative action, the latter being best tried by the oxygen bomb method. Chemical antioxidants are chiefly of use for protection against oxidation induced by heat, whether in service or during vulcanisation in hot air; they are of relatively little use for protection against light, physical antioxidants being the more effective in this direction. Chemical antioxidants, however, are largely ineffective in cold-vulcanised rubber. Possible disadvantages associated with the use of antioxidants are staining of light-coloured goods, "blooming" (especially in air-vulcanised articles), interference with the drying of superposed varnishes, and disturbance of the rate of vulcanisation. The theory of Moureu and Dufraisse accords with many of the phenomena observed in the application of antioxidants to rubber, but, in view of the fact that their important effect is to delay changes in the colloidal state of the rubber, the term "anticollotrope" is preferred. D. F. TWISS.

**[Manufacture of] reclaimed rubber.** R. F. PEARSON (Trans. Inst. Rubber Ind., 1930, 5, 336—340).—Of the three methods based, respectively, on the use of oil, acid, and alkali, the last is to-day used the most extensively. Disintegrated tyre scrap, together with caustic soda solution and possibly softeners, is heated for 14—24 hrs. at a steam pressure of 100—200 lb./in.<sup>2</sup>, whereby the cotton is hydrolysed, the rubber plasticised, and the free sulphur substantially removed. The mass is then washed, dried, strained by forcing through a fine wire screen, ground between powerful rolls rotating at different speeds, and finally sheeted. D. F. TWISS.

**Testing of reclaimed rubber.** W. E. STAFFORD (Trans. Inst. Rubber Ind., 1930, 5, 340—348).—Most commercial "whole-tyre" reclaims would give values falling within the following limits:  $d$  1.16—1.26, moisture below 1%, alkalinity below 0.15%, acetone extract 7—10%, alcoholic potash extract below 2%, chloroform extract (48 hrs.) 20—28%, tensile strength (vulcanised) 600—1200 lb./in.<sup>2</sup>, ultimate elongation 300—500%, ash 18—25%. The determination and significance of these and other features, such as content of free carbon and of rubber, plasticity, and ageing, are discussed. D. F. TWISS.

**"Reclaims" from the rubber manufacturer's point of view.** E. H. HURLESTON (Trans. Inst. Rubber Ind., 1930, 5, 348—355).—Reclaimed rubber made by the alkali process is generally superior. The use of reclaimed rubber aids the softening of the mixed stock and permits the use of less solvent in the production of doughs for spreading. Consistency in composition, sp. gr., and plasticity is important, as also is freedom from grit and metal fragments. Other features of

importance are also considered. The customary procedure of mixing reclaimed rubber merely with 5% of sulphur for a vulcanisation test is not satisfactory; rate of vulcanisation and tensile qualities of the product should be tested after admixture with an equal proportion of new rubber and the necessary vulcanising agents. D. F. TWISS.

**Making electrical contact with ebonite and soft rubber for insulation tests.** H. F. CHURCH and H. A. DAYNES (Trans. Inst. Rubber Ind., 1930, 6, 82—95).—The dried coating left after evaporation of a colloidal aqueous suspension of graphite (diluted "Aquadag") provides a satisfactory means of electrical contact between insulating material such as ebonite and other surfaces, e.g., of a wire. The contact resistance is less than that between ebonite and mercury. The aqueous medium is not objectionable in determinations of volume- and surface-resistivities of soft or hard rubber if measurements are deferred until a few hours after application of the graphite suspension. D. F. TWISS.

**Carbon black.** PARKINSON.—See II. **Rubber-seed oil.** JAMIESON and BAUGHMAN.—See XII. **Butyl alcohol.** GABRIEL and CRAWFORD.—See XVIII.

#### PATENTS.

**Concentration of latices.** K.D.P., LTD. (B.P. 337,269, 19.12.29. Ger., 24.1.29).—Aqueous dispersions of rubber or the like are heated, together with mucilages or similar substances, e.g., diatom, adapted to combine with water without previous hydrolysis, to temperatures substantially above 60°. The concentrated latex which forms as a cream has a low proportion of serum constituents. D. F. TWISS.

**[Spray-]drying apparatus [for rubber latex].** E. HOPKINSON, Assr. to GEN. RUBBER CO. (U.S.P. 1,750,753, 18.3.30. Appl., 10.4.26).—A circular chamber is provided near the top with a spraying apparatus centrally disposed and with adjustable means for supplying a distributed downwards current of drying medium; means are provided to prevent direct access of the drying medium to the side wall and sprayer, and to remove suspended rubber from the medium as it issues at the base by widely distributed vents. D. F. TWISS.

**Immediate production of mechanically unworked pure rubber sheets from latex.** P. KLEIN, Assr. to AMER. ANODE, INC. (U.S.P. 1,750,177, 11.3.30. Appl., 26.1.24. Hung., 11.10.23. Renewed 6.10.27).—The rubber from latex which has been freed from impurities and, if desired, from a great proportion of its serum, by a centrifuging process, is electrophoretically deposited as a coherent sheet on an anodic surface, e.g., of a drum or endless band, from which it is stripped after leaving the bath. If the latex is treated with an organic dye the hydrosol particles of which are negatively charged, the dye is adsorbed by the rubber globules without coagulation, and the unadsorbed portion of the dye remains in the serum or is collected in the sediment separated during centrifugation. (Cf. B.P. 223,189; B., 1925, 46.) D. F. TWISS.

**Treatment of balata, gutta-percha, and the like [for insulation purposes].** W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 335,966, 6.6.29).—Raw

gutta-percha or balata is treated with a suitable liquid and at a co-ordinated temperature, *e.g.*, with ethyl alcohol at 21° or with acetone at 3°, so that only the undesirable fluavil resins are removed; the remaining albane resins do not seriously affect the "leakance" of the product, which may be as low as 10 micromicromhos/cm.<sup>5</sup> D. F. TWISS.

**Recovery of quebrachitol from rubber latex serum.** J. MCGAVACK and G. B. BINMORE, ASSRS. to NAUGATUCK CHEM. CO. (U.S.P. 1,758,616, 13.5.30. Appl., 2.12.25).—The powder obtained by the spray-drying of latex serum is extracted with a hot solvent, *e.g.*, alcohol, miscible with water; the solution, after treatment with animal charcoal, is concentrated and allowed to crystallise. The serum from 100 lb. of rubber contains about 1½ lb. of quebrachitol. D. F. TWISS.

**Dispersion of reclaimed rubber.** R. P. ROSE and H. E. CUDE, ASSRS. to NAUGATUCK CHEM. CO. (U.S.P. 1,749,607—8, 4.3.30. Appl., 20.8.27).—(A) Reclaimed rubber softened by absorption of an aliphatic oil, preferably a mineral oil such as spindle oil, is dispersed in water containing a suitable protective agent such as rosin soap, by a kneading operation. (B) Reclaimed rubber is worked with an emulsion of spindle oil in a small proportion of water containing a hydrophilic protective colloid, such as alkaline sulphonated castor oil, the pasty product being subsequently diluted as desired. In both cases the product is well adapted to mixing with fibre pulp for paper manufacture. D. F. TWISS.

**Treatment of aqueous dispersions of rubber and products obtained thereby.** M. C. TEAGUE, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,750,767, 18.3.30. Appl., 28.10.25).—An acidic ligneous product such as pine tar is neutralised with a base, *e.g.*, soda or ammonia, and is then mixed with a neutral resin such as "cumar" or even with spindle oil; the mixture is then emulsified by any suitable method and blended with an aqueous dispersion of rubber. The final composition is stable and applicable as a shoe cement. D. F. TWISS.

**Manufacture of rubber derivative.** W. N. JONES and H. A. WINKELMANN, ASSRS. to B. F. GOODRICH CO. (U.S.P. 1,751,817, 25.3.30. Appl., 28.4.25).—Rubber is converted into a material of greater heat-plasticity and lower chemical unsaturation by heating with the halide (fluoride excepted) of an amphoteric metal, such as iron, antimony, or zinc; *e.g.*, heating with 10–20% of the halide for 15 hrs. at 160° is effective. D. F. TWISS.

**Working up of rubber or of plastic or elastic polymerisation products of diolefines or mixtures of both.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,095, 12.8.29).—In the manufacture of vulcanisation products from rubber or the polymerisation product of diolefines it is advantageous to incorporate up to 30% of substances obtained by eliminating the volatile constituents of the tars yielded by low-temperature distillation of bituminous materials such as brown coal (*e.g.*, by the processes of B.P. 301,975 or 309,253; B., 1929, 119, 457). Prior to incorporation the substances may be subjected to heat or treatment with chemical agents such as sulphur or phthalic anhydride. The substances

may be used in conjunction with ordinary compounding ingredients. D. F. TWISS.

**Rubber composition and its production.** H. A. WINKELMANN, ASSR. to B. F. GOODRICH CO. (U.S.P. 1,751,848, 25.3.30. Appl., 17.11.24).—Hard-blown products of mixed petroleum residues or other asphaltic bitumens, preferably of softening point near 150° and a penetration between 8 and 16 at 50° (A.S.T.M.), are advantageous ingredients for rubber. Such material may be prepared by "blowing" a mixture of Illinois petroleum residue (60 pts.) with California petroleum residue (40 pts.). A suitable proportion to be added to rubber before vulcanisation is 5–25%. D. F. TWISS.

**Composition containing "chlor-rubber."** C. ELLIS, ASSR. to CHADELOID CHEM. CO. (U.S.P. 1,750,583, 11.3.30. Appl., 27.10.22. Renewed 11.12.29).—A solution of chlorinated rubber, preferably containing 60–75% Cl, mixed with softeners such as ethyl phthalate, phenyl phosphate, or camphor, in a solvent such as benzene or carbon tetrachloride is submitted to partial distillation. This removes the tendency to bubble formation during drying and results in homogeneous, pliable, strong films. If desired, the colour of the films can be improved by treating the solution with a bleaching agent such as fuller's earth. D. F. TWISS.

**Porous rubber for filtering and absorbing purposes.** H. BECKMANN (B.P. 313,052, 4.6.29. Ger., 5.6.28).—Finely-porous rubber (*cf.* B.P. 240,430; B., 1925, 1000), reduced to granules or powder, is used, under compression if desired, as a filtering medium or as an electrolytic diaphragm. Its permeability is influenced by its porosity and by the interstitial spaces, the latter being dependent on the degree of compression. D. F. TWISS.

**Colouring of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. J. S. NAUNTON, A. STEWART, and A. SHEPHERDSON (B.P. 334,688, 15.8.29. Addn. to B.P. 304,376; B., 1929, 257).—In the coating of fillers with vat dyes, *e.g.*, by oxidising a solution of the leuco-compound of the dye in the presence of the finely-divided filler, treatment of the filler, after or (preferably) before application of the dye, with a water-soluble salt of a fatty acid or soap leads to brighter shades in mixtures of the coated fillers with rubber. D. F. TWISS.

**Colouring natural rubber or similar materials.** I. G. FARBERIND. A.-G., and J. Y. JOHNSON (B.P. 336,446, 29.10.29. Addn. to B.P. 241,214; B., 1927, 948).—A water-soluble component of the practically water-insoluble colouring material is incorporated in rubber by the ordinary mixing process, the colouring material being subsequently formed within the product. Examples are the introduction of (a) a vat dye in the form of a paste of leuco-compound, the colour being developed by exposing the rubber sheets to air; (b) an azo dye, *e.g.*, by the successive incorporation of an aqueous dispersion of 2 : 3-hydroxynaphthoic *o*-anisidide and diazotised 5-nitro-*o*-toluidine; (c) a lake, by incorporating an aqueous solution of an acid or basic dye followed by the necessary inorganic constituent. The products are uniformly coloured and give almost transparent solutions. D. F. TWISS.

**Reproduction of images on rubber surfaces.** J. P. BROCKWAY (U.S.P. 1,761,424, 3.6.30. Appl., 28.2.29).—The rubber surface, particularly of vulcanite, is first provided with a highly polished surface, which is then washed with hydrochloric acid and wiped dry. On exposing the treated surface, under a photographic negative, to light and "developing" in a mixture of nitric acid and water (1 : 1) the light-affected portions become etched, a positive image being thereby produced.

D. F. TWISS.

**Manufacture of articles of rubber and similar substances from aqueous dispersions containing such substances.** DUNLOP RUBBER Co., LTD., R. G. JAMES, and D. F. TWISS (B.P. 334,581, 6.6.29. Addn. to B.P. 301,100; B., 1929, 105).—By the addition of formaldehyde to ammonia-preserved latex it is possible to reduce the  $p_H$  value to about 4.5 without substantial aggregation of the globules, which still retain their negative charges. If an acid, *e.g.*, acetic acid, is added to latex which has been thus treated, or even to formaldehyde-treated latex having  $p_H$  7–8 provided an additional protective colloid such as casein is present, the particles assume a positive electric charge. With latices which have been deprived of part of their natural protective colloids, *e.g.*, by centrifuging, the addition of a small proportion of a colloid, *e.g.*, blood-albumin, may be desirable. On application of an electric current the rubber globules in latex so treated migrate to the cathode.

D. F. TWISS.

**Manufacture of articles of organic materials, particularly rubber and similar substances, from aqueous dispersions thereof.** DUNLOP RUBBER Co., LTD., D. F. TWISS, A. A. ROUND, and E. W. MADGE (B.P. 336,659, 18.7.29).—Electrophoretic deposition of organic materials such as rubber, compounded or otherwise, from aqueous dispersions is effected by applying an alternating potential and using dissimilar electrodes one of which, *e.g.*, of aluminium or tantalum, exerts a rectifying action on the current. Efficiency of deposition is enhanced by the addition of the phosphate or borate of an alkali metal to the liquid in contact with the rectifying electrode.

D. F. TWISS.

**Manufacture of artificial rubber and rubber substitutes.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 334,554, 5.6.29. Addn. to B.P. 315,916; B., 1929, 829).—Mixtures of artificial rubber-like masses, *e.g.*, those produced by the polymerisation of mixed diolefine hydrocarbons or by the application of various consecutive polymerisation processes to one and the same hydrocarbon, are compounded with finely-divided carbon and vulcanised in the ordinary manner.

D. F. TWISS.

**Manufacture of artificial rubber-like masses capable of being dispersed in water.** I. G. FARBERIND. A.-G. (B.P. 313,440, 6.6.29. Ger., 11.6.28).—Latex-like emulsions or suspensions of artificial rubber-like materials, *e.g.*, synthetic rubber, are concentrated by known methods in the presence of protective colloids, such as sodium oleate, which do not flocculate from solution when heated. The pastes or solid masses so obtained can be reconverted into fluid dispersions by dilution with water.

D. F. TWISS.

**Curing [vulcanisation] of rubber articles by the circulation of heated gases.** H. R. MINOR, Assr. to LIQUID CARBONIC CORP. (U.S.P. 1,757,053, 6.5.30. Appl., 28.8.26).—A preheated inert gas, such as carbon dioxide, is led continuously to the article to be vulcanised, a pressure difference to accelerate the circulatory flow being created by tapping off a small proportion of the gas from the return conduit. Suitable apparatus for the vulcanisation of motor tyres is described. D. F. TWISS.

**Vulcanisable plastic composition.** N. GOODWIN, Assr. to DELANO LAND CO. (U.S.P. 1,758,151, 13.5.30. Appl., 10.3.24. Renewed 7.11.28).—A finely-divided form of carbon ("Goodwin carbon"; cf. U.S.P. 1,758,152; B., 1930, 1100) with minimum oil-absorption properties and marked reinforcing power is used as an ingredient for vulcanisable rubber mixtures.

D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,756,310, 29.4.30. Appl., 28.11.27).—Vulcanisation is accelerated by the aldehyde derivative of the reaction product of a mercaptan and an aliphatic, preferably a secondary, amine. The mercaptan may be mercaptobenzthiazole or an analogue; examples of the amine are piperidine, *iso*amylamine, diethylamine, and of the aldehyde are aldol, crotonaldehyde.

D. F. TWISS.

**Vulcanisation of caoutchouc.** L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,757,944, 6.5.30. Appl., 17.9.25).—Vulcanisation is effected in the presence of the reaction product of a polyhydroxyaryl compound, *e.g.*, quinol, and an amine, *e.g.*, xylydine, amylamine, dibenzylamine; the products obtained by further oxidation, *e.g.*, bisamylaminoquinone, may also be used. These substances act as vulcanisation accelerators and as antioxidants.

D. F. TWISS.

**Vulcanisation of artificial rubber-like masses.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 335,970, 2.7.29).—If the vulcanisation accelerator for incorporation in synthetic rubber, particularly if this also contains carbon black, is previously liquefied by means of a solvent or liquefying agent, the tensile strength and ultimate elongation of the vulcanised product are very favourably influenced. Aldehyde-ammonia, for example, may be used in aqueous solution, mercaptobenzthiazole in picoline, *s*-ethylcyclohexylthiuram disulphide in toluene, etc.

D. F. TWISS.

**Securing more uniform procedure in the heat-treatment of rubber articles and the like.** INDUSTRIAL PROCESS CORP. (B.P. 334,695, 26.8.29. U.S., 26.11.28).—In the moulding vulcanisation of rubber articles such as tyres with the aid of "air bags," the latter are brought to a definite desired temperature before each operation, and a mixture of two gaseous fluids, such as carbon dioxide and steam, is used inside the air bags as the heating medium. The carbon dioxide, which is conveniently introduced first, supplies part of the desired pressure, whilst the steam gives the desired temperature and the balance of the pressure.

D. F. TWISS.

**Treatment of [rubber] latex.** E. HAZELL, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,757,632, 6.5.30. Appl., 5.10.28).—See B.P. 333,391; B., 1930, 959.

**Manufacture of rubber.** U. PESTALOZZA, Assr. to Soc. ITAL. PIRELLI (U.S.P. 1,750,540, 11.3.30. Appl., 9.3.29. It., 13.3.28).—See B.P. 330,520; B., 1930, 829.

**Removal of plastic material from mills** (B.P. 337,435).—See I. **Waterproof fabrics** (B.P. 338,065).—See V. **Insulating material** (U.S.P. 1,749,008).—See XI.

## XV.—LEATHER; GLUE.

**Change in shrinkage temperature [of leather] as a result of retannage, detannage, basicity raising, and complex salt formation.** G. GRASSER and M. ICHISE (J. Fac. Agric. Hokkaido, 1930, 23, 157—164).—Leathers tanned with aluminium, iron, chromium, cerium, sulphur, tannin, oil, formaldehyde, benzoquinone, 1- or 2-dinaphthylmethane, or commercial syntans were treated with various substances, and the change in the temperature at which the leather begins to shrink was determined. Retanning agents raised this temperature; the other substances had specific effects.

### CHEMICAL ABSTRACTS.

**Adsorption of sulphuric acid by leather.** J. BEKK, JUN. (Bur. Stand. J. Res., 1930, 5, 1109—1121, and Ind. Eng. Chem., 1930, 22, 1373—1378).—The adsorption of sulphuric acid from 0—1.44*M*-solutions by a vegetable-tanned leather has been determined. The quantity of acid adsorbed increases rapidly with increase of concentration up to about 0.2*M* and thereafter diminishes, at first slowly, and then more rapidly. The mechanism of the adsorption apparently involves two processes, viz., a chemical reaction between the acid and the proteins of the leather and a pure adsorption process. By application of the Gibbs adsorption equation, and taking into account the chemical reaction, an equation has been derived which reproduces the total adsorption with considerable accuracy; the equivalent weight of the particular leather used is 4300. Leather may be regarded as a solid which presents a definite surface to the liquid, whereas collagen in equilibrium with an acid solution behaves as a gel which is completely permeable to the solution. H. F. GILLBE.

**Testing of gelatin for jelly strength.** E. J. GUILD (J.S.C.I., 1930, 49, 453—454 T).—A method differing from that described by Lawrie (B., 1930, 1092), in that the bulb is made to travel down through the jelly instead of merely breaking its surface, is given in detail together with precautions to ensure accuracy, which is claimed to within 5—7½%. The formula  $(S-4)/B^2$  is submitted for "specific strength," where *S* is jelly strength in g. and *B* is the weight of bone-dry gelatin in g. This formula can be applied to gelatin itself or to its special solutions, such as papermakers' "sizes."

**Lactic acid.** GARRETT.—See XVIII.

See also A., Dec., 1500, **Fluorescence of gelatin in ultra-violet light** (McNALLY and VANSELOW).

### PATENTS.

**Preparation of tanning substances and their application to the tanning of animal skins and hides.** W. SAILER (B.P. 336,984, 18.4.29).—Sulphite-cellulose waste liquors are mixed with hydrogen peroxide

or other substance able to evolve oxygen, a natural tanning material, phenol and/or cresol, and a ferment of the desmolase group, e.g., peroxydase, and the mixture is set aside for several days; the product may, if desired, be treated with a halogen and then used for tanning.

D. WOODROFFE.

**Manufacture of artificial leather.** Soc. INVENZIONI BREVETTI (B.P. 315,413, 8.7.29. It., 14.7.28).—Leather scrap (e.g., chrome-tanned shavings), after disintegration in the presence of a large quantity of water, is mixed with a vegetable tannin extract, oils, greases, and subsequently rubber latex. The suspension is rapidly felted to produce a sheet.

D. WOODROFFE.

**Production of iridescent rainbow colours on artificial [casein] masses.** COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assees. of W. PLINATUS (B.P. 307,746, 4.3.29. Ger., 12.3.28).—Stresses are set up in cold or lukewarm casein masses, e.g., by means of extrusion presses or rollers, and the material is then heated under pressure and allowed to cool under pressure to prevent retraction of the stresses. Masses of different ages or degrees of hardness may be used or mica, formaldehyde, pigments, bronzes, etc. added. L. A. COLES.

**Rubber dispersions** (U.S.P. 1,750,767).—See XIV. **Skin dressing etc.** (B.P. 313,874).—See XVIII.

## XVI.—AGRICULTURE.

**Soil colloids. III. Flocculation of soil colloids IV. Methods of determining soil colloids. V. Methods of determining saturation capacity and degree of saturation of soils.** A. N. PURI (Mem. Dept. Agric. India, 1930, 11, 101—118, 119—131, 133—141; cf. B., 1931, 36).—III. A logarithmic relationship exists between the flocculating values of alkaline-earth hydroxides and the concentration of clay suspensions. The flocculation by electrolytes of soil clays is markedly influenced by the nature of the exchangeable cations present in the clay. The minimum flocculating value of the calcium ion increases with the proportion of exchangeable sodium in the clay. The apparent flocculating value of acids decreases with the time of contact with the clay as a result of the slow dissolution of aluminium.

IV. Results of determinations of colloidal matter in soils by the adsorption of ammonia or of dyes, or by the heat of wetting, are affected by the proportion of adsorbed bases in the soil. In the hygroscopicity method it is preferable to bring the soil into equilibrium with an atmosphere of 70% R.H., and that the "dry" standard of comparison should be the moisture content in equilibrium with an atmosphere of 10% R.H. Within these limits of humidity the disturbing effects of adsorbed bases is minimised.

V. Well-known methods for determining the base saturation of soils are critically examined. The value (*T*—*S*) is determined by treatment of the soil with calcium sulphate followed by baryta, the excess of alkali being titrated with acid to phenolphthalein. Periods of several hours are allowed for the completion of each stage of the reaction, the whole operation occupying 2—3 days. The value *T* is obtained by



treatment with 0.05*N*-hydrochloric acid, washing out residual acid, adding excess of baryta, and titrating the excess with sulphuric acid as before. A. G. POLLARD.

**Relation between  $p_H$  value and state of unsaturation of soils.** A. N. PURI (Soil Res., 1930, 2, 181—184).—Using the author's method (preceding abstract) the *S*, *T*, and *V* values of a number of soils are determined. *V* values above 100% are recorded for soils containing much sodium bicarbonate. This is due to precipitation of a portion of the baryta as barium carbonate, thus indicating falsely high adsorption values. Such soils appear alkaline after preliminary shaking with calcium sulphate and should be titrated with sulphuric acid without the addition of baryta. General but not mathematical relationships between *V* and  $p_H$  values for soils are noted. The value *T* per g. of clay in soils varies with soil type, and may afford a suitable basis for soil classification. A. G. POLLARD.

**"Single value" soil properties: significance of certain soil constants. V. Changes produced in soil by oven-drying.** J. R. H. COURTS (J. Agric. Sci., 1930, 20, 541—548; cf. B., 1930, 875).—The loss of weight-temperature curves of heated soils, over the range 50—250°, are smooth and indicate no sudden change in structure. The customary determination of the "air-dry" moisture of soils by heating at 100° is not representative of any fundamental soil property. Soils recover a portion of the moisture lost in heating when exposed to an atmosphere of 50% R.H. The non-recoverable loss increases with the temperature of heating, and is attributed to the moisture adsorptively or chemically bound to the mineral colloids, and to the breakdown of organic colloids. Recoverable losses are ascribed to free or interstitial moisture. A. G. POLLARD.

**Nitrogen transformation in cultivated soil.** F. HANSEN (Tidsskr. for Planteavl., 1929, 35, 713; Bied. Zentr., 1930, 59, 485—486).—Ammonium salts applied to soil at the end of April or early in May are completely converted into nitrate by the end of June. After ploughing-in liquid manure or farmyard manure, the ammonia contents are converted into protein-like substances which by mid-June are being rapidly mineralised at a rate which exceeds the assimilation of ammonia by the micro-organisms. Nitrification of the ammonia derived from well-rotted manure is more rapid than that of ammoniacal fertilisers. The effects of various nitrogenous fertilisers on soil reaction are discussed. A. G. POLLARD.

**Nitrate fluctuations in a South Australian soil.** J. A. PRESCOTT and G. R. PIPER (J. Agric. Sci., 1930, 20, 517—531).—Seasonal variations in the nitrate content of soils in fallow and various cropping conditions are recorded. Laboratory tests show nitrification to be most active in moist soils of 60—70% saturation and to be favoured by temperature ranges of 11—34° with no definite optimum. A. G. POLLARD.

**Decomposition of lignified materials by soil micro-organisms.** M. PHILLIPS, H. D. WEIHE, and N. R. SMITH (Soil Sci., 1930, 30, 383—390).—The course of the decomposition by soil organisms of the various constituents of maize stems, oat hulls, wheat straw, etc.

is examined. Pentosans and cellulose are rapidly decomposed. Lignin is also decomposed at a rate which under favourable conditions approaches that of cellulose. A. G. POLLARD.

**Detecting differences in soil organic matter.** E. C. SHOREY (U.S. Dept. Agric., Tech. Bull., 1930, No. 211).—The colour density of alkaline soil extracts cannot be used as a measure of the amount of organic matter present. Soils suspended in alkali and then aerated absorb oxygen with the production of sodium carbonate. The extent of this action varies with soil type. The dark colour of alkaline soil extracts is not the result of oxygen absorption by phenolic substances during extraction. Nitrogen exists in all portions of fractionated alkaline extracts. The presence of chitin and compounds of the indole group in soil organic matter is indicated. Type differences in soil organic matter may be demonstrated by the acid hydrolysis of the nitrogenous matter (Van Slyke), by extraction with alcohol and water, and by the effect of hydrogen peroxide on the nitrogen compounds. The application to soil of numerous tests indicative of the presence of groups of organic substances are recorded. A. G. POLLARD.

**Method and procedure of soil analysis.** W. O. ROBINSON (U.S. Dept. Agric. Circ. No. 139, Oct., 1930, 19 pp.).—Analytical details for the complete mineral analysis of soils by the fusion method, as used in the Division of Soil Chemistry and Physics, are given, together with the determination of organic matter and combined water. A. G. POLLARD.

**Mechanical analysis of soils for field purposes.** P. KAMERNAN (Soil Res., 1930, 2, 152—164).—The soil sample (50 g. of light soil or 25 g. of heavy soil) after being heated for 1 hr. with 100 c.c. of 0.005*N*-sodium oxalate, is transferred to a mortar and pestled thoroughly with a rubber pestle. A little water is added and the mixture allowed to settle for 2½ min., when the supernatant liquid is decanted into a dish and allowed to settle for 2½ min. again, and decanted into a 500-c.c. cylinder. The sediment is washed back into the mortar, and the process repeated until 500 c.c. of suspension are obtained; sufficient 0.5*N*-sodium oxalate is then added to bring the whole to 0.005*N*. With saline soils the soil-sodium oxalate mixture after heating is filtered and washed prior to preparing the suspension. Pretreatments with 0.2*N*-hydrochloric acid and with hydrogen peroxide are adopted for soils rich in chalk and organic matter, respectively. The suspension is sampled by siphoning to a measured depth after a time interval sufficient to exclude all particles except clay and very fine silt. The proportion of solids is determined by a hydrometer calibrated against standard clay suspensions. Temperature effects are minimised by comparison with 0.005*N*-sodium oxalate examined simultaneously. The combined sand fractions are determined by sedimentation (7.5 cm. for 75 sec.), the quantity being calculated from the volume of sediment (10 g. of sands under water  $\equiv$  7 c.c. after 5 min.). A. G. POLLARD.

**Gollan's apparatus for mechanical analysis [of soils].** J. H. GOLLAN and L. SMOLIK (Bull. Czechoslov.

Acad. Agric., 1929, 856; Proc. Internat. Soc. Soil Sci., 1930, 5, 163).—Gollan's apparatus yields results in close agreement with that of Kopecký, and is simpler and more practicable.

A. G. POLLARD.

**Replaceable bases and water sorption of soils.** L. SMOLÍK (Bull. Czechoslov. Acad. Agric., 1930, 10; Proc. Internat. Soc. Soil Sci., 1930, 5, 159).—The moisture-absorbing capacity of soils saturated with different bases varied with the base used in the order  $\text{Na} > \text{Ca} > \text{Mg} > \text{K} > \text{NH}_4$ .

A. G. POLLARD.

**Determination of the replaceable bases and the base-exchange capacity of soils.** H. D. CHAPMAN and W. P. KELLEY (Soil Sci., 1930, 30, 391–406).—Existing methods for determining exchangeable bases in soils are frequently inexact owing to dissolution or decomposition of soil constituents by the various solutions used. The methods of Hissink (sodium chloride), Magistad and Burgess (alcoholic barium chloride), and the alcoholic potassium chloride process give reasonably accurate results for the exchangeable calcium in calcareous soils, provided other soluble or decomposable calcium compounds are absent. The exchange capacity of soils may be determined by digestion and leaching with *N*-ammonium acetate, which effects an approximately complete displacement of hydrogen ions. In this process washing with methyl alcohol (which must be neutral) aids the removal of occluded electrolyte, and the adsorbed ammonia is satisfactorily determined by aerating the soil with sodium carbonate solution.

A. G. POLLARD.

**Rapid colorimetric method for determining the citric-soluble phosphates in soils.** A. NĚMEC [with J. LANÍK and A. KOPPOVÁ] (Verlautbar. Czechoslov. Verb. landw. Versuchsanst., 1930, 5, [1]; Bied. Zentr., 1930, 59, 482–485).—A portion (5 c.c.) of the citric acid extract of soil (10 g. of soil + 100 c.c. of 1% citric acid solution) is heated to boiling with 10 c.c. of 40% sulphuric acid and the citric acid oxidised with 0.1*N*-permanganate. Excess of the latter is removed by hydrogen peroxide. After further heating for 30 min., a few drops of a saturated solution of 2:4-dinitrophenol in 50% alcohol are added, followed by ammonia until the appearance of a yellow colour ( $p_{\text{H}}$  4.0 approx.). The phosphate is then determined colorimetrically by means of ammonium molybdate and stannous chloride. By the use of a smaller soil sample than is customary determinations may be made in soils rich in calcium carbonate.

A. G. POLLARD.

**Colorimetric determination of phosphoric acid in hydrochloric acid and citric acid extracts of soils.** R. G. WARREN and A. J. PUGH (J. Agric. Sci., 1930, 20, 532–540).—Current methods for determining phosphates in soil extracts, by the use of the molybdenum-blue reaction, give unsatisfactory results with clay soils owing to the proportion of ferric salts in the extracts. Details are given for the removal, from acid extracts, of iron by means of potassium ferrocyanide and of organic matter by sodium permanganate. Following adjustment of the reaction of the solution, the Denigès or Fiske-Subbarow methods may be adopted; these give results in close agreement with gravimetric methods.

A. G. POLLARD.

**Determination of carbonates in soil.** H. A. LUNT (Science, 1930, 72, 295; cf. Schollenberger, B., 1930, 1123).—Dilute acetic acid (1:1 or 1:2) is suitable for the determination of carbonates in soil, no carbon dioxide being liberated from acid soils containing no carbonate.

L. S. THEOBALD.

**[Fertiliser] "effect value" of potash in the form of kainite, 40% potash salts, and potassium sulphate.** K. H. RATHSACK (Landw. Jahrb., 1930, 72, 543–571).—With the fertilisers examined, the "effect value" of potash in the absence of sodium salts is only slightly below that in their presence and is the same for both grain and roots. On the basis of total yield and straw yield the effect value of potash in kainite and 40% potash salts is higher than in potassium sulphate. This is ascribed to the presence of sodium and magnesium salts in the first two fertilisers named, but for practical purposes the difference may be ignored. The effect value of potash for barley is different from that for oats and is greater for straw than for grain and roots.

A. G. POLLARD.

**Availability of phosphatic fertilisers as shown by an examination of the soil solution and of plant growth.** A. W. GREENHILL (J. Agric. Sci., 1930, 20, 559–572).—The effects of phosphatic fertilisers and of lime on the phosphate concentration of the soil solution and on the growth of barley were examined. Liming considerably increased the concentration of phosphate in the soil solution. Fertilisers increased the phosphate concentration on lightly limed soils, but had variable effects on those heavily limed, superphosphate producing relatively lower concentrations than basic slag. In untreated soils the phosphate concentration was practically constant, but was increased by cropping on lightly limed soils. Liming increased the tillering and yield of barley, and phosphatic fertilisers increased the yield (especially of grain) without affecting tillering. Slag and superphosphate were equally effective on lightly limed soils, but superphosphate was the more effective on those heavily limed. No relationship was observed between the phosphate concentration in the soil solution and plant growth. Higher yields of barley were obtained on fertilised, lightly limed soils in the season of application, but on heavily limed soils the best yields were obtained in the subsequent year.

A. G. POLLARD.

**Laws of solubility of phosphates and potash in mineral soils.** R. GANSSEN (Mitt. Lab. Preusz. Geol. Landesanst., No. 9, 1929; Proc. Internat. Soc. Soil Sci., 1930, 5, 156).—The influence of the alumina content of soils on the solubility of potassium and phosphates is indirect. A high alumina content in soil usually corresponds with a high proportion of base-exchanging silicates which tend to "fix" potassium, and also with relatively large amounts of alumina gel with which phosphate combines. Humus increases the solubility of phosphates in soil by decreasing the proportion of active alumina.

A. G. POLLARD.

**Influence of manuring on the intake of phosphate and lime by various plants.** D. MEYER and P. OBST (Z. Pflanz. Düng., 1930, 9B, 488–512).—The lime and phosphate contents are recorded of numerous crops grown on soils treated with di- and tri-calcium

phosphates, in comparative series receiving typical nitrogenous fertilisers. A. G. POLLARD.

**State of unsaturation of the soil in relation to its field behaviour and lime requirement.** P. E. TURNER (Soil Sci., 1930, 30, 349—381).—Of the exchangeable ions of humid soils only  $\text{Ca}^{++}$  and  $\text{H}^+$  are present in quantities sufficient to influence soil behaviour. In the determination of the degree of unsaturation of soils by the Page and Williams modified method (B., 1925, 108), leaching to 2 litres does not effect complete displacement, but yields sufficiently reliable values for comparative work since more prolonged extraction yields proportional figures. In the case of saturation capacity and saturation deficit (exchangeable  $\text{H}^+$ ) the ratio of values obtained by 2- and 5-litre extractions decreases with increasing saturation capacity of the soil. Soils classified as exhibiting good, intermediate, or bad physical properties fall within three well-defined grades of unsaturation. The limiting unsaturation values of the three grades are independent of the nature of the soil colloids and their degree of colloidalilty, the history of the soil, and variations in exchangeable bases other than  $\text{Ca}^{++}$  and  $\text{H}^+$ . There is a general inverse relationship between the  $p_{\text{H}}$  value and the degree of unsaturation of soils, but other disturbing factors exist (notably the presence of organic matter). Page and Williams' method of determining the degree of unsaturation gives lower values than Hissink's titrimetric method, which probably involves other than purely exchange reactions. Soils classified as physically good are probably in stable equilibrium with calcium carbonate. Relationships between the degree of unsaturation, saturation deficit, and lime requirement are examined. The extent of liming recommended is that sufficient to repress the ionisation of the calcium salts in the acidoid material and to maintain a degree of unsaturation of approx. 20%. A. G. POLLARD.

**Repressive effect of lime and magnesia on soil and subsoil potash.** W. H. MACINTYRE, W. M. SHAW, and J. B. YOUNG (J. Agric. Sci., 1930, 20, 499—510).—Applications of lime and magnesia in amounts customary in practice decreased the solubility of soil potash when thoroughly incorporated, the effect being more pronounced in the subsoil than in the top soil. Both native potassic material and soluble potash fertilisers were similarly affected. The extent of the reduction in solubility increased with the proportion of bicarbonates of calcium and magnesium present in the soil. Surface applications of chalk or dolomite produced a slight increase in potash solubility in the lower (unlimed) soil layers. A. G. POLLARD.

**Decomposition of urea in soils.** T. GIBSON (J. Agric. Sci., 1930, 20, 549—558).—In numerous soil types examined the decomposition of urea was general and in most cases rapid. Greater activity in this respect is associated with soils of higher organic content, heath, pasture, and forest soils being more active than adjacent cultivated areas. The most rapid decomposition of urea occurred in soils of  $p_{\text{H}}$  5.4—6.1 in pastures and of  $p_{\text{H}}$  6.0—6.5 in cultivated land. Alkaline soils were much less active. In general, the production of ammonia

from urea is sufficiently rapid to prevent loss from the soil by leaching. A. G. POLLARD.

**Spontaneous culture method for studying the non-symbiotic nitrogen-fixing bacteria of soils.** R. H. WALKER, J. L. SULLIVAN, and G. G. POHLMAN (J. Amer. Soc. Agron., 1930, 22, 642—648).—Plates of moist soil samples, pretreated in the field with manure and lime, showed a marked growth of *Azotobacter*; with either fertiliser alone the growth was not so vigorous. Addition of lime or of lime and phosphate in the plate increased the growth of *Azotobacter*.

#### CHEMICAL ABSTRACTS.

**Influence of stall manure on the condition of soil.** S. GOY [with P. MÜLLER] (Z. Pflanz. Düng., 1930, 9B, 481—487; cf. B., 1930, 1123).—The lime status of soils is not markedly affected by stall manure. Neubauer tests of manured soil indicate variable proportions of root-soluble potash and phosphate, presumably as a result of different rates of decomposition of the manure. The nitrogen content of the manure was insufficient to maintain normal soil requirements.

A. G. POLLARD.

**Intensive system of grassland management. I. Chemical composition of intensively treated pasture.** A. W. GREENHILL (J. Agric. Sci., 1930, 20, 573—586).—The nutritive ratio of frequently cut pasturage was wider in a dry than in a wet season, but the fibre content was little altered. A temporary reduction in the protein content and an increase in fibre occurred in the grazing season during drought and in the early summer flush period. Intensive manuring of pasture increased the protein content of the herbage.

A. G. POLLARD.

**Investigation of soils and of the mineral content of pasture grasses at Waranama Ranch, Berbice River.** R. R. FOLLETT-SMITH (Agric. J. Brit. Guiana, 1930, 3, 142—159).—Both soils and pastures examined were deficient in lime, phosphate, and potash, and grazing cattle became quickly emaciated. The feeding of bone meal to cattle is recommended.

A. G. POLLARD.

**Nitrogen nutrition of meadows.** A. VON CARON (Landw. Versuchs-Stat., 1930, 111, 163—168).—Examination of the nitrogen balance in meadow land indicates that *Gramineae*, through the agency of soil micro-organisms, can assimilate atmospheric nitrogen.

A. G. POLLARD.

**Relation of nitrates in soils to the response of crops to potash fertilisation. I. Factors contributing to the unproductiveness of alkali soils in Illinois.** O. H. SEARS (Soil Sci., 1930, 30, 325—347).—Failure of the soils examined is due to low availability of potash resulting from alkalinity and to an excessive accumulation of nitrate. Improved crop production following straw mulching is attributed to the added potash in the straw and the reduced nitrate content.

A. G. POLLARD.

**Effect of associated soil treatments on the availability of applied phosphates to field crops on different soil types.** G. ROBERTS (J. Amer. Soc. Agron., 1930, 22, 489—497).—No relation was observed between the effects of rock phosphate or superphosphate

and the total or soluble phosphorus content, lime requirement, soluble calcium, or  $pH$  of the soil. Only when lime used alone produces a significant effect as compared with superphosphate used alone does rock phosphate appear to function well in the presence of lime.

## CHEMICAL ABSTRACTS.

**Phosphorus content of the soil solution and its relation to plant growth.** J. W. TIDMORE (J. Amer. Soc. Agron., 1930, 22, 481—488).—Maize, tomato, and sorghum grow in soil the displaced solution of which contains less than  $0.05$  in  $10^6$  of phosphate, but in a culture solution  $0.1$  in  $10^6$  is necessary for satisfactory growth.

## CHEMICAL ABSTRACTS.

**Soil moisture content at which barley wilts.** L. SMOLIK (Bull. Czechoslov. Acad. Agric., 1930, 166; Proc. Internat. Soc. Soil Sci., 1930, 5, 165).—Barley plants can utilise, at least partly, the hygroscopic moisture of soils, particularly in heavy soils.

## A. G. POLLARD.

**Toxic action of magnesia on sugar cane.** M. BRID (Internat. Sugar J., 1930, 32, 531—532).—Cane cultivated on certain lands in British Guiana is often subject to unsatisfactory growth in its later stages, i.e., withering and even ultimate death of stalks and stools. This "disease" is believed by the author to be due to the high  $MgO : CaO$  ratio in the soil, which sometimes may reach  $3 : 1$ ; the ash of the cane may contain as much as  $15$ — $25\%$   $MgO$ . Liming the soil has assisted in ameliorating this condition.

## J. P. OGILVIE.

**Nutritive value of pasture. VI. Utilisation by sheep of mineral-deficient herbage.** H. E. WOODMAN and R. E. EVANS (J. Agric. Sci., 1930, 20, 587—617; cf. B., 1930, 637).—Sheep-feeding experiments with herbage notably deficient in calcium and phosphates are recorded. Malnutrition on a mineral-deficient pasturage is due directly to an insufficiency of the inorganic substances necessary for body construction and the maintenance of the normal balance of minerals in the blood and tissues. It cannot be ascribed to lack of palatability or to incomplete digestion or the organic constituents of the herbage.

## A. G. POLLARD.

**Wood carbolineum as an insecticide, from the physico-chemical view-point.** S. JENČIČ (Arh. Hemiju, 1930, 4, 176—194).—The insecticidal value of wood carbolineum used for spraying fruit trees is a function of the stability of the emulsions produced.

## R. TRUSZKOWSKI.

**Degree of saturation of the adsorptive complex in Moravian soil types.** B. MALÁČ (Bull. Czechoslov. Acad. Agric., 1929, 853; Proc. Internat. Soc. Soil Sci., 1930, 5, 182—183).

**Physics and the chemical industry.** GMELIN.—See I.

## PATENTS.

**Production of fertilisers.** A. HOLZ and T. V. D. BERDELL (B.P. 336,692, 30.7.29).—Finely-ground phosphate rock is treated with a mixture of sulphuric and nitric acids. The mother-liquor, containing calcium nitrate and free phosphoric acid, is decanted from the calcium sulphate, and potassium hydroxide or carbonate is added, the dipotassium phosphate produced reacting

with the calcium nitrate to form dicalcium phosphate and potassium nitrate. The former is filtered off and dried, and the solution of the latter is evaporated to dryness. The two salts may then be mixed in any desired proportions.

## W. J. WRIGHT.

**Manufacture of artificial manure and its application to mushroom beds.** E. BLOCH (B.P. 311,405, 6.5.29. Fr., 12.5.28).—Cellulosic material (straw, sawdust, paper, etc.) is moistened with water and mixed with the products of autolysis of vegetable or animal matter (e.g., brewers' yeast). Nutrient salts may be added, and the whole allowed to ferment to produce a suitable substratum for mushroom culture.

## A. G. POLLARD.

**Immunisation of seed grain or the like [such as wood, glue, etc.].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,847 and 335,527, [A] 1.5.29, [B] 24.5.29).—(A) The halogenated hydroxylated di- and tri-arylmethanes mentioned in B.P. 334,876 (B., 1930, 1167) are used for immunisation of seed grain. (B) Seeds, wood, glue, etc. are protected from bacteria, mildew, etc. by treatment with mercury compounds of the type,  $X \cdot Hg \cdot [CH_2]_2 \cdot OR$ , in which  $X$  is hydroxyl or an acid radical and  $R$  is hydrogen, a hydrocarbon radical, or the group  $X \cdot Hg \cdot [CH_2]_2$ , and in which the methylene groups may carry substituents. Examples are:  $\beta$ -methoxyethyl mercuriacetate,  $\beta$ -ethoxyethyl mercurichloride,  $\beta$ -methoxy-*n*-propyl mercuriacetate,  $\beta$ -phenoxyethyl mercurichloride,  $\beta$ -benzyloxyethyl mercurichloride, m.p.  $81^\circ$ ,  $\beta$ -methoxyethyl mercurioxalate,  $\beta$ -methoxyethyl mercurihydroxide.

## C. HOLLINS.

**Insecticide [for plants].** W. MOORE, ASSR. to AMER. CYANAMID Co. (U.S.P. 1,761,144, 3.6.30. Appl., 7.6.27).—Stable insecticides, compatible with a large number of substances, consist of aminonitriles such as aminoisobutyronitrile and other basic nitriles, used either alone or in combination with other substances such as cresylic acid, kerosene, etc. Salts of these nitriles with organic and inorganic acids are used, and also nitriles such as acetone- and furfuraldehyde-cyanohydrins may be added to the aminonitrile compositions.

## E. II. SHARPLES.

**Superphosphate (B.P. 338,075). Leaching of phosphate rock (B.P. 338,079). Mixed phosphates (B.P. 316,550). Lime-nitrogen (B.P. 336,677).—See VII. Nicotine-containing gases (B.P. 337,311).—See XX. Combating insect pests (U.S.P. 1,758,936).—See XXIII.**

## XVII.—SUGARS; STARCHES; GUMS.

**Seeding low-grade massecuites.** G. F. MURRAY (Internat. Sugar J., 1930, 32, 595—596).—As a means of utilising vacuum-pan capacity to fullest advantage, the following method of boiling for low-grade massecuites is recommended. Enough *C*-molasses is drawn into the pan to cover the calandria and boiled under slightly reduced vacuum ( $24$ — $25$  in.) to heavy string-proof ( $92$ — $93^\circ$  Brix); steam is then shut off, the vacuum raised to normal, and  $1$  lb. of screened powdered sugar drawn into the mass to serve as seed. After  $10$ — $15$  min., when the grain is just detectable, the steam is again turned on and the strike fed with small charges

of syrup and molasses, tightening up between charges, until the crystals have reached the proper size, after which the strike is finished on molasses, being struck at about 90° Brix to a receiving tank below.

J. P. OGILVIE.

**Presence of non-sugars in sugar crystals and their effect on white sugar production.** P. HONIG (Internat. Sugar J., 1930, 32, 594).—After-product sugars were thoroughly washed free of adhering molasses, and the original massecuites, the centrifuged molasses, and the washed sugars obtained were analysed. From the results it is concluded that (a) the sugars contained twice as much ash as reducing sugars, whereas in the original massecuites the ash was only a fraction of the reducing sugars content; (b) the lime content of the ash of the original massecuites was about 10%, but this ratio had increased in the sugars to about 20%; (c) the sugars contained only about 1/20th of the iron originally present in the massecuites. Sulphitation sugars contain considerably more colouring matter than do carbonatation sugars.

J. P. OGILVIE.

**Slow-boiling massecuites.** S. A. WICKEY (Internat. Sugar J., 1930, 32, 594—595).—At Koloa factory, Hawaii, half the total lime added was used in the settlings, the heavily over-limed juice from the filters, frequently having  $p_H$  above 9.5, being returned to the mixed juice. This method of working had been found to be responsible for the slow boiling of the massecuites, which had been remedied to some extent by the addition of tallow or mineral oil to the pan. However, on reducing the  $p_H$  of the press filtrate to 8.0—8.3, an improvement in the time of boiling in the case of A- as well as of B-strikes was at once noted. This improvement was maintained so long as the  $p_H$  of the syrup entering the pans was not above 7.0. J. P. OGILVIE.

**Dilution [of sugar-factory products] in relation to comparative purities.** H. H. HILL (Facts about Sugar, 1930, 25, 568—571).—An essential to be observed in determining the direct purity is that in using the Brix hydrometer the total solids of each product should differ from the true solids as nearly as possible by the same amount as the difference present in the case of the juice. In order that the direct purity of the syrup be comparable with that of the juice from which it was obtained, it must be diluted previous to spindling to approximately the same density and non-sugar concentration as the juice. In the case of final molasses, diluting it 1:1 will give a purity more nearly comparable than if diluted to a greater degree, e.g., to the density of the juice. Syrup may be diluted to approximately the same density and non-sugar concentration as the juice.

J. P. OGILVIE.

**Effect of humidity on the deterioration of raw sugars on storage.** R. H. KING and D. SUERTE (Internat. Sugar J., 1930, 32, 542—543).—Philippine raw sugars were found to show no deterioration at a relative humidity of about 68%, but at higher values, e.g., about 84%, sucrose was rapidly destroyed. There occurred no increase in the micro-organism content at R.H. below 70%, but above 75% there was a marked increase at the same rate in all the sugars examined. Caking was found to be due to the presence of a gum of indefinite composition, which caused the sugar to

be dry at a low water content, but extremely sticky when more water was present. Essential features of the construction of a warehouse for storing raw sugar are described.

J. P. OGILVIE.

**Coloration of [sugar] juices in pressure and vacuum evaporators.** K. ŠANDERA, P. PAVLAS, and A. RUŽICKA (Z. Zuckerind. Czechoslov., 1930, 55, 9—14).—Pressure evaporators in beet factories should cause no undue coloration of the juice, the amount of colour produced on the average in a properly arranged plant being of the order of 0.65  $E_k$ , whereas in an ordinary vacuum evaporator the value may be 0.71. Actually, however, the coloration occurring depends on the several units and on their arrangements, and under unfavourable conditions, as when circulation is not normal, the pressure system may show unfavourable results.

J. P. OGILVIE.

**Volumes of sugar solutions at different temperatures.** L. KARAS (Z. Zuckerind. Czechoslov., 1930, 55, 15—16).—Gerlach's table, giving the variation of the volume of sugar solutions at different temperatures, which was based on 17.5°, has been recalculated by the author with 20° as a basis.

J. P. OGILVIE.

**Determination of the purity quotient of beet juice.** F. HERLES (Z. Zuckerind. Czechoslov., 1930, 55, 139—141).—Determination of the purity of the juice as ordinarily expressed from the beet gives unreliable results, which vary according to the subdivision of the material and to the pressure allied; reliable results are obtainable when a press, such as the Herles, capable of effecting an extremely fine subdivision of the pulp, is used. 52 G. of pulp thus obtained are shaken with 156.4 c.c. of cold water and at once filtered, the density of the filtrate being determined by hydrometer or refractometrically. Then 100 c.c. are mixed with 10 c.c. of basic lead acetate, and the mixture is filtered and polarised. This reading referred to the Schmitz table enables the purity quotient to be calculated; the same reading increased by  $\frac{1}{10}$ th gives the sugar content of the root.

J. P. OGILVIE.

**Determination of sugar by double polarisation, using baryta-alumina clarification.** J. H. HALDANE and N. DEERR (Internat. Sugar J., 1930, 32, 419—420).—Determinations were made in five factories in India on mixed juice, syrup, and molasses, using the following methods: (1) conventional direct polarisation with dry lead (Horne); (2) double polarisation (Steuerwald); (3) direct polarisation with baryta and alumina clarification, the volume being corrected; and (4) double polarisation, according to the same procedure elaborated by Deerr (B., 1915, 503). Accepting that direct polarisation with lead is the commonly used method, and that double polarisation with baryta-alumina clarification gives the least incorrect results, the error amounts to 0.68%, an exactly equal error being introduced if the conventional double-polarisation method with lead be used. A few determinations were made by the double-polarisation method using invertase as hydrolyst, and the results agreed within experimental error of observation with those obtained by method (4) above.

J. P. OGILVIE.

**Adsorption from sugar solutions.** I. Sucrose adsorption of activated carbons. J. VACHA (Z.

Zuckerind. Czechoslov., 1930, 55, 123—127).—Vašátko's experiments (B., 1927, 952; 1928, 29) have been repeated with modern carbons made by impregnation and heat-activation methods, and the same general rules in respect of sucrose and water adsorption were found to obtain. Following are the  $k$  values now found: Standard Norit 0.197; Supra-Norit 3X 0.583, Carboraffin 0.653. J. P. OGILVIE.

**Density of wet and dry beet pulp.** F. KRYŽ (Z. Zuckerind. Czechoslov., 1930, 55, 128—129).—Pulp separated from raw beet juice by the factory catchers was washed with water and pressed between filter-paper, its density (the average of 10 determinations, pycnometrically, using petroleum) being found to be 1.060. Practically the same figure was obtained with the unwashed pulp. Pulp similarly obtained and dried at 105° had  $d$  1.245. The air-dried pulp was found to weigh 279 g. per litre. Exhausted diffusion pulp polarising 44.5% after washing and drying between filter-paper to a dry-substance content of 5.95% had  $d$  0.935, weighing 677.2 g. per litre. This pulp after drying at 105° had  $d$  1.023; its average weight per litre was 123.9 g. J. P. OGILVIE.

**Isolation of choline from beet molasses.** V. STANĚK (Z. Zuckerind. Czechoslov., 1930, 55, 103—105).—It is found that even at a dilution of 0.002%, and in the presence of 20% of sugar, choline can be precipitated by means of Reinecke's salt. Betaine, at least in pure solution, is separated by this reagent only in acid solution. This method was used for the isolation of choline from beet molasses, 0.028—0.049% being thus obtained. A little betaine was also carried down, but this was separated by the crystallisation of the hydrochlorides from absolute alcohol. J. P. OGILVIE.

**Calculation of production in raw sugar factories and refineries.** V. SÁZAVSKÝ (Z. Zuckerind. Czechoslov., 1930, 55, 105—113).—A method of calculating the various products and by-products from the periodic inventory is given, this being as accurate as the usual procedure, but more direct. Attention is called to the value in such calculations of the ash coefficient as rapidly determined conductimetrically. J. P. OGILVIE.

**Toxic action of magnesia on sugar cane.** BIRD.—See XVI. Ethyl alcohol. BACKHAUS. Lactic acid. GARRETT.—See XVIII.

#### PATENTS.

**Manufacture of beet sugar.** ARCOS, LTD. From I. TISCHTSCHENKO and V. TCHEFRAKOV (B.P. 330,685, 11.4.29. Addn. to B.P. 329,112; B., 1930, 1085).—The thick juice obtained as described in the prior patent is passed directly from the diffusion batteries, through charcoal or sand filters, to the boilers without further purification. A. R. POWELL.

**Fermentation of sugars** (B.P. 315,002). **Growth of yeast** (U.S.P. 1,761,515).—See XVIII.

### XVIII.—FERMENTATION INDUSTRIES.

**Analysis of hops.** W. WÖLLMER (Woch. Brau., 1930, 47, 521—525).—Sampling and preparation should be according to the directions of Meindl. Exact details are given for carrying out the determinations of total and

hexane-soluble resins and humulone. Fine comminution of the sample is important as favouring sampling, rapid extraction, and dense settling of the extracted residue. It is shown that practically complete extraction may be obtained in  $\frac{1}{2}$  hr. by shaking with ether in the cold; the 2 hrs.' extraction specified is ample. If the light petroleum extract does not settle clear it is preferably treated by centrifuging. In extracting 15 c.c. of the methyl-alcoholic extract with light petroleum the added water may vary from 10 to 35 c.c. without appreciably affecting the results. The lead acetate solution used for precipitation of the humulone may contain up to 1% of free acetic acid without affecting the precipitation; the slightly higher results obtained by the use of a less acid reagent is attributed to slight precipitation of basic lead salts. F. E. DAY.

**Modern yeast industry.** C. N. FREY (Ind. Eng. Chem., 1930, 22, 1154—1162).—An outline is given of the history of the yeast industry and of the various theories of fermentation which have arisen during the development of the industry. Most of the yeast output is taken by the bread industry. Smaller amounts find use in applications based on its vitamin content, protein value, or its content of invertase. It is an important therapeutic agent, and increasing quantities are being used in the manufacture of synthetic plastics, in sewage disposal, etc. C. RANKEN.

**Determination of the liquefying power of malt amylase.** L. FLETCHER and J. B. WESTWOOD (J. Inst. Brew., 1930, 36, 550—557).—The liquefying power of malt is determined by measuring the diminishing viscosity of a uniform and stable starch paste to which malt amylase is added, and which is prepared according to the method of Jozsa and Gore. In their determinations of the liquefying power these workers made the assumption of a proportionality between the enzyme and the substrate over the whole range of action. It is now proved that the assumption is incorrect and that the changes comply within certain limits with Kjeldahl's law of proportionality. In addition, the viscosity of the starch paste over a considerable range of concentration is a linear function of the square root of the concentration of the starch. C. RANKEN.

**Determination of the "tannin-nitrogen" and the "tannin number" as a new method for defining the protein conditions in beer.** H. FINK and W. RIEDEL (Woch. Brau., 1930, 47, 491—495, 501—505, 511—515).—The authors designate the nitrogen precipitable by tannin as "tannin-nitrogen" and define the "tannin numbers I and II" as the percentages of the total nitrogen precipitated by additions of 1 pt. of 0.1% and 0.2% tannin solution, respectively, to 9 pts. of beer. The amounts of nitrogen so precipitated vary widely with different beers; for any one beer it is not affected by the rate of addition of the reagent or by the temperature. Dark beers give a lower tannin number than pale beers, and the value is unaffected by pasteurisation. It is considered that these numbers are a better guide to the presence of proteins likely to cause haze than is the heat-coagulable nitrogen determined usually. It is noted that precipitation of the "tannin-nitrogen" increases the capacity of the beer for head formation. F. E. DAY.

**Action of ultra-violet rays on the germination of barley in malting.** R. DE FAZI (Atti R. Accad. Lincei, 1930, [vi], 11, 1022—1024).—The action of ultra-violet rays on germinating barley accelerates the germination even at low temperatures 10—15°, prevents mould growth, facilitates the formation of enzymes, and increases the vitamin content. Experiments on the large scale show that malt of improved quality is obtained in this way. T. H. POPE.

**Fermentation problems.** E. I. FULMER (Ind. Eng. Chem., 1930, 22, 1148—1150).—The chemical changes produced by the action of micro-organisms on carbohydrates are complex, and are profoundly affected by the physical and chemical environment. There is as yet no explanation of the phenomena involved in synergistic action in which two organisms cause chemical actions which neither alone can effect. A chart is given which shows the interrelationships between substrates and products of fermentation. C. RANKEN.

**Fermentation and maturation of beers.** L. NATHAN (J. Inst. Brew., 1930, 36, 538—550).—The author describes his system of brewing in which the boiled wort after passing through the hop back is collected in a closed aluminium container furnished with a series of horizontal aluminium plates which can be withdrawn from the liquid, and on which the wort deposit ("trub") settles from the cooled wort. The hot wort is pumped from the top of this vessel to a refrigerator situated in a chamber to which sterile air alone is admitted, and from which the steam is exhausted. The cooled sterile wort is returned to the bottom of the original container, where it displaces, but does not mix with, the hot wort. When all the wort is chilled the "trub" is allowed to settle out on the horizontal plates and the clarified wort is run to the fermenting vessels, which consist of closed, conical, aluminium vessels fitted with cooling jackets; the vessels are sterilised by alcohol vapour. The wort is fermented at 3—5° by a pure culture yeast forced by pressure of carbon dioxide from the yeast vessel into the fermentation vessel and thoroughly mixed into the wort by sterile air. The fermentation carbon dioxide is collected and purified, and is used in the subsequent operations. When the yeast has settled and has been withdrawn, the beer is matured by blowing the pure fermentation gas through the beer until the latter is freed from all immature flavours. After this washing process, which lasts for 24—36 hrs., the beer is cooled to 0—1°, saturated with carbon dioxide at 0.3—0.4 atm., and left for 12 hrs. to allow any remaining yeast to deposit. C. RANKEN.

**Wines of Comtat-Venaissin.** J. BORDAS and G. MATHIEU (Ann. Falsif., 1930, 23, 454—467).—A discussion of the quality of the wines of Vaucluse with respect to their geographical and geological situation. B. W. TOWN.

**Application of adsorption to sterilisation of wines.** P. MALVEZIN (Bull. Soc. chim., 1930, [iv], 47, 1194—1196).—Enzymes causing alcoholic fermentation and organisms giving rise to diseases can be completely removed from wine by treatment with adsorbents such as kaolin and kieselguhr. For the removal of iron activated carbon is recommended. R. CUTHILL.

**Butyl [alcohol]-acetone fermentation industry.** C. L. GABRIEL and F. M. CRAWFORD (Ind. Eng. Chem., 1930, 22, 1163—1165).—The history of the industry is outlined, and a brief description is given of the present commercial process. The process was first used as a source of raw material for synthetic rubber, and was exploited during the war as a source of acetone. An increased demand for *n*-butyl alcohol has recently been created by the discovery that *n*-butyl acetate is superior to amyl acetate as a lacquer solvent. C. RANKEN.

**Ethyl alcohol.** A. A. BACKHAUS (Ind. Eng. Chem., 1930, 22, 1151—1153).—The methods used in carrying out the large-scale fermentation of a molasses mash are described, and the theories concerning the mechanism of alcoholic fermentation as developed by Harden and by Neuberg are discussed. C. RANKEN.

**Lactic acid.** J. F. GARRETT (Ind. Eng. Chem., 1930, 22, 1153—1154).—Lactic acid is manufactured by the comparatively high-temperature fermentation of cane or beet sugar, maize starch, or maize sugar by a pure culture of lactic bacteria. The fermentation is completed in 5—6 days, and the acid is recovered as a technical or a food product. The leather industry consumes 80—90% of the entire output, and small amounts are used in food and beverage products. C. RANKEN.

**Production of fuel gas by anaerobic fermentations.** A. M. BUSWELL (Ind. Eng. Chem., 1930, 22, 1168—1172).—A summary is given of the recent work on the production of methane by the biological decomposition of municipal wastes. Such fermentations result in a 90% conversion of the sludge into a stoichiometric yield of carbon dioxide and methane, and, provided proper conditions are maintained, a fuel gas containing 50% or more of methane can be obtained. C. RANKEN.

**Fermentation in the food industries.** F. C. BLANCK (Ind. Eng. Chem., 1930, 22, 1166—1168).—A survey is made of the fermentation processes involved in the manufacture of bread, tea, coffee, cocoa, sauerkraut, pickles, vinegar, sauces, olives, cheeses, etc. C. RANKEN.

**Minor industrial fermentations.** O. E. MAY and H. T. HERRICK (Ind. Eng. Chem., 1930, 22, 1172—1176).—A review is given of the fermentation methods used for the production of citric, gluconic, and gallic acids, and glycerol. C. RANKEN.

**Briquetting of pulp.** GOODWIN. **Alcohol as motor fuel.** UHLMAN.—See II.

See also A., Dec., 1620, **Influence of catalytic elements on alcoholic fermentation** (ROSENBLATT and MARCH). 1621, **Acetone fermentation.** *Bacillus macerans* (ZACHAROV).

#### PATENTS.

(A) **Manufacture, (B) growth, of yeast.** A. K. BALLS, Assr. to M. NILSSON and A. W. HIXSON (U.S.P. 1,759,536 and 1,761,515, [A] 20.5.30, [B] 3.6.30. Appl., [A] 24.12.25, [B] 24.1.24).—(A) The hydrogen-ion concentration of a carbohydrate and phosphate mash is controlled by the use of carbamide as the nitrogen nutrient, and by the periodic addition of a salt such as calcium lactate during



the period of increasing acidity of the fermenting mash. When the period of decreasing acidity is reached in the fermenting mash, salts such as ammonium chloride and sulphate are added also. (B) Yeast is grown in a medium consisting of molasses mixed with juice expressed from sugar cane. C. RANKEN.

**Manufacture of yeast.** DISTILLERS CO., LTD., and E. A. MEYER (B.P. 334,502, 1.5.29).—In the continuous process the growth of undesirable organisms is inhibited and the yeast stimulated by treatment of the wort with halogens, certain alkaloids, chloramine-T, etc. The sterilising agents may be added also to the nutrient solution or to the water which is added continuously to the fermenter. C. RANKEN.

**Production of enzymes and particularly proteolytic enzymes secreted by micro-organisms for use in tanning, skin dressing, and the like.** A. R. BODIN and J. A. EFFRONT (B.P. 313,874, 4.6.29. Fr., 18.6.28).—A low proportion of protein nitrogen in the fermenting mash is maintained by the addition of 20–50 g. of carbohydrates (dextrin) per litre to the nutritive medium, which may consist of residues from oil mills. Addition of 5–10 g. of alkaline-earth salts increases the protease secretion and maintains a  $p_H$  of 6–7. The whole is intensively aerated under pressure, with alternations of compression and expansion, and even of pressure and vacuum. C. RANKEN.

**[Production of acetone and butyl alcohol by] fermentation of sugars.** C. F. BURGESS LABS., INC., Assees. of J. A. VILJOEN (B.P. 315,002, 5.7.29. U.S., 6.7.28).—A starter mash which contains less than 3% of sugars (6% of molasses) and a protein such as keratin or soya-bean meal is inoculated with *Clostridium acetobutylicum* in a maize suspension. When the bacilli reach the highest possible concentration, a second mash containing at least 8% of sugars (16% of molasses) is gradually mixed with at least an equal volume of the starter mash, and the combined mashes are allowed to ferment. Butyl alcohol and acetone are then distilled from the fermented mash by the ordinary methods. The amount of proteolysable protein in the starter mash must be sufficient to furnish enough buffer nitrogen to neutralise the organic acids formed during the fermentation of the final mash and to maintain the final mash at  $p_H$  4.4–4.8. C. RANKEN.

**Production of a ferment.** W. MATZKA (U.S.P. 1,781,788, 18.11.30. Appl., 2.3.26. U.K., 8.4.25).—See B.P. 259,895; B., 1926, 1027.

**Anhydrous alcohol** (U.S.P. 1,751,211).—See III. **Tanning substances** (B.P. 336,984).—See XV. **Artificial manure** (B.P. 311,405).—See XVI. **Treatment of cereals etc.** (B.P. 311,424). **Dough bleaching product** (B.P. 335,943).—See XIX.

## XIX.—FOODS.

**Fat of asses' milk.** O. LAXA (Ann. Falsif., 1930, 23, 474–475).—A complete analysis is given.

B. W. TOWN.

**Cause of beetroot-like odour and taste in milk and butter.** P. POST (Pharm. Weekblad, 1930, 67, 1309–1313).—Feeding conditions liable to cause the

appearance of a beetroot-like taste and odour in cows' milk and in butter are discussed. By distillation of a sample of such milk, after rendering alkaline with calcium hydroxide, trimethylamine has been isolated, whereas ordinary milk is free from this substance; on addition of 10 mg. of trimethylamine to 100 c.c. of milk, the characteristic taste immediately develops. The amine is probably produced by hydrolysis of betaine, and since it exists in the butter in combination with fatty acids simple washing does not effect its removal; if, however, the butter be washed first with very dilute acid (lactic, citric, hydrochloric, etc.) and then with water the amine is completely removed and the objectionable taste and odour simultaneously vanish.

H. F. GILLBE.

**Use of plant lecithin in confectionery.** H. HAUPT (Deut. Nahrungsmittel-Rundsch., 1930, 17–20; Chem. Zentr., 1930, i, 2178).—The use of soya-lecithin instead of egg-yolk is reported. Denaturation of plant lecithins, e.g., with sesame oil, is advocated. A. A. ELDRIDGE.

**Composition and feeding value of tapioca roots.** N. HANSSON and S. BENGTSSON (Tierernährung, 1930, 1, 369–387; Bied. Zentr., 1930, 59, 499–501).—Tapioca meal has a high starch content, but is somewhat deficient in protein and fat. Used in conjunction with protein concentrates it forms a valuable fattening food for pigs.

A. G. POLLARD.

**Determination of phosphoric acid in organic materials, especially feeding stuffs.** W. LEPPER (Landw. Versuchs-Stat., 1930, 111, 159–161).—The sample (5 g.) is mixed with 2 g. of calcium carbonate and ashed. The residue is rinsed into a 500-c.c. flask, the dish being finally rinsed with a little nitric acid. The phosphate in the ash is dissolved (cold) by adding 20–25 c.c. of nitric acid and, after adjusting the volume determined in 15 c.c. of the filtrate by the Lorenz method. The method yields results in agreement with those obtained by the Kjeldahl and sulphuric-nitric acid digestion methods.

A. G. POLLARD.

**Use of copper sulphate in the place of mercury in Kjeldahl decompositions.** W. LEPPER (Landw. Versuchs-Stat., 1930, 111, 155–158).—The use of varying amounts of potassium sulphate in Kjeldahl digestions did not affect the nitrogen values recorded. For the determination of the crude protein in feeding stuffs, 1 g. of material with 1 g. of copper sulphate, 15 g. of potassium sulphate, and 20 c.c. of sulphuric acid are used. After the solution has clarified (15 min.), heating is continued for 30 min. For 5-g. samples the proportion of sulphuric acid is increased to 50 c.c. and the final heating period to 1 hr.

A. G. POLLARD.

**Yeast industry.** FREY. **Fermentation in food industries.** BLANCK.—See XVIII. **Phloroglucinol in vegetable products.** NIETHAMMER.—See XX.

## PATENTS.

**Treatment of cereals and leguminous products.** H. H. and H. LAHMANN (DR. LAHMANN'S SANATORIUM WEISSER HIRSCH) (B.P. 311,424, 11.5.29. Ger., 12.5.28).—A product of high nutritive value, containing vitamins and vitasterols, is prepared from the bran and germ of cereals by adding 5–10% of a diastatic barley malt

and digesting with distilled water at 55–65° with exclusion of air. Bitter substances are removed from the aqueous extract by steam distillation or evaporation *in vacuo*.  
E. B. HUGHES.

**Dough bleaching product and its preparation.** D. VERON (B.P. 335,943, 3.7.29).—Ground soya beans are wetted with a green malt infusion and kept at 15.5° for 12 hrs., after which the product is re-ground and bolted. Addition of 0.5–2% of this to flour has a bleaching effect in baking due to carotene-destroying enzyme in the preparation.  
E. B. HUGHES.

**Manufacture of leavened bread.** H. A. GILL. From STANDARD BRANDS, INC. (B.P. 337,930, 11.10.29).—A bread improver consists of a mixture of urea, urease, and mucic acid or acid calcium phosphate. The urea decomposes to give ammonia, which assists in producing a satisfactory colour of crust and is afterwards neutralised by the slightly soluble acid ingredient.  
E. B. HUGHES.

**Manufacture of whey products.** N. L. SIMMONS (U.S.P. 1,763,633, 10.6.30. Appl., 2.4.30).—Whey is concentrated to a 70% solids content in a vacuum pan and subsequently cooled, when the material sets to a pasty mass owing to the crystallisation of the lactose and the salts. The drying is completed in a current of warm air at a temperature below that of the caramelisation of lactose.  
E. B. HUGHES.

**Extraction of tea aroma.** O. STROBACH and P. A. WICKMANN (B.P. 335,901, 1.7.29).—Tea is extracted with a suitable solvent (light petroleum, chloroform, etc.) and the extract purified by dissolution in alcohol, removal of the latter, and redissolution in petroleum spirit. The final product is a yellow-green to golden-yellow paste.  
E. B. HUGHES.

**Desiccation of foodstuffs.** A. W. KOON, ASSR. to A. M. KOON (U.S.P. 1,759,702, 20.5.30. Appl., 7.9.29).—An apparatus is described for the desiccation of such substances as fodder, forage, etc. The finely-divided material is blown along a system of conduits in an atmosphere of steam and inert gaseous products of combustion, at 150–650°, mechanically collected, and cooled by air-blast.  
E. B. HUGHES.

**Preservation of food products.** R. A. LEGENDRE (B.P. 336,014, 13.7.29. Belg., 3.11.28).—Mixtures of straw and molasses used as cattle feeds may be preserved from mould attack by increasing the  $p_H$  to 6–11 by treatment with a dilute alkali (e.g., sodium carbonate or ammonia gas).  
E. B. HUGHES.

**Production of vitamin food.** H. PLACAK, ASSR. to G. E. CONKEY Co. (U.S.P. 1,764,085, 17.6.30. Appl., 3.9.27).—A vitamin food for poultry and animal feeding is produced by mixing into melted paraffin wax (a) cod-liver oil, (b) dry yeast, soya-bean meal, etc., to produce a granular product, and then allowing the mixture to cool.  
E. B. HUGHES.

**Production of caseinates.** W. O. FROHRING and W. F. RICHARDS, ASSRS. to LABORATORY PRODUCTS Co. (U.S.P. 1,762,169, 10.6.30. Appl., 20.4.27).—Casein obtained from washed acid curd is dried in a current of hot air, and added as required to a solution of freshly precipitated and washed (but not dried) casein in

alkali (sodium bicarbonate). The mixture is then spray-dried.  
E. B. HUGHES.

**Kiln for automatic steam-cooking [of tapioca etc.] and treatment with aeriform fluids.** R. L. M. MORIN (B.P. 338,286, 28.9.29. Fr., 3.10.28).

**Nicotine-containing gases** (B.P. 337,311).—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Preparation of [stable] concentrated phosphorus solutions [for pharmaceutical use].** M. H. P. SITSSEN (Pharm. Weekblad, 1930, 67, 1313–1316).—About 5 g. of phosphorus are dried, washed with ether, dried with filter paper, and, after weighing in a tared vessel containing liquid paraffin, added to an almost full 1-litre flask containing liquid paraffin which has been heated to 150° and cooled; the mixture is heated at 60° to effect dissolution, a few drops of ether being added to the flask to hinder evaporation and oxidation of the phosphorus. A volume of the solution containing 5 g. of the element is transferred to a slightly larger flask and is diluted to 991 g. by addition of dry paraffin; finally, 4 g. of lemon oil and 5 g. of ether are added.  
H. F. GILLBE.

**Assays of some official iron preparations containing organic matter.** W. P. BRIGGS (J. Amer. Pharm. Assoc., 1930, 19, 1191–1198).—Satisfactory methods for the analysis of the following solutions are described: peptonised iron and manganese, N.F.V., albuminised iron, N.F.V., iron and ammonium acetate, U.S.P. X. The U.S.P. X method for the assay of iron in the last compound is considered inaccurate.  
E. H. SHARPLES.

**Rapid iodometric determination of iron in scale and other pharmaceutical salts.** G. J. W. FERREY (Quart. J. Pharm., 1930, 3, 471–481).—The method of U.S.P. X is criticised and various modifications of the original Mohr method are discussed. In the presence of 6 mols. of potassium iodide per atom of ferric iron, reduction to ferrous iron occurs rapidly if the concentration of hydrochloric acid is sufficiently great (cf. Grey, A., 1929, 286). Iodine may be titrated accurately with 0.1N-thiosulphate in the presence of 3N-hydrochloric acid. Based on these observations, details are given for the determination of iron in pharmaceutical preparations. In the case of organic salts, the solution acidified with hydrochloric acid is boiled for 15 sec. and cooled before titration to ionise the iron.  
R. CHILD.

**Effect of buffering the outer phase on the stability of certain [mineral oil] emulsions.** J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1930, 19, 1181–1185).—Using acacia as emulsifier and buffered solutions as the outer phase, no variation in stability of mineral oil emulsions was observed within the  $p_H$  range 2–10.5. With tragacanth the emulsions are most stable within  $p_H$  2–5, but without a buffered outer phase the most stable range is  $p_H$  1.9–2.3 (cf. B., 1929, 861; A., 1929, 1234).  
E. H. SHARPLES.

**Determination of mercury in pharmaceutical preparations.** F. REIMERS (Dansk Tidsskr. Farm., 1930, 4, 213–229).—The method of Spacu and Suciu (A., 1929, 901) has been modified as follows to permit

its use for the determination of mercury in pharmaceutical substances. The neutral or weakly ammoniacal solution of the preparation (which must contain about 0.2 g. of mercuric salt in 100–200 c.c.) is treated with sufficient 2*N*-potassium iodide to provide an excess of at least 0.5% in the liquid. After heating nearly to boiling, the freshly made ethylenediamine-cupric reagent (prepared by mixing 20 c.c. of 0.5*M*-copper sulphate with 80 c.c. of 0.5*M*-ethylenediamine and 100 c.c. of water) is added in sufficient amount to give 10 c.c. in excess for each 100 c.c. of solution. The whole is quickly cooled while being stirred, the precipitate filtered and then washed, first with a dilute solution of the precipitant containing potassium iodide and finally with alcohol and ether. The porcelain filter crucible and precipitate are then weighed after drying in a vacuum desiccator for 10 min. In the case of plasters and ointments the material is first extracted with ether-light petroleum or acetone, the solution separated by filtration, and the residue transferred with the filter to a flask provided with a ground-in distillation tube dipping into water to condense any volatilised mercury compounds. The organic matter is destroyed by treatment with concentrated sulphuric and nitric acids, and the mercury in the resulting solution is determined as above after addition of the liquid from the receiver.

H. F. HARWOOD.

**Diacolation [percolation] of condurango bark.** H. BREDDIN (*Pharm. Ztg.*, 1930, **75**, 1261–1262).—The preparation of the extract by the author's system of countercurrent percolation is described.

S. I. LEVY.

**Determination of total alkaloids in cinchona bark.** P. A. W. SELF and C. E. CORFIELD (*Quart. J. Pharm.*, 1930, **3**, 410–416).—The B.P., D.A.B. VI, and U.S.P. X methods of assay are considered unreliable and the following method is recommended. The bark (10 g. in No. 60 powder) is mixed with lead subacetate solution followed, in 1 hr., by ammoniacal alcohol, and the whole, after further mixing, is set aside for 1 hr., transferred to a continuous extractor with more of the ammoniacal solvent, and extracted for 3–4 hrs. The greater part of the alcohol is then distilled off from the extract, 0.2*N*-sulphuric acid added, and the solution heated to boiling, cooled, and filtered through cotton wool; the residue is treated with boiling 0.1*N*-sulphuric acid, cooled, filtered, and washed with cold acidulated water until the washings give no opalescence with Mayer's reagent. The combined filtrate and washings are shaken with three successive portions of chloroform, the extract being run off into a separator containing 0.25*N*-sulphuric acid and shaken with the latter before rejection; this last lot of acid is added to the contents of the first separator, the whole basified with ammonia, and alkaloids are extracted completely with chloroform. The chloroform extract is washed with water and evaporated to dryness; the residue is dissolved in alcohol, evaporated, dried at 100°, and weighed.

R. CHILD.

**Determination of morphine in opium.** F. REIMERS (*Dansk Tidsskr. Farm.*, 1930, **4**, 230–258).—A review of the methods employed for the above purpose, especially those developed during the last ten years.

Physical (refractometric and polarimetric) methods and those based on direct precipitation or oxidation of morphine do not yield reliable results. Full details are given of the most satisfactory methods, which depend on the precipitation of the morphine by ammonia from either an aqueous extract or one obtained by treating the opium with milk of lime; other alkaloids present are removed by shaking with benzene or other solvent. The morphine is finally determined by weighing or titration. Experiments on the absorption spectra of morphine solutions showed that appreciable oxidation to oxydimorphine occurs in alkaline solution even at the ordinary temperature; in acid solutions oxidation takes place only on prolonged boiling. Whilst in a 1% solution having  $p_H$  5.98 only 3.4% was oxidised after 2 hrs.' boiling, in a solution having  $p_H$  11.7 more than half the morphine was oxidised in the same period.

H. F. HARWOOD.

**Colorimetric test for determination of adrenaline.** J. RAE (*Pharm. J.*, 1930, **125**, 451).—The ammonium molybdate method described previously for tannic acid (A., 1928, 539) is applied to adrenaline. To liquor adrenaline hydrochlor. (1 c.c.) is added 10% aqueous ammonium molybdate (2 c.c.) and the mixture is diluted in a Nessler cylinder to 50 c.c. The depth of colour is compared with that given by an adrenaline solution of known concentration or by a standard solution of tannic acid; 1% aqueous tannic acid is treated as above, and 5 c.c. of the coloured solution are diluted to 50 c.c., the colour thus obtained matching that given by 1 c.c. of liq. adrenal. hydrochl. B.P. Cocaine hydrochloride, keroacaine, stovaine, benzamine lactate, borax, sodium chloride, boric acid, chlorbutol, aq. chlorof., aq. rosæ, and aq. sambuci do not interfere; reducing agents (ferrous salts, bisulphites, alcohol, etc.) and urine interfere.

R. CHILD.

**Dioxypyramidone, a new analgesic.** H. SZANCER (*Pharm. Zentr.*, 1930, **71**, 675–677).—Dioxypyramidone (cf. Charonnat and Delaby, A., 1930, 329, 351) does not give the characteristic colour reactions of antipyrine or pyramidone, which, as adulterations, are easily detected by means of nitrous acid. It reduces Fehling's solution, gives with potassium ferrocyanide and warm hydrochloric acid a dark green coloration, and when heated with sodium hydroxide yields sodium oxalate and alkaline vapours.

H. E. F. NOTTON.

**Microchemical localisation of phloroglucinol and its derivatives in vegetable products and drugs.** A. NIETHAMMER (*Pharm. Zentr.*, 1930, **71**, 673–675).—The results of tests using the vanillin-hydrochloric acid, diaminobenzaldehyde-sulphuric acid, and vacuum-sublimation methods on a large number of dried roots, rhizomes, and barks, also seeds, fresh fruit, and flowers are given. Phloroglucinol is widely, but not universally, distributed, and its identification in a vegetable product may be useful for purposes of characterisation.

H. E. F. NOTTON.

**Reactions of anæsthesin.** L. EKKERT (*Pharm. Zentr.*, 1930, **71**, 677).—Modification of the diazotisation and the indophenol colour reactions for anæsthesin are described.

H. E. F. NOTTON.

**Reaction of atophan and novatophan.** L. EKKERT (Pharm. Zentr., 1930, 71, 678).—The test previously described for these compounds (A., 1928, 139) may be used also to distinguish between them. The red solution from atophan with  $\beta$ -naphthol and fuming hydrochloric acid remains clear when decolorised by sodium hydroxide, whilst that from novatophan becomes milky from separation of the base. H. E. F. NOTTON.

**Vacuum microsublimation of synthetic drugs.** R. EDER and W. HAAS (Mikrochem., Emich Festschr. 1930, 43—82).—Various methods available for the sublimation of small quantities of material are described. The types of sublimate obtained with Eder's microsublimation apparatus and with Diepolder's vacuum apparatus are described, and details, including photomicrographs, are given of the results obtained with a number of synthetic drugs. Four groups are recognised, according to the ease with which a crystalline sublimate is obtained. Although the m.p. is of little significance for the sublimation process, the difference between the sublimation temperature and the m.p. is a deciding factor as to whether or not well-defined sublimate can be obtained. Tables are given showing for a number of drugs the m.p., initial and optimum sublimation temperatures, and the nature of the sublimate, and these data, in conjunction with the crystal form of the sublimate, serve in most cases for identification. Separation of a mixture of such compounds by sublimation is rarely possible, as for the majority the sublimation temperature lies between 100° and 140°.

H. F. GILLBE.

**Crystallographic investigation of microsublimates of synthetic drugs.** W. HAAS (Mikrochem., Emich Festschr., 1930, 83—119).—Details are given of the determination of the optical properties of sublimate, and tabulated data are presented for a number of synthetic drugs, comprising the appearance and crystalline form of the sublimate, the extinction, interference colours, optical character, dispersion, and refractive indices  $\alpha$ ,  $\beta$ , and  $\gamma$ .

H. F. GILLBE.

**Steam-distillation of nicotine from tobacco.** A. SCHMUK and M. CHMURA (U.S.S.R. State Inst. Tobacco Inv., Bull. 69, 1930, 71—80).—Addition of lime and sodium chloride to tobacco not only shortens the time of steam-distillation, but results in increased yields of nicotine.

T. H. POPE.

**Free nicotine or free alkalinity of tobacco.** A. SCHMUK and M. CHMURA (U.S.S.R. State Inst. Tobacco Inv., Bull. 69, 1930, 65—69).—The content of nicotine in tobacco is related to the reaction of the material. Distillation in a current of steam of tobacco which has not been rendered alkaline yields high proportions of nicotine (35—40%), which increases progressively as increasing amounts of alkali are added. Extraction of tobacco with light petroleum gives considerably smaller yields of nicotine. The proportion of free nicotine present is much greater than that calculated theoretically from the degree of hydrolysis of nicotine salts and the dissociation constants.

T. H. POPE.

**Determination of pectin substances in tobacco.** V. BALABUCHA-POPCOVA (U.S.S.R. State Inst. Tobacco

Inv., Bull. 69, 1930, 55—64).—The pectic substances in tobacco may be determined by extracting the tobacco on a water-bath with several portions of 0.5% ammonium oxalate solution precipitating the pectins from the united extracts by addition of alcohol, boiling the precipitate with 12% hydrogen chloride solution, collecting the carbon dioxide generated in standard baryta solution, and titrating the latter. A simpler method, which gives results comparable with those of the above procedure, consists in extracting the tobacco with hot 75% alcohol, boiling the extracted tobacco with 12% hydrochloric acid, and determining the liberated carbon dioxide as above. Since the encrusting substances of the cellulose of tobacco also yield appreciable amounts of carbon dioxide, it is necessary to diminish the result obtained by 0.5% when the cellulose content of the tobacco reaches 10%; 1 pt. of carbon dioxide  $\equiv$  5.66 pts. of pectic acid. This method is accurate to within 2—3%. Gabel and Kiprianov's method (A., 1929, 1347) gives results which may be too high to the extent of 35%.

T. H. POPE.

**Determination of the essential oils of tobacco.** V. V. SEMENOVA (U.S.S.R. State Inst. Tobacco Inv., Bull. 69, 1930, 45—53).—The following procedure does not determine the total essential oils in tobacco, but serves for obtaining a comparative measure of the proportions present in different tobaccos. A weighed quantity (5 g.) of the finely-divided tobacco, heated in a flask in a boiling water-bath, is distilled in a current of steam under definite conditions, 4 c.c. of water being distilled from a small boiler to furnish the steam. Air is drawn through the apparatus at constant rate (30 bubbles in 15 sec.) during the whole time of the distillation and for 30 min. afterwards, and passes through three washing vessels each containing 3 c.c. of sulphuric acid ( $d$  1.84). The whole of the acid, together with washing water, is made up to 20 c.c. and mixed, and is then compared colorimetrically with standard solutions of tobacco essential oils in sulphuric acid.

T. H. POPE.

**Electrodeposition of metals and its use in pharmacy.** LIVERSEDGE.—See X. Cod-liver oil. DYER and WOKES.—See XII. Yeast industry. FREY.—See XVIII.

See also A., Dec., 1561, Glucosides of *Digitalis lanata* (MANNICH and others). 1579, Naphthyl esters and naphthylamides of chaulmoogric acid (DE SANTOS and WEST). 1593, Piperidine derivatives (CROOK and McELVAIN). Local anæsthetics derived from quinoline and isoquinoline (BAILEY and McELVAIN). 1597, Stereoisomerism of carnosine (GULEVITSCH). 1598, 1:3:4-Triazoles (BHAGAT and RAY). 1600, Synthesis of thiazoleamines (HINEGARDNER and JOHNSON). Alkaloids of yohimbine (LILLIG). 1602, Mercuration of some polyhydroxybenzaldehydes and their monomethyl ethers (HENRY and SHARP). 1605, Colour reactions of eugenol and clove oil (SZANCER). Colorimetric determination of alkaloids (ROJAHN and SEIFERT). 1623, Hormone of the adrenal cortex (HARTMAN and BROWNELL). 1624, Preparation of the gonad-stimulating hormone (DICKENS). 1626, Alkaloids in *Buxaceæ* (MARTIN-SANS). 1627, Active

constituent of "Hsi-sin" (KONDÔ). 1628, Polyphenols etc. in tobacco (SCHMUK and others).

#### PATENTS.

**Preparation of *Digitalis* glucoside.** WELLCOME FOUNDATION, LTD., and S. SMITH (B.P. 337,091, 8.8.29).—The total glucosides of the leaf of *Digitalis lanata* are treated with acetone or methyl ethyl ketone; sparingly soluble glucosides are separated, and the filtrate is fractionally precipitated with water. When no more solid separates the liquid is saturated with salt and the precipitate extracted with methyl or ethyl alcohol. On dilution of the extract with water, crystals of a glucoside having m.p. 265° (corr.),  $[\alpha]_{D}^{25} +17.9^{\circ}$  (1.5% in equal vols. of methyl alcohol and chloroform) separate. It is purified by treatment with chloroform and/or acetone or methyl ethyl ketone and crystallisation from dilute methyl or ethyl alcohol or from pyridine by the addition of water or ether. Further quantities may be obtained by treating the aqueous filtrate from the salt precipitate with chloroform. On hydrolysis the glucoside gives a *genin* having m.p. 222° (corr.),  $[\alpha]_{D}^{20} +27^{\circ}$  (in methyl alcohol 1.7), and digitoxose.

E. H. SHARPLES.

**Ephedrine oil solution.** E. H. STUART, ASSR. to E. LILLY & Co. (U.S.P. 1,762,108, 3.6.30. Appl., 10.5.29).—Solutions of ephedrine in oil, particularly mineral oil, that will maintain the ephedrine in solution under all ordinary conditions, are prepared by the addition of acetals such as acetal, *tert.*-butylacetal, etc. to the solution, in amounts at least equal to that of the ephedrine.

E. H. SHARPLES.

**Production of nicotine-containing gases from impregnated paper.** BRABENDER ELEKTROMASCHINEN GES.M.B.H. (B.P. 337,311, 3.2.30. Ger., 1.3.29).—Nicotine-containing gases, particularly for combating pests such as the flour moth (*Ephestia kuehniella*, Zeller), are continuously produced by preparing the suitably impregnated paper in the form of a web and positively feeding it between heating units, preferably electric, which are maintained at such a temperature that the nicotine is vaporised without the paper being ignited. Suitable apparatus is described.

E. H. SHARPLES.

**Production of preserved vaccines.** L. DE BLIECK (B.P. 318,585, 29.5.29. Holl., 6.9.28).—Material containing the virus of the diseases known as pox and diphtheria of poultry or other birds is dried *in vacuo*, by means of a hygroscopic substance, until it is readily pulverisable (2–3 days). The cake is then powdered and stored in evacuated containers.

E. H. SHARPLES.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Liquid filters for the preparation of artificial sunlight.** R. LUTHER (Z. wiss. Phot., Schaum-Festschr., 1930, 29, 234–238).—Solutions of copper glycine ( $\text{H}_2\text{O}$ ) are recommended for use as liquid "sunlight" filters. Compared with the copper sulphate-pyridine-mannitol filter, the copper glycine shows a lower extinc-

tion in the ultra-violet, whilst over the visible spectrum the extinction curves are very similar, that of the new filter being displaced about 8–10  $\mu$  towards the red. This can be compensated by lowering the copper sulphate concentration in the second cell. The new filter possesses the advantages that it contains no volatile or easily oxidisable substances, is very stable, does not attack the glass cell, and its extinction curve is very little affected by temperature changes. It is, however, sensitive to acids and alkalis, but this may be remedied by the addition of buffer salts. At room temperature the solubility of the glycine compound is only about 0.03 mol./litre. Experiments on the preparation of pure crystalline copper sulphate, cobalt sulphate, cobalt ammonium sulphate, and copper glycine show that these salts may be obtained with stoichiometrically correct water of crystallisation by final drying over moistened zinc sulphate, potassium chloride, or sodium nitrate.

J. W. GLASSETT.

**Gelatin.** GUILD.—See XV.

See also A., Dec., 1534, **Reflexion and transmission of light by photographic plates** (McRAE and TOLMAN). **Interpretation of solarisation phenomena** (ARENS and KIPPAN).

#### PATENTS.

**Gelatino-silver halide emulsion.** P. C. SEEL, ASSR. to EASTMAN KODAK Co. (U.S.P. 1,757,482, 6.5.30. Appl., 23.2.29).—To minimise static charges during the coating of film, the electrical conductivity of the emulsion is increased by the addition of mono- or polyethylene glycol.

J. W. GLASSETT.

**Anti-abrasion photographic coating.** S. E. SHEPARD and J. G. McNALLY, ASSRS. to EASTMAN KODAK Co. (U.S.P. 1,762,935, 10.6.30. Appl., 19.2.29).—The emulsion layer is surface-coated with a 10% aqueous solution of polyvinyl alcohol, which readily dissolves on application of the developer.

J. W. GLASSETT.

**Photographic roll-film without protective paper strip.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,206, 27.5.29).—Both ends of the film band are coated on the near side with a dye solution which does not transmit actinic rays and is insoluble in the developing bath. (Cf. B.P. 287,124; B., 1928, 914.)

J. W. GLASSETT.

**Sensitive surfaces for producing photographic copies having a charcoal or pastel-like aspect.** F. BUYLE (B.P. 335,357, 9.9.29).—The support is covered with an adhesive not readily soluble in water, upon which a finely-powdered substance, e.g., glass, china clay, chalk, is sprinkled. A sensitive emulsion containing a low concentration of binding medium is then applied so that the sensitive particles are deposited upon the granular material.

J. W. GLASSETT.

**Treatment of exposed chromate-sensitised colloidal photographic layers.** C. ROEHRICH (B.P. 335,128, 11.12.29. Ger., 23.4.29).—The exposed colloid layer is treated with water, but prior to a second uniform exposure the traces of surface moisture are removed by treatment with volatile liquid, e.g., alcohol or ether, which may be added alternatively to the water in the preliminary bath.

J. W. GLASSETT.

**Colour photography.** SPICERS, LTD., and (A) J. N. GOLDSMITH, (B) J. N. GOLDSMITH, T. T. BAKER, and C. BONAMICO (B.P. [A] 334,243, 30.5.29, and [B] 334,265, 1.6. and 1.8.29).—(A) In the preparation of colour screens, the first series of lines are applied with a medium consisting of a fatty resist admixed with a suitable dye and a solvent for the dye, which enables it to penetrate into the film from the resist. By subsequent application of a second dye and removal of the resist the series of lines in alternate colours are left. Suitable media for the first application are given. (B) The penetration of the dye and the bleaching operation are facilitated if the celluloid, cellulose, or cellulose ester film is covered first with a thin layer of cellulose acetate deposited from its solution in carbon tetrachloride, methylated spirit, and acetone. J. W. GLASSETT.

**Photogravures.** L. HUGHES (U.S.P. 1,761,125, 3.6.30. Appl., 17.8.27).—After transference of the carbon tissue to the roller a solution composed of alcohol 1—2 pts., acetone 1 pt., and benzene 8 pts. is applied to the back of the tissue to facilitate the stripping of the support when hot water is subsequently applied. J. W. GLASSETT.

**Reproduction of images on rubber surfaces** (U.S.P. 1,761,424).—See XIV.

## XXII.—EXPLOSIVES; MATCHES.

**Structure of nitrated cellulose. I. Swelling and disintegration of ramie cellulose in nitrating acids.** F. D. MILES and M. MILBOURN. **II. X-Ray examination of nitroramie.** F. D. MILES and J. CRAIK (J. Physical Chem., 1930, 34, 2598—2606, 2607—2620).—I. A series of nitrations aiming at the production of cellulose nitrates containing less than 10.5% N has been carried out and the disintegration of the material which accompanies such preparations has been studied. When the composition of nitrating acids is plotted with the percentages of nitric acid as ordinates and with those of sulphuric acid as abscissæ, there appears an intermediate area in which lie the compositions of all nitrating mixtures which have an appreciable swelling or disintegrating effect. The line representing all mixtures which nitrate to 9% N lies completely within this area, and it is probable that the physical changes which are observed are to be associated with some change in structure of the nitrated fibre which becomes apparent at 7.5% N. Acids containing a high water content have little or no nitrating power, and disintegration still occurs when sulphuric acid is omitted or is replaced by acetic anhydride. An explanation based on varying composition of the acids is not wholly satisfactory.

**II. X-Ray diagrams** have been obtained for nitrated cellulose containing (i) less than 7.5% N and showing unimpaired fibrous structure, (ii) 7.5—10.5% N and showing a damaged fibrous structure, and (iii) more than 10.5% N. The cellulose (ramie) was treated with nitrating acids of varying composition. For (i) and (ii) the data, in general, indicate the presence of mercerised cellulose, but certain peculiarities appear. For the development of the sharp diagram of the fully trinitrated cellulose, nitration in an acid containing at least 45%

HNO<sub>3</sub> is essential. With nitrated ramie containing less nitrogen than that giving rise to the trinitrate diagram, the fundamental structural dimensions appear to vary with the degree of nitration. The results do not support the views of Herzog and Szabó (A., 1928, 48) that such products consist of mixtures of the trinitrate and unaltered cellulose. Assuming Meyer and Mark's view (A., 1928, 621) of cellulose structure, the X-ray diagrams are discussed; in (i) nitration may be intermicellar and in (iii) it is intramicellar with a structure which can expand to accommodate more nitrate groups, the definite trinitrate being reached after 12.7—12.8% N has been gained by nitration. L. S. THEOBALD.

**Sulphuric acid-nitric acid baths.** ALBERTO.—See VII.

## XXIII.—SANITATION; WATER PURIFICATION.

**Solubility of lead in water with reference to the Leipzig water supply.** STRICH (Pharm. Ztg., 1930, 75, 1262—1263).—The solubility of lead was found to be greatest in presence of carbon dioxide and air together, in pipes of small diameter. The carbonate hardness should be kept high, especially when new pipes are brought into use, or where water may remain for many hours in the pipes. S. I. LEVY.

**Volumetric determination of calcium and magnesium in drinking water.** M. E. STAS (Pharm. Weekblad, 1930, 67, 1245—1259).—In applying the method worked out by von Luck and Meyer (B., 1929, 114), the solution is allowed to cool before filtering off the calcium oxalate. In the magnesium determination it is necessary to stir during addition of 8-hydroxyquinoline, and then to warm to 40° to ensure complete precipitation. Iron must be removed before precipitation of magnesium. If bicarbonate is present, the water is first made nearly neutral to methyl-orange by addition of hydrochloric acid. The results obtained with a large number of samples are compared with those found gravimetrically. S. I. LEVY.

**Air-conditioning in ceramics.** MATZEN and PARMELEE.—See VIII. **Cement pipe-lining.** CHAPPELL.—See IX. **Fuel gas by fermentation.** BUSWELL. **Yeast industry.** FREY.—See XVIII.

See also A., Dec., 1541, **Calculation of mineral water analyses** (FRESENIUS and FUCHS). **Analysis of water** (STEINER). 1543, **Determination of boron in natural waters** (WILCOX).

## PATENTS.

**Combating and extermination of vermin and insect pests.** K. DOBMAIER, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,758,936, 20.5.30. Appl., 16.2.28. Ger., 24.2.27).—Water-soluble, molecular additive compounds of furfuraldehyde with basic compounds, such as ethylenediamine, pyridine, acetamide, acetaldehyde-ammonia, aminoethyl alcohol, diglycolimine, etc., and also with inorganic salts such as stannic chloride are used as insecticides. E. H. SHARPLES.

**Treatment of waste sulphide liquors** (B.P. 337,533). **Zeolites** (B.P. 313,522).—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JAN. 30 and FEB. 6, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Atomisation dryers.** O. ZAHN (Chem.-Ztg., 1930, 54, 973—975).—Patents covering dryers of this type are reviewed. The essential points in design are the atomisation of the liquid and the course of the hot air current. In the author's "Ravo-Rapid" system the drying chamber is a vertical cylinder. The liquid is allowed to fall centrally on to a rapidly rotating disc while the main air feed rises in an ascending spiral and thus passes transversely across the course of the droplets. Subsidiary air feeds assist the atomisation, and the dried powder is collected at the bottom of the chamber. It is claimed to yield a very uniform product and to have a high thermal efficiency. C. IRWIN.

**[Automatically controlled thermostat for] regulating high-pressure organic reactions.** P. H. GROGGINS and R. HELLBACH (Chem. & Met. Eng., 1930, 37, 693—694).—A description is given of an apparatus for the experimental ammonolysis of halogenoanthraquinones, using steel bombs in a thermostat, the apparatus being a modification of that described by Phillips (B., 1925, 665). Automatic operating devices for the thermostat are described, by means of which the reaction bombs are removed at the end of a predetermined interval while the thermostat heating elements are shut off. By the use of a special autoclave release, some reaction products while still hot can be blown by their own pressure into a solvent, thus permitting the removal of undesirable by-products of amination. E. A. RYDER.

**Viscosity data in graphical form.** R. P. GENTREAUX (Ind. Eng. Chem., 1930, 22, 1382—1385).—Two graphs are given in which the viscosities of gases and vapours and of liquids are plotted against temperature on log/log paper. The gases for which data are given are oxygen, nitric oxide, carbon dioxide and monoxide, hydrogen chloride, air, nitrogen, sulphur dioxide, methane, steam, ammonia, ethane, benzene, hydrogen, and mixtures of nitrogen and hydrogen in the proportions 9:1 and 3:1. The liquids dealt with include the following classes: acids, chloro-compounds, hydrocarbons, alcohols, esters, as well as water, mercury, and ammonia. Data are given for 33 substances. H. INGLESON.

**Application of nomograms to the calculation of gas quantities.** A. BOEHM (Gas- u. Wasserfach, 1930, 73, 1045—1047).—The volume of gas, reduced to N.T.P., passing an orifice or venturimeter per unit of time, is given by  $V_o = kC\sqrt{h}$ , where  $h$  is the observed pressure difference,  $k$  a constant of the instrument, and  $C$  a function of the density, temperature, pressure, and

degree of saturation of the gas. A nomogram has been constructed from which the required value of  $C$  can be obtained directly. A. B. MANNING.

**Freezing and flow points of glycerin etc.** OLSEN and others.—See III. **Filter cloth.** GRADL. See V. **Helium.** SNYDER and BOTTOMS.—See VII. **Metals for high-temperature service.** KANTER and SPRING.—See X. **Carbon tubes in Cottrell units.** CAMP.—See XI. **[Filters in] pulp and paper mills.** MARX. **Industrial wastes.** KERSHAW. **Wool-scouring effluents.** KING.—See XXIII.

### PATENTS.

**Furnaces.** BABCOCK & WILCOX, LTD. FROM FULLER LEHIGH Co. (B.P. 338,671, 10.10.29).—A form of boiler or other furnace is described in which the ash is retained in a molten condition and tapped off at suitable intervals. B. M. VENABLES.

**Rotary[-hearth] furnace.** M. SKLOVSKY, ASSR. to DEERE & Co. (U.S.P. 1,765,949, 24.6.30. Appl., 10.11.27).—A labyrinthian metallic seal, without sand or water, for a furnace having a rotating hearth is described. B. M. VENABLES.

**Continuous-car tunnel furnace and method of operating the same.** D. P. OGDEN, ASSR. to E. F. PLUMB (U.S.P. 1,768,486, 24.6.30. Appl., 12.11.24).—The kiln is suitable for burning bricks etc. to the following scheme in which a large amount of heat is supplied up to 480°, an oxidising atmosphere is maintained from 480° to 870°, the goods are heated to 1080° and then cooled with recovery of heat. Air enters the kiln at the end at which the goods leave and continues counter-current right through the kiln except that it skips the high-temperature central zone. Combustion furnaces are placed at the sides of the latter zone and the products of combustion eventually mix with the above-mentioned air. The gases are circulated transversely by ceramic fans situated at intervals in suitable side refuges. B. M. VENABLES.

**Heating process [utilising metallic vapours].** J. F. WATT, ASSR. to SUN OIL Co. (U.S.P. 1,760,095, 27.5.30. Appl., 26.7.23).—The vapour of metal from an external boiler is condensed in the lower part of an intermediate exchanger containing liquid metal which is immersed in the body of liquid to be ultimately heated. The metals may be of the same or different composition, a suitable alloy for the intermediate one containing 25—35% Cd and 75—65% Hg. The temperature of the intermediate metal is controlled by adjusting its b.p. by a vacuum, air- and water-cooled condensers being provided to return any vapour produced, and a thermostat

\* The remainder of this set of Abstracts will appear in next week's issue.



placed in the ultimate liquid to control a valve leading to the vacuum pump. Under 28½ in. of vacuum the alloy mentioned will melt at 110–140° and boil at 260–280°.

B. M. VENABLES.

**Heat exchangers.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 338,818, 13.2.30).—In a counter-current heat exchanger it is undesirable for the dividing wall to have good longitudinal conductivity, whilst transverse conductivity is essential. In the construction described the fluid passages are formed through strata which are alternately good and bad conductors.

B. M. VENABLES.

**Composition for preventing dispersion of heat in thermic apparatus.** J. CHATWIN. From SOC. ANON. INVENZIONI GUADAGNIN ("S.A.I.G.") (B.P. 338,743, 13.12.29).—A heat-insulating composition of alkali silicate, asbestos, cork, magnesium carbonate, refractory earth, and hemp is claimed.

B. M. VENABLES.

**Refrigeration.** E. HUSEMANN (B.P. 338,537, 20.8.29).—In order that a cold-storage chamber may be kept in a dry condition it is necessary for frost to form on the ice-box or cooler, which therefore must be below the temperature of melting ice. In this invention a block of ice is supported by a grating below which is a freezing salt, e.g., ammonium chloride, floating on a heavy liquid, e.g., carbon tetrachloride; the salt solution formed, which also floats on the heavy liquid, is drained away from above the grating through cooling coils in the cold-storage chamber.

B. M. VENABLES.

**Dryers and their operation.** A. E. WHITE. From COE MANUFACTURING CO. (B.P. 338,585, 20.8.29).—In order to regulate the humidity conditions of a dryer operating at above 100°, a portion of the air is passed through a cooler at a rate which is determined by a dry-bulb thermostat in the cooled air so that its lower temperature is held constant; the air then passes over a wet-bulb thermostat which regulates the steam sprays etc. in the dryer.

B. M. VENABLES.

**Pulp drying.** E. F. SUNDSTRÖM and K. E. STEEN (B.P. 338,547, 18.7.29).—An apparatus suitable for collecting paper pulp or peat fibre from suspension in water and drying it sufficiently for transport comprises a vessel, maintained under a considerable head of the liquid, of which the bottom is formed of a perforated roller, and the upper part may be maintained full of compressed air to such an extent that the upper part of the roller is exposed to the air which passes through and partly dries the collected pulp. The drying may be completed in another vessel, supplied with warm air, of which the bottom is formed by the sheet of pulp supported on perforated rollers.

B. M. VENABLES.

**Means for dehydration of substances suspended in water.** R. SALWÉN and J. OEHRN, ASSRS. to MORGARDSHAMMARS MEKANISKA VERKSTADS AKTIEBOLAG (U.S.P. 1,768,215, 24.6.30. Appl., 26.1.28. Swed., 18.8.26).—The apparatus comprises a bumping table which, longitudinally in the direction of the bump, slopes first downwards from the feed end and then upwards to the discharge end. The table also has a side slope to discharge water, but the tops of longitudinal riffles are approximately level.

B. M. VENABLES.

**Method for dealing with particles in gaseous suspension.** [Direct formation of printers' ink.] A. L. MILLER and H. J. MASSON (U.S.P. 1,767,089, 24.6.30. Appl., 30.4.25).—Lampblack in process of manufacture is immediately caused to combine with a liquid base in an apparatus comprising a number of rotating apertured discs dipping into the liquid; the products of combustion passing through any disc impinge on the wetted surface of the next one. It is stated that 4000 ft./min. is a suitable speed for the gases passing through the apertures, and if the liquid medium is oil the temperatures of the gases may be 430° at the inlet and 250° at the outlet.

B. M. VENABLES.

**Tube mills.** J. R. KUENEMAN (B.P. 317,769, 21.8.29. U.S., 21.8.28).—A ball mill embodies a cylindrical casing which has helical ribs formed on the lining so that the material and grinding bodies are piled up towards the inlet end. The other end is formed as an outlet grid, and outside it is a cylindrical screen; a conical deflector is interposed to return intermediate-size particles with a spray of water.

B. M. VENABLES.

**Grinding material and extracting volatile matter therefrom.** H. W. MOIR (U.S.P. 1,766,447, 24.6.30. Appl., 20.2.26. Renewed 12.8.27).—A disc grinder has non-fluid (e.g., electric) heaters buried in one or both of the grinding discs.

B. M. VENABLES.

**Screening apparatus.** G. C. OAKES and RANSOMES, SIMS, & JEFFERIES, LTD. (B.P. 338,608, 30.4.30).—The screen surface is composed of bars supported, in two groups of alternate bars, at one end on links or slides giving a substantially straight-line motion, and at the other on cranks at 180° giving a rotary out-of-phase motion. The bars may be formed of stamped and bent steel and may have serrations on their upper edges. The apparatus is suitable for screening cereals etc.

B. M. VENABLES.

**Screening apparatus and the like.** GEN. ELECTRIC CO., LTD., and W. S. EDWARDS (B.P. 338,651, 20.9.29).—A method of tensioning the cloth or wire mesh of a screen by means of wooden clamps and wedges is described.

B. M. VENABLES.

**Separating, disintegrating, and mixing apparatus.** (BARON) E. DE DORLODOT (U.S.P. 1,767,353, 24.6.30. Appl., 13.11.28. Fr., 18.11.27).—The apparatus comprises a screen through which the undersize material is forced by a revolving pneumatic-tyred wheel, giving a rubbing action.

B. M. VENABLES.

**Filter press.** A. F. WENDLER, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,765,389, 24.6.30. Appl., 14.5.26).—In a press of the archimedean type the outlet for solids is through a ring of holes in a split plate which is easily changed so that the apertures may be suited to the material. The outlet for liquid is through a cylindrical filter medium sandwiched between two perforated metallic cylinders, the perforations of which are not in line.

B. M. VENABLES.

**Leaf filters.** E. A. ALLIOTT, and MANLOVE, ALLIOTT & CO., LTD. (B.P. 338,510 and 338,567, [A] 15.8.29 and 28.3.30, [B] 15.8.29).—In (A) a pressure-type leaf filter is described. To open it, one half of the casing is swung on a vertical hinge, and the filtrate header with

attached leaves is drawn forward on rollers. (B) In a filter of the above type, each leaf is provided with two (or more) internal outlet pipes for filtrate extending to different levels; the lower pipes permit free outlet of filtrate, the upper ones outlet of air during filtration and afterwards may be used to allow air to enter so that the filtrate spaces may be entirely drained. The pipes from different levels are connected to different passages in the header.

B. M. VENABLES.

**Filtering apparatus.** J. M. CALLOW, ASSR. to GENTER THICKENER Co. (U.S.P. 1,766,166, 24.6.30. Appl., 31.3.26).—In a filter of the type which is entirely submerged in the prefiltrate and is operated by a wet vacuum pump in the filtrate conduit, a small storage tank is provided for filtrate after leaving the pump, and the pipes are connected to a four-way reversing cock so that the filter medium can be cleaned by a back-flush of filtrate without checking the pump.

B. M. VENABLES.

**Filter.** E. J. SWEETLAND, ASSR. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,768,167, 24.6.30. Appl., 3.6.25).—A form of pressure filter with rotating filter leaves is described. The leaves are detachably mounted on an overhanging portion of the shaft; the casing has one fixed end combined with a bearing for the shaft, and the remainder of the casing is removed as one piece in a horizontal direction when it is desired to open the filter.

B. M. VENABLES.

**Continuous centrifugal separator.** W. C. WALKER (U.S.P. 1,767,905, 24.6.30. Appl., 27.2.26).—The pulp to be separated is applied to the interior of a foraminous belt passing round pulleys at high speed, flanges attached to the edges keep the belt clear of the pulleys, the material is fed by means of a special hollow pulley, and the dry material is discharged by means of a reverse bend.

B. M. VENABLES.

**Hydro-extractor.** F. SCHAUM, ASSR. to FLETCHER WORKS (U.S.P. 1,766,310, 24.6.30. Appl., 29.3.28).—A centrifuge for material that may be out of balance is constructed so that the whole machine, including the motor, is suspended from three slings provided with springs; the basket is attached to the shaft by a ball-and-socket joint so that the axis of the basket need not coincide with the axis of the shaft. The machine is run above the critical speed.

B. M. VENABLES.

**Art of distillation.** H. V. WELCH, ASSR. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,768,179, 24.6.30. Appl., 22.7.25).—In a fractionating tower it is considered that an important function of the bubbling action is to collect mist. In the fractionating condensers described in this patent there is no bubbling, but the condensed fractions are allowed sufficient contact with the vapours to keep them hot, and mist is condensed in the zone where it is formed by arranging the condenser as an electrical precipitator. A tower or a horizontal series of vertical pipe-like condensers may be used.

B. M. VENABLES.

**Apparatus for controlling the density or viscosity of liquids.** C. O. LAVETT (U.S.P. 1,765,433, 24.6.30. Appl., 7.1.28).—The liquid is circulated by a pump through a device, *e.g.*, an evaporator, which causes an increase of viscosity. In the outlet conduit of the pump

is placed a paddle device which is rotated by power and tends to drag round a false wall of the paddle chamber; when the drag reaches a certain amount a diverting valve snaps over and the liquid is delivered to another place instead of back to the evaporator.

B. M. VENABLES.

**Producing non-turbulent circulation of liquids.** J. F. WAIT, ASSR. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,765,386, 24.6.30. Appl., 17.1.25).—A propeller is situated in the lower part of a tank which is so shaped and provided with guides as to produce an even circulation with a minimum of turbulence.

B. M. VENABLES.

**Emulsification apparatus.** G. C. HURRELL, and ROAD DEVELOPMENT Co., LTD. (B.P. 338,496, 13.8.29).—The constituents to be emulsified, *e.g.*, bitumen and an aqueous solution of a protective colloid, are fed by separate pumps to the emulsifier; one pump is driven direct from the emulsifier and the other from the first through variable gearing.

B. M. VENABLES.

**Aeration and gas-purification apparatus.** G. E. SEIL, ASSR. to KOPPERS Co. (U.S.P. 1,755,614, 22.4.30. Appl., 9.4.25).—An apparatus for aerating a liquid, which may contain finely-divided solids in suspension, comprises a frame, preferably cylindrical in shape, to which is attached an envelope of pliable porous material, *e.g.*, a woven linen or cotton fabric, and within which is a perforated air-supply pipe. The porous material is so supported that it can vibrate as the air is pumped through it into the liquid, so that any solid matter which lodges thereon is automatically detached. The apparatus is particularly suitable for the aeration of gas-purification liquids, and for the flotation of the precipitated sulphur thereon and its removal as foam.

A. B. MANNING.

**Treatment of flue and like gases with the object of removing therefrom objectionable solid or gaseous products. Apparatus for washing flue gases.** P. J. ROBINSON (B.P. 338,492 and 338,527, [A, B] 18.5. and 11.7.29).—(A) The gases are dry-cleaned in a centrifugal separator to such an extent as to leave a modicum of fine dust in them, and are then treated with finely-divided water sprays or steam which, in conjunction with the dust as nuclei, produces a fog favourable to the oxidation of sulphur dioxide. The fog is removed by passing the gases tangentially into the base of a tower over curved surfaces which are wetted by coarse water sprays from the top of the tower. In (B) are described forms of construction of the tower utilising alloy- or lead-lined steel, with expansion joints for the tower and flues.

B. M. VENABLES.

**Separation of constituents from gas or vapour mixtures.** N. CARO and A. R. FRANK (B.P. 338,497, 13.8.29).—By the use of a condenser having cooling surfaces which are maintained considerably cooler than the outgoing gases it is possible to obtain a good recovery of a condensable constituent while permitting the remaining gases to leave unsaturated with the vapour of that constituent, *i.e.*, at a higher temperature than would be permissible in an ordinary type of cooler. The process may be applied to the preparation of nitrogen tetroxide or nitric acid by the catalytic oxidation of

ammonia or hydrocyanic acid, in which case a contact mass, a boiler to supply steam, and a condenser are arranged one behind the other. B. M. VENABLES.

**Separation of gaseous mixtures.** STANDARD OIL DEVELOPMENT Co., Assecs. of W. K. LEWIS (B.P. 338,719, 11.11.29. U.S., 12.11.28).—Gases, *e.g.*, methane and hydrogen, are separated by preferential absorption in charcoal or other absorbent while under pressure. After collecting the unabsorbed hydrogen and giving a displacement wash with previously obtained methane, the pressure is reduced and/or the temperature increased to recover the methane. B. M. VENABLES.

**Liquid seals for gas chambers.** LODGE-COTTRELL, LTD., and L. LODGE (B.P. 338,827, 4.3.30).—A bell forming, with an oil bath, a seal for an electrostatic separator is provided with an in- and up-turned lower edge which prevents any condensed moisture from spreading over the surface of the oil, it being conveyed below the surface and allowed to fall to the bottom of the bath, whence it is withdrawn at intervals. As an additional precaution, cleaned gas is injected into the bell through a hollow inner wall of the oil-trough.

B. M. VENABLES.

**[Reversing valve for] regenerative furnaces.** O. REINER (B.P. 338,788, 3.1.30. Ger., 10.1.29).

**Brickwork of retort settings, furnaces, and the like.** SOUTH METROPOLITAN GAS Co., and W. T. SEACH (B.P. 339,268, 3.9.29).

**Compression refrigerating machines.** BRIT. THOMSON-HOUSTON Co., LTD., Assecs. of C. STEENSTRUP (B.P. 339,519, 30.12.29. U.S., 29.12.28).

**Compression refrigerating machinery.** J. BARR (B.P. 339,272, 28.5.29).

**Heating of absorption refrigerating apparatus.** ELECTROLUX, LTD. FROM PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 339,042, 5.10.29).

**Refrigeration and apparatus connected therewith.** G. MAIURI and R. F. BOSSINI (B.P. 338,935, 27.7.29. Fr., 31.5.29).

**Refrigerators. Heat-insulating containers and their production.** COCA-COLA Co., Assecs. of J. C. STATON (B.P. 338,959—60, 29.8.29. U.S., 9.1.29).

**Thermostatic control in the heating of liquids [by electricity].** JACKSON ELECTRIC STOVE Co., LTD., and F. SMITH (B.P. 338,597, 23.8.29).

**Removing carbon deposits from engine cylinders** (B.P. 335,963).—See II. **Fire-extinguishing preparations** (B.P. 319,372).—See VII. **Rotary kiln** (U.S.P. 1,766,453).—See IX. **High temperatures** (B.P. 338,468). **Treatment of air blast for furnaces** (B.P. 338,244). **Non-ferrous alloy [for condenser tubes, evaporators, etc.]** (B.P. 338,676).—See X. **Precipitation of particles from gases** (B.P. 337,389, 337,642, 337,685, 338,739, and 338,796).—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Supervision of coal purity.** H. KOELSCH (Gas- u. Wasserfach, 1930, 73, 1047—1050).—The water and ash contents of coal deliveries should be constantly checked, although for this purpose high accuracy is

unnecessary. The procedure in use at the Munich gas works is as follows: the coal sample (2 kg.) is weighed out on to metal tray, heated for 2 hrs. in a vacuum oven, allowed to cool in the air for  $\frac{1}{2}$  hr., and again weighed; the loss of weight gives the moisture content. The dry coal is ground in a ball mill and a part is transferred to a bottle for the subsequent determination of ash by incineration in a muffle furnace. Care is taken that the coal does not re-absorb an appreciable amount of moisture from the air while being weighed out for incineration. The cost of this sampling and analysis at Munich during 1929 amounted to 0.12 RM. per ton of coal delivered. Some of the results are illustrated graphically. A. B. MANNING.

**Effects of prolonged weathering on the friabilities of certain coals.** J. H. H. NICOLLS and E. SWARTZMAN (Canada Dept. Mines, Invest. Fuels, 1930, No. 712, 27—28; cf. B., 1926, 697).—Four samples of Canadian domestic coal and one of a Scottish semi-anthracite have been subjected to 3 years' exposure in an open shed, screening and tumbler tests being carried out at intervals. None of the five samples underwent any very great disintegration during that period. Prolonged storage did not affect the friability of the Scottish semi-anthracite, but increased that of the Canadian coals.

C. B. MARSON.

**Effect of the rate of heating on the properties of [Japanese] coke.** T. SHIMMURA (J. Fuel Soc. Japan, 1930, 9, 97—100; cf. B., 1929, 461).—The degree of swelling has been measured by heating samples of coal at rates varying between  $1^{\circ}$  and  $10^{\circ}$  per min. It is shown that the faster the rate of heating, the greater is the degree of swelling. Micro-examination of the cokes, carbonised at  $900^{\circ}$  in a laboratory apparatus, indicated that the slower the rate of heating the more uniform was the cell structure of the coke. The amount of oil which liquated from the coal at the temperature of its softening point, at various time intervals, was determined, and it is concluded that liquation phenomena are partly responsible for the difference in the degree of swelling, according to the rate of heating.

C. B. MARSON.

**Slacking of coal and its proper interpretation.** S. W. PARR and D. R. MITCHELL (Ind. Eng. Chem., 1930, 22, 1211—1212).—Slacking (or disintegration) tests on coal are carried out by bringing the coal into moisture equilibrium with the air, drying for 23 hrs., submerging in water for 1 hr., drying and submerging again, and repeating this routine through 8 cycles; after each drying the amount passing a  $\frac{1}{4}$ -in. sieve is weighed and the cumulative percentage after 8 cycles is taken as the slacking index. It has been suggested that this index may be used as an indication of rank, beginning with the lignites and extending to the truly bituminous coals. Slacking, as related to bituminous coals, is directly dependent on the thickness, distribution, and porosity of fusain bands rather than on the absorptive capacity of the coal proper as in the case of lignites. It is concluded that whilst the slacking test, as a qualitative test for lignite, has value, it is inapplicable to bituminous coals as an indication of their rank.

C. B. MARSON.

**Determination of moisture in solid fuels.** M. DOICH (Brennstoff-Chem., 1930, 11, 429—432; cf. B., 1930, 172).—The values of the moisture content of solid fuels obtained by the usual methods, *e.g.*, by determination of the loss of weight on heating at 105°, or by distillation with xylene, are, in general, too low. An exact and rapid method of making the determination consists in refluxing the fuel (5—20 g.) with alcohol (100 g.) for 5 min., cooling, filtering, and determining the temperature of miscibility of the filtrate (20 c.c.) with an equal volume of light petroleum. The temperature of miscibility of the light petroleum with alcohol having already been determined as a function of the water content of the alcohol, the moisture content of the fuel is then readily calculated. A. B. MANNING.

**Development of Dakota lignite. I. Aqueous tension of the moisture in lignite.** I. LAVINE and A. W. GAUGER. **II. Sorption of water vapour by lignite, peat, and wood.** M. LARIAN, I. LAVINE, C. A. MANN, and A. W. GAUGER (Ind. Eng. Chem., 1930, 22, 1226—1231, 1231—1234).—I. Investigations relating to the adsorption of water vapour by solid adsorbents are reviewed. The desorption and adsorption of water vapour by three different lignites from North Dakota are studied. It is shown that the vapour pressure over the lignite decreases as the moisture content is reduced, and depends on whether the lignite is being dehydrated or hydrated. The reduction in vapour pressure during either dehydration or hydration varies only slightly with the different Dakota lignites. The effect of temperature on dehydration is studied. Calculation of the radii of the capillaries shows a variation of from  $56.73 \times 10^{-7}$  cm. for nearly saturated lignite to  $0.3 \times 10^{-7}$  cm. for the dry lignite. The average value for the latent heat of vaporisation at 20—40° is found by calculation to be 609.1 g.-cal. per g.

II. Studies of the adsorption and desorption of water by North Dakota lignite, Minnesota peat, and birch wood show the characteristics of the two processes to be the same for the three materials. With each material the curve showing the relation between vapour pressure and moisture content due to adsorption is displaced from the corresponding curve obtained by desorption of water vapour. The pore radii of the materials are calculated by means of the Thompson equation, and the following classification is obtained in order of decreasing pore size: peat, birch wood, lignite, brown coal.

H. E. BLAYDEN.

**Development of Dakota lignite. III. Drying of lignite without disintegration.** I. LAVINE, A. W. GAUGER, and C. A. MANN (Ind. Eng. Chem., 1930, 22, 1347—1360; cf. preceding abstract).—Samples of lignite were dried by the Fleissner process, in which the material is brought in direct contact with saturated steam, the steam pressure gradually increased to 13 atm., after which it is gradually diminished to atmospheric, when dry air is blown through the autoclave containing the coal. Under the optimum conditions of treatment the moisture content was reduced from 36 to 16%, whereby the calorific value correspondingly increased. Modified shatter and tumbler tests of the dried lignite showed that it was able to withstand general conditions of handling without excessive

breakage, that it had good weathering properties, and that steam-drying did not increase the tendency to spontaneous combustion.

C. B. MARSON.

**Under-water storage of Saskatchewan lignite.** J. H. NICOLLS and E. SWARTZMAN (Canada Dept. Mines, Invest. Fuels, 1930, No. 712, 25—26).—Preliminary tests on the storage of lignite under water are described. It is shown that 3—4 months' storage has little effect on the fuel ratio (*i.e.*, the ratio of fixed carbon to volatile matter) or the calorific value, but removes small quantities of mineral matter.

C. B. MARSON.

**Preliminary carbonisation and briquetting tests on lignite from Northern Ontario.** R. A. STRONG (Canada Dept. Mines, Invest. Fuels, 1930, No. 712, 7—12).—Low-temperature carbonisation tests carried out on a Northern Ontario lignite show that a char is obtained which can be briquetted satisfactorily. Although the yields of by-products are low, sufficient gas is produced to effect carbonisation. It is estimated that  $2\frac{1}{2}$  tons of the lignite will be required per ton of briquettes.

C. B. MARSON.

**Avoidance of the effect of alkalis during the preparation and purification of humic acid.** K. SIMON (Z. Pflanz. Düng., 1930, 18A, 323—336).—Methods are described for the separation of humic acid based on extraction by 1% sodium fluoride or oxalate solutions and subsequent precipitation with weak acid or neutral salt solutions. The preparation of a water-soluble humic acid is described.

A. G. POLLARD.

**Open-hearth combustion.** W. P. CHANDLER, JUN. (Proc. Eng. Soc. W. Pa., 1930, 46, 242—255).—The important factors in maintaining proper combustion in, and the economical operation of, an open-hearth steel furnace are described. The use of liquid fuels, producer gas, natural gas, coke-oven gas, and mixtures of the last-named with blast-furnace gas is discussed. In open-hearth furnace practice only the heat developed above the temperature of the materials in the bath is available for absorption, and as high flame temperatures give high rates of heat transfer and consequent shortening in the time of the process it is essential that the flame temperature be maintained as high as possible consistent with furnace life. High flame temperatures are obtained by high preheating of the air and in some cases of the fuel gas, by proper air regulation, intimate mixing of the air and fuel, and the correct design of furnace and ports.

C. B. MARSON.

**[Carbonisation in] vertical retorts.** H. B. KENDRICK (Gas J., 1930, 192, 796—798).—Decomposition of gaseous hydrocarbons by cracking in the hottest parts of vertical retorts has been followed by taking gas samples from the coal core and from the completely carbonised zone. Possible means of reducing losses of this kind are illustrated by gas analyses, which show the effect of breaking up the coal core, and of withdrawing the carbonisation products rapidly. Two tests were carried out with different sizes of the same coal; when using large pieces it was possible to work the retorts under a slightly diminished pressure, with reasonably low off-take temperature and small leakage

loss, but with small coal none of these conditions could be attained.

R. H. GRIFFITH.

**Relation between oven width, coking time, and [working] capacity.** T. KURODA (J. Soc. Chem. Ind., Japan, 1930, 33, 485—486B).—Coking times for Japanese coal in Solvay ovens of 370, 400, and 420 mm. width were measured and compared with that taken when ovens of the standard width (470 mm.) were employed. It was concluded that, taking charging and discharging time into consideration, the 400-mm. oven had the greatest output capacity.

C. IRWIN.

**Influence of the inorganic constituents in the carbonisation and gasification of coal: the liberation of ammonia.** Institution Gas Research Fellowship Report, 1929—30. A. H. EASTWOOD and J. W. COBB (Gas J., 1930, 192, 745—746).—Additions of lime, calcium carbonate, and iron oxide were made to a fusible coal containing 1% of ash, each mixture containing 5% (calc. as oxide) of one of the three compounds; a mixture containing 5% of crushed "pure" coke was also investigated. The "pure" coal and mixtures were carbonised in atmospheres of nitrogen, hydrogen, and nitrogen-steam mixtures, the total time of carbonisation being 9 hrs. and the temperature being advanced in stages of 100° from 200° to 1000°. The ammonia was collected and determined at each stage, and after the cokes had been analysed nitrogen balance sheets were made up. The pure coal experiments showed that (a) ammonia evolution commenced at 300° and reached a maximum at 700°; (b) carbonisation up to 1000° in a nitrogen atmosphere yielded 17% of the nitrogen of the coal as ammonia and substitution of hydrogen for the nitrogen doubled the yield; (c) the use of a 60% of steam-40% of nitrogen atmosphere gave a yield of ammonia equivalent to 25% of the nitrogen; (d) in no case was all the nitrogen liberated obtained as ammonia. It is concluded that ammonia is formed by (i) the decomposition of compounds, probably ulmic, under the influence of heat, which commences at 300° and gives its maximum yield at 700°; hydrogen and steam do not appear to affect it apart from exercising a protective action on the ammonia formed; and (ii) the action of hydrogen on some nitrogen compound in the coke; this reaction does not occur at or below 500°, and nitrogen thus gasified is all liberated as ammonia, although some of this ammonia may be afterwards lost by thermal decomposition. The results of experiments in which oxides were added showed that (a) with all mixtures ammonia evolution again commenced at 300° and reached a maximum in all cases (except iron oxide coal in nitrogen) at 700°; (b) in a nitrogen atmosphere, the addition of lime or calcium carbonate raised the ammonia obtained up to 1000° from 17 to 22%, whilst iron oxide decreased the yield to 15%, there being a marked diminution from 700°; (c) in a hydrogen atmosphere, the yield of 33% was raised to 37% by the addition of calcium carbonate; (d) in a mixture of 60% of steam and 40% of nitrogen, lime raised the yield from 25 to 34% and calcium carbonate to 32%. It is concluded that the effects of the inorganic constituents are due to an action leading to an increased formation of ammonia, the actual yield

being greater in all cases up to 600°, and to an action above 600° leading to an increased loss of ammonia by thermal decomposition.

C. B. MARSON.

**Use of wood waste for heating and generation of power.** O. WANS (Inst. Chem. Eng., Dec., 1930, 49—55).—The net calorific value of the gas produced in a generator fed with wood is about 155 B.Th.U., the methane content being higher than with anthracite. Saw-mill refuse contains 20—40% of moisture, and freshly cut wood 50—60%. Not more than 45% is desirable in a gas generator. The gas from a generator using soft wood is corrosive. A suitable generator, rotary tar extractor fed with water, and sawdust scrubber are described. Wrought and cast iron are more resistant to acetic acid than is mild steel. Stainless steel is suitable for the extractor impeller wheel. The effluent is usually neutralised with limestone. The consumption of wood waste per b.h.p.-hr. is about 2.25 lb. If the gas is used for heating, a temperature of 1000° can be obtained. The dust collector and generator are provided with water seals as a safeguard against small explosions in starting up. Generators may be either of the up- or down-draught type. The maximum size is about 8 ft. 6 in. grate diam.

C. IRWIN.

**Distillation of wood waste and utilisation of the products.** M. SCHOFIELD (Inst. Chem. Eng., Dec., 1930, 30—41).—Apart from distillation methods, a recent process is the manufacture of "synthetic" boards. Large forest waste is usually carbonised in steel lattice cars four or more of which form a charge for a standard oven. The American Badger-Stafford retort handles pieces below 8 in. in length. Sawdust and the like are much more difficult to handle. They are to-day being distilled successfully in rotary retorts containing loose steel breakers, or in rectangular retorts fitted with scrapers. A flow sheet is given for a works producing brown and grey acetate of lime, wood tar and oils, wood spirit, and "methyl acetone." The last-named is a mixture of methyl alcohol and acetone, and is used as a solvent. The applications of the different products are discussed. It is stated that a successful process has been developed in which comminuted wood impregnated with calcium acetate is carbonised, the yield of acetone being much increased, whilst the charcoal is obtained as high-grade activated carbon.

C. IRWIN.

**Production of carbon black from propane.** W. B. PLUMMER and T. P. KELLER (Ind. Eng. Chem., 1930, 22, 1209—1211).—The effect of various factors on the production of carbon black from propane, in an apparatus similar to that of the commercial channel process, has been studied. It is shown that carbon black equal in quality to that made from methane may be made from propane; that the function of the channels is mainly a cooling effect; and that the type of carbon black produced is not appreciably affected by the atmosphere surrounding the flames, *i.e.*, the percentage excess air for similar temperature conditions. It is concluded that the type of black is determined by conditions within the flame itself, *e.g.*, by such factors as size, shape, temperatures of the gaseous layers and luminous particles, etc.

C. B. MARSON.

**From coal to gas. II.** F. SCHUSTER (Gas-u. Wasser-fach, 1930, 73, 1033—1038; cf. *ibid.*, 1929, 72, 713).—Diagrams have been constructed illustrating the heat distribution for the production of mixed gas (a) from coal gas and "double gas," the latter being a mixture of coal gas and water-gas produced in a suitable generator; (b) from coal gas and producer gas; and (c) by the steaming of gas retorts. The calculations are given in full.

A. B. MANNING.

**The back-run process for the manufacture of carburetted water-gas. I.** Rept. 27 of Joint Res. Comm., Inst. Gas Eng. and Univ. Leeds. F. J. DENT, W. H. BLACKBURN, N. H. WILLIAMS, and E. PARRISH (Gas J., 1930, 192, 704—711).—The test, which was carried out on a Humphreys & Glasgow carburetted water-gas set at the Stratford works of the Gas Light & Coke Co., was of 96 hrs.' duration, and the results obtained, including carbon and heat balances, are given in detail. The coke used, made from a Durham coal in horizontal retorts, contained 4% of moisture and 9% of ash, and the oil, which was an American petroleum distillate, was used at the rate of 2.1 gals. per 100 cub. ft. The coke consumption was 34.08 lb. per 1000 cub. ft. of carburetted water-gas and the steam decomposition in the fuel bed was 68%. The efficiency of blue water-gas production, taking into account the fuel required for extra steam to the generator and for the steam to auxiliary plant, was 49.3%, whilst if these items were omitted it was 55%. The efficiency of oil cracking in the carburettor was 1.21 therms of oil gas per gal. of oil. The thermal efficiencies of the production of carburetted water-gas, no account being taken of the potential heat in the ashes, clinker, and "fliers," was 69.3%.

C. B. MARSON.

**Humidity effects in the iron oxide process for removal of hydrogen sulphide from [coal] gas.** C. G. MILBOURNE and W. J. HUFF (Ind. Eng. Chem., 1930, 22, 1213—1224).—The object of the investigation was to determine the correct humidity conditions for maintaining the optimum moisture content for sulphiding, revivifying, and simultaneous sulphiding and revivifying. A laboratory method was devised for sulphiding iron oxide under varying conditions of temperature, humidity, and surface. It is shown that the optimum humidity for sulphiding is about 65% at 38°, that revivification is very much slower than sulphiding, and that the optimum relative humidity for this stage is very high. Purification procedure involving simultaneous revivification *in situ* requires constant high-humidity conditions, but the use of humidities approaching saturation is inadvisable if conditions permit of the free deposition of liquid, as this deposition greatly lowers the efficiency of the purification process. The laboratory tests and conclusions were confirmed by large-scale experiments. It is suggested that the use of automatic mechanical means for controlling the temperature and humidity of the gas is desirable.

C. B. MARSON.

**The Feld processes for extraction of ammonia and hydrogen sulphide from coal gas. Report III of the Ammonia Sub-Committee.** A. PARKER (Gas J., 1930, 192, 734—736).—The two known processes

(developed by Feld) for the simultaneous extraction of ammonia and hydrogen sulphide from coal gas with consequent production of ammonium sulphate were examined. The development of troublesome side-reactions caused difficulties, and the processes are considered too complex for satisfactory gasworks' operation. One of the primary considerations in a comparison of existing practice with processes similar to those of Feld is the relative cost of oxidation of hydrogen sulphide to produce sulphate, and in this respect the Feld processes do not show any indication of offering advantages.

C. B. MARSON.

**By-product ammonia [from gasworks]. Report III of the Ammonia Sub-Committee.** H. HOLLINGS and E. W. SMITH (Gas J., 1930, 192, 739—742).—A review of the present status of by-product ammonia is given. The use of ammonia in gasworks as an agent for the removal of hydrogen sulphide from the crude gas, and the Burkheiser, Feld, Koppers C.A.S., and Cobb's zinc processes are all rejected as means for improving the economic position of ammonia in the gas industry. Investigations are being made in the fixation of ammonia as phosphate, but this method has not reached the stage at which it offers a solution of the problem. The application of the direct and semi-direct processes to works carbonising more than 300 tons of coal daily is worthy of further examination; where effluent troubles are serious, the cost of a more efficient extraction of tar from the hot gases, which would be necessary in either case, should not be charged against ammonia recovery, and if the direct process were adopted the recovery of phenols from the subsequent condensates of ammonia-free liquor might be made to pay. Savings may be effected by eliminating the use of water in the ammonia washers and by avoiding the dilution of liquor caused by inefficient decomposition of steam admitted to the retorts. Centralised sulphate plants in some districts, for the local production of concentrated liquor, might be economically sound, and consideration should be given to the possibility of making the gas industry self-supporting in respect of sulphuric acid.

C. B. MARSON.

**[Disposal of liquor effluents from gasworks.] 5th Rept. of Liquor Effluents Sub-Comm., Inst. Gas Eng. and Univ. Leeds** (Gas J., 1930, 192, 730—733; cf. B., 1930, 174).—Experiments made with a Lodge-Cottrell electrostatic tar precipitator are described. The removal of heavy tar before condensation of the liquor effected a marked reduction in the higher tar acid content of the liquor, whilst its monohydric phenol content was increased; monohydric phenols, however, are more readily removed by solvents, evaporation, or bacterial treatment than are the higher tar acids, and do not produce the intense reddish-brown colours normally characteristic of vertical and low-temperature liquors. Distillation of the modified liquor yielded an effluent which had a comparatively low higher tar acid content; the "devil" liquor, with a phenol content of 1.12 g. per 100 c.c., was satisfactorily disposed of by evaporation on the producer bars of a vertical retort setting. A system of condensation at the Coventry Gas Works, consisting of a liquor-circulation system and a Colman cyclone tar extractor was investigated,



and it was found that the installation of a tar and liquor separator at the condensers gave ammonia and spent liquors considerably lower in their higher tar acid contents. The biological purification of effluent liquors, carried out on the Foleshill bacteria beds at Coventry, was further examined, and the results suggest that this method is technically effective but at a heavy monetary expenditure. Laboratory experiments have been made on the removal of phenol and higher tar acids by solvents; using benzol, the cresols were more readily removed than phenol, but pyrocatechol, as representative of dihydric phenols, was removed only to a small extent; with a 4:1 mixture of benzol and aniline oil 50% removal of higher tar acids was obtained compared with 8% with benzol alone. C. B. MARSON.

**Products of combustion from typical gas appliances.** V. Rept. 27 of Joint Res. Comm., Inst. Gas Eng. and Univ. Leeds. J. W. Wood (Gas J., 1930, 192, 714—716; cf. B., 1929, 743).—Further examinations of the combustion products from typical gas-burning appliances, with special reference to carbon monoxide, have been made. The small amounts of carbon monoxide were determined by the iodine pentoxide method as hitherto. A surface-combustion water heater, consuming 100 cub. ft. of gas per hr. and heating 40 gals. of water per hr. through 100° F. when supplied with 10—15% of excess air, produced 0.05 pt. per 10,000 in the exit gases from the appliance; with less than 5% of excess air, a condition not likely to occur in practice owing to the characteristic appearance of the combustor surface, the production of carbon monoxide rose rapidly. The carbon monoxide productions from a Fairweather recording gas calorimeter and an inverted burner without a mantle were negligible. An inverted burner fitted with a bare mantle and with normal aeration gave, over the normal working range, 20—70 pts. and a similar burner and mantle, with an open vitreous globe, gave 18—50 pts. CO per 10,000 of gas burned. It is concluded that (1) monoxide production is lowest over that range of gas rate at which the burner will be normally used; (2) the gas rate, and hence the total amount of monoxide produced per burner per hr., is small, especially when compared with typical room capacities and ventilation; (3) the products of combustion will in every case be greatly diluted before reaching breathing level and the monoxide concentration in practical operation will be harmless; (4) from the viewpoint of economy, efficiency, and hygiene, it is not desirable to operate an incandescence burner at a rate much below its normal working rate; where a small light is required smaller units should be installed. C. B. MARSON.

**Phenols of brown-coal tar.** F. VON HESSERT (Z. angew. Chem., 1930, 43, 771—774).—The fraction, b.p. 90—160°/12 mm., of a brown-coal tar product consisting principally of tar acids was purified by dissolving in alkali, passing air through the warm solution in order to oxidise the less stable constituents, and then acidifying and redistilling the recovered phenols under reduced pressure. The product was stable to air, and could be distilled to 260° under the ordinary pressure without decomposition. The phenols in this

oil were identified by conversion into the corresponding hydroxycarboxylic acids. For this purpose the oil was fractionated, and the individual fractions were converted into the corresponding potassium aryl sulphates by treating the concentrated alkaline solution with potassium pyrosulphate (cf. Baumann, A., 1878, 148); the esters were oxidised by alkaline permanganate, and the hydroxy-acids were recovered after acidifying the product with hydrochloric acid (cf. Heymann and Koenigs, A., 1886, 542). The acids were separated and identified. The brown-coal tar product examined contained all the phenols (*o*-, *m*-, and *p*-cresol, *m*- and *p*-ethylphenol, and all the xlenols) which had previously been found in bituminous coal tar. The high proportion of ethylphenol is noteworthy. No tri-alkylphenols were found. Naphthols were present in the higher fractions (260—290°). A. B. MANNING.

**High-pressure hydrogenation of neutral and phenolic oil in low-temperature tar.** S. TASHIRO and S. ANDO (J. Fuel Soc. Japan, 1930, 9, 102—104; cf. Tashiro, B., 1928, 776).—Hydrogenation experiments, using the phenolic portion of low-temperature tar, are described and the influences of the catalysts, catalyst poisons, and temperature on the end-product are discussed. C. B. MARSON.

**Extraction of bases from the aqueous distillate of primary tar.** A. TRAVERS and FRANQUIN (Compt. rend., 1930, 191, 951—952).—The aqueous solution is made alkaline with 30% sodium hydroxide solution, the mixture distilled, the bases are extracted from the distillate by repeated extraction with ether, the ethereal solution is evaporated at 0°, and the resulting aqueous solution (water is dissolved by the ether) concentrated and freed from traces of ammonia by passing carbon dioxide-free air through it at 0°. The bases are then liberated by the addition of 40% sodium hydroxide solution. The method is satisfactory when the concentration of bases is 2.5%. H. BURTON.

**Determination of paraffin in asphalts.** W. J. PIOTROWSKI and H. BURSTIN (Przemysl Chem., 1930, 14, 503—509).—A comparison of a number of methods shows that the most consistent results are given by that of Engler and Holde, which is adopted as the standard method for Poland. R. TRUSZKOWSKI.

**Oil shale from Pictou County, Nova Scotia.** A. A. SWINNERTON (Canada Dept. Mines, Invest. Fuels, 1930, No. 712, 13—24).—Channel samples from 5 outcrop beds have been examined. Oil yields varying between 6 and 26 gals. per ton were obtained; it is concluded that three of the beds are worthy of further study. C. B. MARSON.

**Cracking value of straight-run and cycle gas oil.** H. SYDNOR and A. C. PATTERSON (Ind. Eng. Chem., 1930, 22, 1237—1240).—The cracking value of cycle gas oil is investigated by passing it several times, without recycling, through a small-scale cracking unit. The combined results of four successive passes are compared with a recycling operation on the same equipment, the gasoline yield being the same in each process. With the same operating conditions, in successive passes through the apparatus the yield of gasoline, calculated on the



feed stock to each pass, decreases; the ratio of fuel oil yield to gasoline yield increases; the boiling range of the cycle gas oil is lowered; and the sp. gr. (Amer. Petroleum Inst. units) corresponding to a given boiling range is lowered. During the operations a decided change in chemical composition of the cycle gas oils occurs, as indicated by the decrease in the aniline point. It is concluded that the cracking value of cycle gas oils in terms of the products that can be derived from them decreases with successive passes through the apparatus. The same results are obtained either by recycling the fractions of cycle gas oil or by cracking them separately under the same operating conditions.

H. E. BLAYDEN.

**Motor spirits containing alcohol.** VII.—IX. W. SWIENTOSLAWSKI (Przemysl Chem., 1930, 14, 482—485, 497—501, 501—503).—VII. [With S. BAKOWSKI.] The volatility of various motor spirits containing alcohol is a function of their vapour pressure, and the curves obtained are of the same type as for pure petrol.

VIII. [With J. PFANHAUSER and S. BAKOWSKI.] The critical temperature of mutual solubility of the constituents of a number of motor fuels varies from  $-18^{\circ}$  for a mixture containing 50% of 94° alcohol, 30% of petrol, 20% of benzene, and 3% of ether to  $-75^{\circ}$  for a mixture containing 50% of absolute alcohol, 40% of petrol, and 10% of benzene. The greatest readiness to separate into layers on addition of water is found for a mixture containing only alcohol and petrol, and the least for mixtures containing oleic acid and solvent naphtha.

IX. [With B. KARPIŃSKI.] The flash and inflammation points of a number of mixtures vary from  $-11^{\circ}$  to  $-32^{\circ}$ , and from  $-4^{\circ}$  to  $-30^{\circ}$ , respectively, for mixtures containing 86% of 85° alcohol, 10% of petrol, 2% of benzene, and 2% of ether, and 50% of 92° alcohol, 20% of gasoline, and 20% of benzene.

R. TRUSZKOWSKI.

**Determination of the carbon residue from petroleum products.** (Carbon residue by Conradson's method.) B. ROSENBAUM (Chem.-Ztg., 1930, 54, 857—858).—The carbon residue from 17 motor lubricating oils determined by Conradson's method (A.S.T.M. Serial Designation D.189—27, or I.P.T. Serial Designation G.O.9) varied from 0.106% (Gargoyle Arctic) to 2.37% (Gargoyle B). The ash content of the oils averaged 0.09%. In general the carbon residue increased with increasing viscosity of the oil. The agreement between repeat determinations was satisfactory, the deviations from the mean varying from 0.3% to 6% with different oils.

A. B. MANNING.

**"Sulphuric acid test" of motor benzol.** F. KNÖTTNER (Brennstoff-Chem., 1930, 11, 432—435).—The sulphuric acid test (cf. Kaudela, B., 1929, 1004) is unreliable as a criterion of the suitability of a benzol as a motor spirit and should be replaced by an evaporation test. The latter is preferably carried out by evaporating 200 c.c. of the benzol in a current of air; the benzol is contained in a 250-c.c. wide-mouthed flask, provided with air inlet and outlet tubes, and is gently heated. The air is passed at such a rate that the bulk of the benzol is evaporated in 2—3 hrs., and the process

is continued until the residue is of constant weight. It is suggested that under these conditions the residue from a suitable motor spirit should not exceed 10 mg. The results of examining a number of motor spirits by both methods are tabulated.

A. B. MANNING.

**Divergence of the viscosity and flash point of oil mixtures [from the arithmetic mean].** E. KADMER (Chem.-Ztg., 1930, 54, 871).—The viscosity and the flash point of a mixture of two oils can be calculated from the formulae:  $V = \{aA + bB - v(A - B)\}/100$  and  $F = \{aA + bB - f(A - B)\}/100$ , where  $a$  and  $b$  are the percentages of each oil in the mixture,  $A$  and  $B$  are their respective viscosities or flash points, and  $v$  and  $f$  are quantities which have been determined experimentally. The values of  $v$  and  $f$  are tabulated as functions of  $a$  (or  $b$ ).

A. B. MANNING.

**Method for sweating of paraffin [wax].** ANON. (Petroleum, 1930, 26, 1217—1220).—Molten crude paraffin is carried continuously against a stream of cold water and cold air, the solidified material is then fractionally melted by suitable re-heating in such a way that different grades can be collected separately. The construction of the system, which can be operated at different speeds of raw material and heating or cooling media to give any desired product, is fully described and illustrated.

R. H. GRIFFITH.

**Viscosity data.** GENEREAX.—See I. **Emulsifying agents.** BENNETT.—See III. **Pulp industry waste.** GRIFFITH.—See V. **Ammonium sulphate.** PARRISH.—See VII. **Refractories in vertical retorts.** GREEN.—See VIII. **Fuel economy in ironworks.** BAINBRIDGE.—See X. **Carbon tubes in Cottrell units.** CAMP.—See XI. **Industrial wastes.** KERSHAW. **Wool-washing effluent.** SMITH.—See XXIII.

#### PATENTS.

**Coking retort ovens.** J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,755,382 and 1,756,235, [A] 22.4.30, [B] 29.4.30. Appl., [A] 16.5.21, [B] 23.2.23).—(A) The heating walls of the oven are provided with horizontal combustion flues. Below the coking chambers and heating walls and parallel thereto are regenerators disposed into two series located on opposite sides of the battery respectively, one series operating for inflow and the other for outflow. The regenerators of each series are grouped into pairs, one inflow-operating regenerator of each pair supplying gas and the other air. The corresponding pairs of regenerators of the two series communicate with opposite ends of the horizontal flues of the same heating wall by means of substantially straight vertical ducts. The setting may be heated by means of an extraneously produced gas, e.g., producer gas, or by means of coke-oven gas. In the latter case the gas is supplied through special ducts, and some of the regenerators may then be used for conveying a neutral gaseous diluent to lengthen the flames in the flues. (B) The heating walls of the oven are provided with vertical combustion flues which are connected together in sets by a number of horizontal flues lying at the top of the vertical flues. Each of the horizontal flues is connected with a corresponding flue in an adjacent heating wall. A pair of end-to-end positioned regenerators below each heating

wall are directly connected with its combustion flues, whereby the alternate regenerators of the battery may be operated for flow in one direction while the intermediate regenerators are operated for flow in the opposite direction. Gas-supply channels extend horizontally in each pillar wall below the corresponding heating wall, and communicate with the individual flues by means of short vertical ducts. A. B. MANNING.

**Low-temperature carbonisation of fuels and apparatus therefor.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,740, 30.9.29. Addn. to B.P. 301,975; B., 1929, 119).—A uniform movement of the material being carbonised as described in the prior patent is assured by the provision of impact devices in the carbonisation vessel, arranged at a suitable height above the bed of fuel in motion. The devices are preferably of louvre pattern and are constructed of sheet metal or other refractory material. A. B. MANNING.

**Apparatus for low-temperature distillation of pulverulent or dusty carbonaceous materials.** I. G. FARBENIND. A.-G. (B.P. 309,501, 11.4.29. Ger., 11.4.28).—Liquid carbonaceous materials are sprayed into the retort at the point where the initial materials are fed in and where simultaneously the gases have their outlet, so that the initial materials are moistened and at the same time all solid constituents are precipitated from the effluent gases. A. B. MANNING.

**Apparatus for calcining seaweed.** J. C. MORRISON (B.P. 336,500, 16.12.29).—The seaweed is spread through a cutting machine and thence through a drying chamber to a furnace which is utilised to generate the steam for heating the drying chamber. The latter consists of a long tunnel extending along one side, one end, and partly along a third side of the building, and encloses three endless conveyors in series. The seaweed is spread evenly over the conveyors by means of rotating distributors. The steam-pipes which heat the drying chamber are mounted just above the conveyors. The drying chamber is surrounded by a jacket for receiving and condensing the steam rising from the seaweed through openings in the roof of the chamber. A. B. MANNING.

**Removing carbon deposits from engine cylinders.** A. E. WHITE. From THOMAS & HOCHWALT LABORATORIES, INC. (B.P. 335,963, 5.6.29).—A composition containing a halogenated aromatic hydrocarbon is used. A suitable composition is formed by dissolving monochloronaphthalene in a mixture of benzol and ethyl alcohol. A. B. MANNING.

**Final cooling of gases from coal-distillation ovens and the like.** H. KOPPERS A.-G. (B.P. 336,484, 27.11.29. Ger., 27.9.29).—In order to avoid difficulties due to the separation of naphthalene during the final cooling of coal-distillation gases, the circulating cooling water is subjected to a tar washing before it is re-cooled. The tar washer, in which the tar and water are passed counter-current to one another, is preferably arranged below the gas-cooling appliance and within the same vertical tower. A. B. MANNING.

**Production of a high-grade lump coke from coal.** A. POTT (B.P. 335,910, 27.4.29).—The coal is dried and heated to just below the decomposition temperature in

one apparatus, *e.g.*, an externally heated rotary retort, and is then transferred to another apparatus wherein it is distilled at a temperature not above 550°.

A. B. MANNING.

**Pitch-coking process and product.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,756,234, 29.4.30. Appl., 16.10.19. Renewed 21.3.24).—The pitch is mixed with a sufficient amount of bauxite to prevent foaming and is then coked. A product of higher carbon content may be produced by mixing some of the coke so formed with a further quantity of pitch, and coking the mixture. The products are suitable for the manufacture of electrodes. A. B. MANNING.

**Manufacture of carbon black, acetylene, and hydrogen.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,261, 10.7.29).—Hydrocarbons, or gas mixtures containing hydrocarbons, *e.g.*, methane or natural gas, are passed through an electric arc at about 2500°, under such conditions that the ratio of the amount of gas, measured in cub. m., which is passed hourly through the arc, to the power of the arc, measured in kw., lies below 0.6. A. B. MANNING.

**Destructive hydrogenation of coal and other carbonaceous materials.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,543, 24.6.29).—Coal, lignite, tars, phenols, or other oxygen-containing carbonaceous materials are treated with carbon monoxide, or gases containing carbon monoxide, under pressure and at a raised temperature, and the substantially oxygen-free product is then hydrogenated by treatment with hydrogen under pressure in known manner. Both stages of the process may be carried out in the presence of suitable catalysts. A. B. MANNING.

**Destructive hydrogenation of coal extracts.** A. POTT and H. BROCHE (B.P. 309,446, 8.4.29. Ger., 10.4.28).—Extracts obtained by treating coal with a suitable solvent, *e.g.*, a mixture of tetralin and low-temperature tar phenols (cf. B.P. 293,808; B., 1930, 314), are subjected to a preliminary hydrogenation under pressure at moderately raised temperatures in the presence of hydrogenating catalysts, and the middle fraction of the product, b.p. 200–350°, is then destructively hydrogenated, preferably in the vapour phase, in the presence of a heavy-metal halide, *e.g.*, manganous chloride, or other suitable catalyst. A. B. MANNING.

A. B. MANNING.

**[Gas] producers.** C. H. LANDER and T. F. HURLEY (B.P. 336,022, 19.7.29).—The opposite walls of a producer, which is preferably rectangular in cross-section, are formed of banks of vertical water tubes forming part of a boiler or other water-heating system. The tubes are arranged to permit the passage of gas between them, and means are provided for passing air or air and steam through one of the walls, and for leading away the gaseous products through the other. The spaces between the walls and the outer casing may be divided into compartments with independent offtakes, whereby the air and steam may be supplied at different rates at different levels of the chamber. A. B. MANNING.

**Gas producers.** F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (B.P. 336,335, 1.8.29).—A

producer with a cylindrical casing has a rectangular grate comprising a number of bars which are pivotally mounted in such a way that they can be oscillated in unison. The upper surface of each bar is curved, and relatively deep slots spaced at short distances extend inwards from the opposite edges of the curved face. The bars have spaces therein for holding water and/or for the passage of steam, which issues around the bars through suitably placed nozzles and passes up into the producer.

A. B. MANNING.

**Method of operating a water-gas generator.** H. A. BRASSERT & Co., and WESTERN GAS CONSTRUCTION Co., Assees. of W. B. CHAPMAN, C. W. ANDREWS, and H. B. YOUNG (B.P. 336,203, 7.6.29. U.S., 27.11.28).—The generator is provided with a device for continuously removing ash and clinker; this preferably takes the form of a water-cooled beam which is situated just above a stationary grate, and which revolves slowly about a vertical axis. The beam is so shaped as to produce an up- and out-stroking effect. The relative proportions of air, down-stream, and up-stream are so regulated as to maintain a fuel bed of substantially constant density, within which is a zone of sufficiently high temperature to at least partly liquefy the ash and a lower cooler zone in which the ash tends to re-solidify. The ash-removing device operates in the lower zone.

A. B. MANNING.

**Prevention of gum formation in gas-distribution systems.** HUMPHREYS & GLASGOW, LTD., Assees. of W. H. FULWEILER (B.P. 336,456, 4.11.29. U.S., 17.12.28).—Small quantities of mono- or poly-hydroxybenzene derivatives, *e.g.*, cresol, quinol, added to the gas prevent the formation of gums due to the oxidation or polymerisation of indene, styrene, or other unsaturated hydrocarbons.

A. B. MANNING.

**Apparatus for modifying the calorific power of gases produced from fuel.** F. RIPPL (B.P. 314,863, 24.6.29. Ger., 3.7.28).—Solid fuel is distilled in an externally heated retort, which is divided into two compartments by a partition in which are gas passages; one compartment contains the charge which is being distilled, and the other contains incandescent coke from the preceding distillation. The distillation gases from the former compartment pass through the hot coke in the latter, and thence through one or more gas-education channels to the main.

A. B. MANNING.

**Production of mixed oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of J. A. PERRY (B.P. 336,525, 31.1.30. U.S., 2.3.29).—The apparatus comprises a generator, carburettor, and superheater, and the mode of operation includes the following steps. The fuel bed is air-blasted and the blast gases are burned in the carburettor and superheater; a supply of fluid hydrocarbon, *e.g.*, refinery oil gas, is introduced into the generator, and simultaneously another supply of fluid hydrocarbon, *e.g.*, gas oil, preferably together with some steam, is introduced into the carburettor; the former supply is passed through the fuel bed, wherein it is cracked, and the resultant gases are led through an offtake to storage, while the latter supply is passed through the carburettor and superheater and the resultant lightly cracked oil gas is also led to storage;

the fuel bed is steamed, the excess carbon deposited therein being thereby converted into water-gas, which may be carburetted if desired. The three gases so produced are mixed.

A. B. MANNING.

**Manufacture of hydrogen and gases containing hydrogen.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,635, 8.4.29).—Gaseous hydrocarbons, *e.g.*, methane or gas mixtures containing methane, such as the waste gases from the synthesis of ammonia or from the destructive hydrogenation of carbonaceous materials, admixed with steam, and if desired with air, are passed into the charge in a water-gas producer in which coke is undergoing gasification. [Stat. ref.]

A. B. MANNING.

**Purification of coke-oven gas with a view to the separation of constituents thereof by low-temperature cooling.** GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 316,948, 6.8.29. Ger., 6.8.28).—The gas, freed as far as possible from the higher hydrocarbons, hydrogen sulphide, carbon dioxide, and water, is passed over an adsorbing agent, *e.g.*, activated carbon, which adsorbs both nitrogen oxides and the reactive hydrocarbons, and acts catalytically to cause the interaction of both groups of substances in the adsorption layer. The adsorbing agent is preferably impregnated with metal salts, particularly of iron. The exhausted agent is regenerated by heating in a current of dry nitrogen.

A. B. MANNING.

**Purification of gases containing hydrogen sulphide and ammonia.** F. BÖNNEMANN (B.P. 336,425, 12.10.29).—Gases containing ammonia and hydrogen sulphide in which the proportion of the latter is greater than that represented by the ratio  $2\text{NH}_3:1\text{H}_2\text{S}$  are divided into two portions of which one contains only as much hydrogen sulphide as can be combined with the whole of the available ammonia. The ammonia is then concentrated into this portion of the gas, from which the ammonia and hydrogen sulphide are eliminated together by scrubbing with a thionate solution. The other portion of the gas may be treated separately for the removal of the hydrogen sulphide therefrom, or may be utilised for some purpose for which the removal of sulphur compounds is unnecessary.

**Purification of gases by washing with organic liquids.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,319, 20.7.29. Cf. B.P. 329,688; B., 1930, 853).—Gases are freed from volatile sulphur compounds, and to a large extent from carbon dioxide if this is present in appreciable amounts, by washing with hydrocarbons of high b.p., or with phenols, under pressures of at least 10 atm., and at such a temperature and for such a period of time that the concentration of hydrogen sulphide in the gas is reduced to less than 0.3%.

A. B. MANNING.

**Manufacture of bituminous emulsions.** J. S. MILLER, JUN., Assr. to BARBER ASPHALT Co. (U.S.P. 1,752,449, 1.4.30. Appl., 17.7.20).—A non-fluid bituminous base is ground in a ball or other suitable mill in the presence of water containing a protective colloid, *e.g.*, rosin-soda soap, to a degree of fineness at which it will emulsively combine with the water in a solid state without the aid of heat, and the grinding is continued

in the presence of a finely-divided inorganic emulsifying agent until a homogeneous emulsion is obtained.

H. S. GARLICK.

**Treatment of petroleum oil.** A. G. BOGARDUS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,753,769, 8.4.30. Appl., 30.1.25. Renewed 2.7.28).—Oil under superatmospheric pressure is passed continuously through heating tubes disposed within a furnace, and is discharged into one of a series of interconnected zones of reaction where precipitation of carbon takes place, and from which the separated gases are removed for dephlegmation, condensation, and collection. Discharge of heated oil into this reaction zone is interrupted and diverted to another of the interconnected zones. The superatmospheric pressure of the first zone is gradually reduced and simultaneously imposed upon the zone into which the discharge of heated oil has been diverted. Leakage of heated oil into the zone of reduced pressure is prevented by maintaining a neutral zone of oil in the vapour connexion between the zones of reaction at a temperature materially below that of the oil being treated, and under substantially the same pressure as that on the system.

H. S. GARLICK.

**Conversion of heavy hydrocarbon oils into lighter products.** F. S. WORDICH (U.S.P. 1,754,136, 8.4.30. Appl., 8.7.24).—Charging oil is heated under pressure in a pipe still to a temperature at which no substantial decomposition takes place, and delivered under reduced pressure into the median portion of a reaction tower where oil and vapour are separated, by causing the stream of heated oil to divide into two portions which flow annularly in opposite directions, and are subjected to successive impacts and deflexions, and, while still in rapid movement, make contact with each other. The heavy residual oil is cracked by causing it to flow over the surfaces of baths of liquid metals heated to a temperature sufficient to produce the Leydenfrost phenomenon, whereby vapour-phase cracking is effected, and then downwardly passing the remaining liquid over surfaces heated to a temperature sufficient to cause liquid-phase cracking of the oil. The cracked vapours are introduced near the bottom of the reaction tower and ascend, making intimate contact with the liquid descending from the upper part of the tower, whilst the residual unvaporised oil is collected in a pool at the bottom of the converter. The mixed vapours are subjected to rectification and dephlegmation in the upper part of the tower and then to fractional condensation, whereby finished products are obtained. Uncondensed gases are separated from the gasoline and scrubbed in countercurrent flow with the gasoline before being reintroduced under pressure into the heavy residual oil undergoing cracking.

H. S. GARLICK.

**Continuously hydrolysing acid sludge [from the refining of petroleum oil].** R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, ASSRS. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,752,555, 1.4.30. Appl., 15.11.26).—A heated mixture of acid sludge and water is continuously passed into a chamber under sufficient pressure to maintain the hydrolysing temperature, and the volumes of produced acid and tar are so regulated that the acid is subjected to hydrolysis for sufficient

time to complete the reaction and entirely separate the tar, whilst the tar is subjected to hydrolysing temperature for a relatively short time which is insufficient to produce coking. The clean acid and liquid tar are continuously withdrawn from the reaction chamber.

H. S. GARLICK.

**[Stabilisation of mineral or vegetable] oil compositions etc.** W. W. EVANS, ASSR. to R. T. VANDERBILT Co., INC. (U.S.P. 1,752,945—6, 1.4.30. Appl., [A] 23.9.27, [B] 26.12.28).—Oxidation or decomposition of mineral oils and greases is prevented, and rancidity of animal and vegetable oils and fats inhibited, by adding less than 1% of a diarylalkylenediamine, *e.g.*, diphenyl- or di-*p*-tolyl-ethylenediamine.

H. S. GARLICK.

**Immersion wax.** F. W. SULLIVAN, JUN., ASSR. to STANDARD OIL Co. (U.S.P. 1,752,933, 1.4.30. Appl., 17.2.28).—The rate of development of acidity of a paraffin wax immersion bath is considerably diminished by the addition of 0.05—0.5% of an aromatic compound such as  $\beta$ -naphthol, pyrogallol, diphenylamine, gallic acid, quinol, and *p*-phenylenediamine.

H. S. GARLICK.

**Washer boxes for coal and the like.** B. NORTON (B.P. 339,376, 16.9.29).

**Straightening the walls of the chambers of ovens or retorts for producing gas and coke.** STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER (B.P. 339,578, 26.3.30. Ger., 16.7.29).

**Doors of gas-making retorts.** R. & J. DEMPSTER, LTD., and C. E. HOLT (B.P. 339,557, 19.2.30).

**Burners particularly for furnaces fired with pulverised fuel.** C. CHAPMAN & Co., LTD., and W. A. WOODSON (B.P. 339,010, 12.9.29).

**Liquid fuel, gas, and like burners.** T. B. COLLINS (B.P. 339,190—2, 5.2.30).

**Pulp drying (B.P. 338,547).** Particles in gaseous suspension (U.S.P. 1,767,089). Distillation (U.S.P. 1,768,179). Emulsifier (B.P. 338,496). Gas-purification apparatus (U.S.P. 1,755,614). Separation of gaseous mixtures (B.P. 338,719).—See I. Artificial silk from peat (B.P. 337,360).—See V. Sulphuric acid from oil treatment (U.S.P. 1,764,210). Ammonium sulphate (B.P. 338,373).—See VII. Bituminous emulsions (B.P. 319,663 and 335,668). Non-slippery road surfaces (B.P. 337,928).—See IX. Collecting electrode (B.P. 337,389).—See XI. Heat-treated fatty oils (B.P. 339,048). Lubricant (U.S.P. 1,752,309).—See XII.

### III.—ORGANIC INTERMEDIATES.

**Accurate evaluation of acetic anhydride.** A. ROTT (Chem.-Ztg., 1930, 54, 954—955).—The method depends on boiling a known weight of the anhydride with a definite weight of water and deducing the water content of the resultant mixture by determination of the f.p. The difference between the water found after boiling and that added corresponds to the anhydride content. The anhydride (10 c.c.) is weighed in a stoppered

flask, 1—2 c.c. of water (6—7% excess over the theoretical quantity) are added, and the mixture is again weighed, the weight of water added being deduced. The mixture is heated under reflux for 1 hr. and cooled. The f.p. of the mixture is then determined and the water content corresponding to this f.p. is found from tables. The amount of water thus found present is subtracted from the amount of added water to give the weight of water used in the reaction (1 g. of water  $\equiv$  5.67 g. of anhydride). Examples are given. Impurities such as traces of hydrochloric acid, acetyl chloride, and higher homologous acids do not markedly affect the determination; in each case the f.p. is lowered, thus giving a smaller anhydride content. A table of f.p. of the most usual range is appended. H. E. BLAYDEN.

**Freezing and flow points for glycerin, prestone [ethylene glycol], denatured alcohol, and methyl alcohol.** J. C. OLSEN, A. S. BRUNJES, and J. W. OLSEN (Ind. Eng. Chem., 1930, 22, 1315—1317).—Commercial samples of the above liquids, as sold for use as antifreeze materials, were tested in a range of concentrations in water and their freezing and flow points are recorded. The flow point was found by observing the temperature at which the liquids flowed readily after having melted. H. INGLESON.

**Commercial emulsifying agents.** H. BENNETT (Ind. Eng. Chem., 1930, 22, 1255—1256).—The use of trihydroxyethylamine stearate and ammonium linoleate as emulsifying agents is discussed, and details are given for the preparation of emulsions of mineral and vegetable oils and of various waxes in water. The use of these emulsifying agents in accelerating many synthetic reactions by increase in the surface of the emulsified substance is pointed out. H. INGLESON.

**Physical characteristics and commercial possibilities of chlorinated diphenyl.** C. H. PENNING (Ind. Eng. Chem., 1930, 22, 1180—1182).—Chlorination of diphenyl produces first a mixture of the 2- and 4-chloro-derivatives (cf. Jenkins and co-workers, B., 1930, 182), which is a liquid of marked solvent properties and has practically the viscosity of water. Continued chlorination gives a range of products varying in consistency from a light mobile oil, through a thick syrupy stage, to a solid resinous or crystalline state. The oils are non-drying, whilst the resins are apparently permanently thermoplastic. The physical properties of a range of these materials (which are sold under the trade name of "Aroclors") are tabulated. "Aroclors" find commercial application in the protective coating, water-proofing, flameproofing, moulding, electrical insulation, and adhesive industries, etc. S. S. WOOLF.

**Control of high-pressure reactions.** GROGGINS and HELLBACH. **Viscosity data.** GENEREUX.—See I. **Products from wood distillation.** SCHOFIELD. **Carbon black from propane.** PLUMMER and KELLER. **Gasworks' effluent.** INST. GAS ENG. **Phenols of brown-coal tar.** VON HESSERT. **Hydrogenation of phenolic oil.** TASHIRO and ANDO. **Bases from tar distillate.** TRAVERS and FRANQUIN.—See II. **Pulp industry by-products.** GRIFFITH.—See V. **Tannery waste.** LLOYD.—See XV. **Tetranitromethane.** STETTBACHER.—See XXII.

## PATENTS.

**Manufacture of polymerisation products of diolefines.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,339, 2.8.29).—In order to facilitate removal of the product the polymerisation vessel is lined with paper (e.g., a paper bag), cardboard, or low-melting alloy (e.g., Wood's metal), or a coating of water-soluble material (sodium silicate) is applied. C. HOLLINS.

**Catalytic alkylation of hydrocarbons, alcohols, organic acids, and amines.** RHEINISCHE KAMFFER FABR. G.M.B.H. (B.P. 319,205, 8.8.29. Ger., 17.9.28).—Olefines, or alcohols or ethers which generate them, are heated with the hydrocarbon, alcohol, organic acid, ammonia, or amine to be alkylated or aralkylated, in presence of a catalyst consisting of activated hydrosilicates or hydrated silicic acids obtained by the action of mineral acid on hydrosilicates. Examples are: naphthalene and 20% of tonsil heated at 230° with ethylene under 20—40 atm. give ethylnaphthalenes, b.p. 250—260°; cyclohexanol, benzene, and tonsil at 200° give cyclohexylbenzene, b.p. 238°; benzyl alcohol, benzene, and tonsil at 230° give diphenylmethane, m.p. 26—27°, b.p. 264°, and dibenzylbenzenes, m.p. 80.5—81.5°; ethyl alcohol vapour led over tonsil at 150—200° gives ethyl ether; benzoic acid, tonsil, and propylene at 230° and 50—60 atm. give isopropyl benzoate, b.p. 218°; aniline, methyl alcohol, and tonsil are heated at 230° for 12 hrs., water is removed, and the residue heated with more methyl alcohol for 12 hrs., to give dimethylaniline. C. HOLLINS.

**Manufacture of 4 : 10-diaminoperylene.** F. BENSA (B.P. 336,144, 25.11.29. Austr., 22.12.28).—4 : 10-Dinitroperylene is not reducible by alkaline hyposulphite, but gives the diamine when treated in alcohol suspension with sodium hydroxide and sodium sulphide or sodium hydrogen sulphide. C. HOLLINS.

**Acetylene** (B.P. 336,261). **Hydrocarbons from heavy oils.** (U.S.P. 1,752,692).—See II. **Fatty acid esters** (B.P. 336,276).—See XII. **Aliphatic acids by fermentation** (B.P. 337,153).—See XVIII. **Organo-aluminium compounds** (B.P. 336,922).—See XX.

## IV.—DYESTUFFS.

**Preparation of 1-chloro-5-aminoanthraquinone and 1-amino-5-[p]-tolylaminoanthraquinone.** T. MAKI and Y. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 464—466B).—Potassium anthraquinone-1-sulphonate (40 g.) with sulphuric and nitric acids gives 5- and 8-nitroanthraquinone-1-sulphonic acids (28 and 14 g., respectively; cf. Schmidt, A., 1904, i, 256; Bayer & Co., B., 1906, 369). The 5-isomeride with hydrochloric acid and sodium chlorate in aqueous solution gives 1-chloro-5-nitroanthraquinone. This is reduced by sodium sulphide to the amine, which is a yellow-orange dye with good affinity for acetate silk. 1-Chloro-5-aminoanthraquinone heated with *p*-toluidine and sodium acetate gives 1-amino-5-*p*-tolylaminoanthraquinone, which dyes acetate silk violet-red, and after sulphonation is a fuchsin-red acid dye. L. J. HOOLEY.

**The indanthrone [alkali] melt.** III. **Effect of phenol.** IV. **Effect of temperature in presence of**

**phenol.** T. MAKI (J. Soc. Chem. Ind., Japan, 1930, 33, 456—460 B, 461—463 B).—III. In the production of indanthrone by fusion of 2-aminoanthraquinone (1 pt.) with potassium hydroxide (4 pts.) at 180° for 15 min., 4.2 pts. is an optimum quantity of phenol, the yield being 45.4% as against 18.2% without phenol. Increasing the fusion time to 1 hr. gives 51.5% and to 6 hrs. 50.5% of indanthrone. Curves are traced for the yields of alkali-soluble matter, nitrobenzene-soluble matter, and indanthrone.

IV. Similar curves are traced showing the yields of the three products with (a) varying temperature, using 5-min. heating and the optimum proportions as described above, whereby a new optimum is formed at 210° with a yield of 49.5%; and (b) varying time with the new optimum of 210°, the yield being 50.5% after 5 min., 51.5% after 15 min., and then falling.

L. J. HOOLEY.

## PATENTS.

**Manufacture of fast sulphur dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,061, 2.9.29).—The 6-arylamino-2-*p*-hydroxyarylamino-naphthalenes obtainable by condensing a 6-arylamino- $\beta$ -naphthol with a *p*-aminophenol are sulphurised, e.g., with alcoholic sodium polysulphide, to give bluish-green sulphide dyes. Examples are 6-anilino-2-*p*-hydroxyanilino- and 2-*p*-hydroxyanilino-6-*p*-anisidinonaphthalenes.

C. HOLLINS.

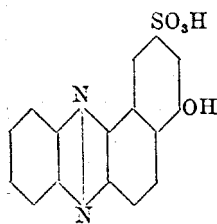
**Manufacture of vat dyes of the anthanthrone series.** L. CASELLA & Co., G.M.B.H. (B.P. 336,495, 9.12.29. Ger., 10.12.28. Addn. to B.P. 260,998; B., 1928, 225).—Anthranthrone is halogenated (e.g., brominated) in aqueous suspension, preferably in presence of a carrier (iodine, ferric chloride) and/or an acid-binding agent.

C. HOLLINS.

**Manufacture of [acid] azo dyes.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 336,350, 15.8.29).—*m*-Tolidine (2 : 2'-dimethylbenzidine) is tetrastotised and coupled with 1 mol. of salicylic acid and 1 mol. of  $\beta$ -naphthol, a  $\beta$ -naphtholmonosulphonic acid, Brönner acid, or methyl- $\beta$ -naphthylamine-7-sulphonic acid for orange acid dyes fast to milling.

C. HOLLINS.

**Manufacture of [azo] dyes [of the azine series].** W. W. GROVES. From Soc. Chem. Ind. in Basle (B.P. 335,896, 25.6.29).—Hydroxysulphonaphthaphenazines are coupled with diazo components. Examples are : 4-hydroxy-2-sulpho- $\alpha$ -naphthaphenazine (annexed formula) with diazotised *o*-anisidine (brown on wool), *p*-nitroaniline (red on wool), sulphanilic acid (red-brown), 4-nitro-*o*-aminophenol-6-sulphonic acid (chrome dark green on wool; coppered, dark violet on wool; after-coppered on cotton, violet; prechromed, green-grey on cotton; precoppered, violet on cotton); pretreated with nickel sulphate, green-violet on cotton); 4-hydroxy-2-sulpho-10-methyl- $\alpha$ -naphthaphenazine with diazotised anthranilic acid (bordeaux-red on wool, brown after-chromed; chromed on cotton, brown; precoppered, violet on cotton); 4 : 10-dihydroxy-2 : 12-



cotton; pretreated with nickel sulphate, green-violet on cotton); 4-hydroxy-2-sulpho-10-methyl- $\alpha$ -naphthaphenazine with diazotised anthranilic acid (bordeaux-red on wool, brown after-chromed; chromed on cotton, brown; precoppered, violet on cotton); 4 : 10-dihydroxy-2 : 12-

disulpho- $\alpha\alpha'$ -dinaphthazine with diazotised *p*-nitroaniline *o*-sulphonic acid (red-brown on wool, red-violet on cotton), 4-nitro-*o*-aminophenol-6-sulphonic acid (green-grey on cotton, reddish-grey after-coppered; black-green on wool, grey after-coppered; pre-coppered, grey on cotton; prechromed, green-grey on cotton, green on wool). The last-mentioned azine may be reduced to hydroazine and coupled on the fibre with diazotised 4-chloro-2-aminodiphenyl ether for olive-brown.

C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Hibiscus fibres from India and Iraq.** ANON. (Bull. Imp. Inst., 1930, 28, 284—289).—Analysis of the fibre (9.6% of moisture) from *H. Sabdariffa*, var. *alissima*, grown in India gave the following percentage results (on moisture-free material): ash 0.6, loss on  $\alpha$ -hydrolysis 10.7, loss on  $\beta$ -hydrolysis 13.7, loss on water-washing 0.9, cellulose 75.6%. The strength was comparable with that of jute, but the fibre was harsher and rather coarser. Five samples of *H. cannabinus* fibres from Iraq were also examined; four were of Indian Deccan hemp (cut before, at the beginning, and at the time of flowering, and at the period of seed formation) and one was of local Jiljil (cut at flowering time). After washing with hot water and air-drying, they gave the following respective analyses (%): moisture 10.1, 13.1, 13.7, 13.0, 11.3; (the following are expressed on moisture-free fibre) ash 1.9, 2.2, 1.1, 1.0, 2.4; loss on  $\alpha$ -hydrolysis 10.7, 8.4, 7.5, 10.2, 12.1; loss on  $\beta$ -hydrolysis 13.2, 12.4, 11.4, 14.6, 15.8; loss on water-washing 1.3, 0.9, 0.1, 1.5, 1.5; cellulose 73.7, 75.8, 76.5, 75.0, 73.2.

E. H. SHARPLES.

**Microscopic structural units of wood fibres.** G. J. RITTER and R. M. SEBORG (Ind. Eng. Chem., 1930, 22, 1329).—Delignified white spruce fibres are treated with phosphoric acid (78%) at 75° on glass slides, and on the application of slight pressure to the cover glass over the specimen the spindle-shaped or "fusiform" bodies, which have hitherto been regarded as the smallest building units visible under the microscope, are resolved into smaller units. When detached these are spherical, but a study of their optical properties and general considerations lead to the belief that this is not their original shape (cf. B., 1929, 390).

C. JEPSON.

**Filter cloth from nitrocellulose.** H. GRADL (Chem.-Ztg., 1930, 54, 871—872).—The nitrogen content of the various textile fibres, wool, cotton, silk, artificial silk, etc., can be regarded as a measure of their resistance to chemical action. The difficulties of weaving nitrocellulose fibres have been overcome and the fabric so produced is particularly suitable for filtering strongly acid solutions. It is much more resistant to the action of acid than a cotton fabric which has been nitrated after weaving.

A. B. MANNING.

**Utilisation of industrial by-products [of the American pulp industry].** R. W. GRIFFITH (Inst. Chem. Eng., Dec., 1930, 42—44).—Sulphite liquor has found some use, when concentrated, as a cheap adhesive, as a filler in leather, etc. When chestnut wood is

treated by the soda process the tannin must be recovered before digestion; the only by-product of the actual process, recovered in small amount, is methyl alcohol. The residual carbon of the black ash derived from the lignin is used as fuel or refined into decolorising carbon. The volatile products of the digestion of pine wood with sulphate liquor consist of turpentine, methyl sulphide, and pine oil. The second of these products is employed as a warning agent for gas leaks. Sodium resinate is separated from the recovered sulphate liquor. The problem of the economical recovering of tannin from the bark of trees used for pulp-making has not so far been solved. C. IRWIN.

**Testing of wood pulp for strength.** K. G. JONAS (Papier-Fabr., 1930, 28, 800—805).—It is considered essential that the experimental beater used for pulp-strength determinations should follow closely the action of the hollander type used in mill practice, the pressure should be capable of accurate variation, and beating should be according to time and beating degree jointly, as beating to one of these factors alone gives inconsistent results. An apparatus is described, consisting of 6 cylindrical boxes mounted on a disc capable of rotation. Each box is fitted with a miniature hollander roll, and the whole is fitted with a cover. Weighed amounts of pulp are placed in each box with the requisite quantity of water, the cover is placed in position, and the whole apparatus rotated at a constant speed. After each period at which it is required to obtain the strength of the pulp one box is removed, replaced by an empty box of equal weight, and the process continued until the contents of all 6 boxes have been beaten. The sheet-making machine is described, which follows more or less conventional lines. Details of the degree of reproducibility and of the conditions to be observed for different types of pulp are given. T. T. PORTS.

**Viscose. XXXV. Effect of chlorination on the properties of the pulp.** J. MURAKAMI (J. Cellulose Inst., Tokyo, 1930, 6, 223—225).—In recent work by Mazuda and Suzuka (B., 1930, 759) it was found that chlorination of the pulp followed by treatment with an alkali solution caused a decrease of copper number, whilst the viscosity of the material in cuprammonium remained practically constant. Somewhat different results are now obtained, and it is found that the copper number of the standard pulp always increases with increasing time of chlorination, whilst the viscosity decreases, at first considerably, and then slowly, independent of the time of treatment with 1% sodium hydroxide solution, although the copper number of purified pulp decreases with increasing time of treatment with alkali. The copper number of sulphite pulp diminishes after such treatments, as stated in the previous work, but a definite decrease in viscosity with time of chlorination is observed. B. P. RIDGE.

**Viscose. XXXVI. Spinning experiments with viscose by a two-bath process using sulphuric acid of the ordinary concentration, and of that employed in the Lilienfeld process.** S. IWASAKI, F. FUJITA, and S. REE (J. Cellulose Inst., Tokyo, 1930, 6, 237—240).—Viscose threads were spun into the ordinary bath of dilute sulphuric acid and then passed

through a second bath of the concentrated acid. The effects of spinning velocity, length and concentration of the second bath, and the properties of the viscose were examined. The threads obtained in this way are not so strong as those given by the Lilienfeld process, but, in general, they are stronger than the ordinary threads and of normal extensibility. B. P. RIDGE.

**Action of alkali on cellulose. III. Solubility of artificial silk in caustic soda solution.** K. ATSUKI and K. SHIMOYAMA (J. Cellulose Inst., Tokyo, 1930, 6, 219—233).—Samples of air-dried viscose rayon were immersed for 4 hrs. in solutions of sodium hydroxide of various concentrations at different temperatures, and, after filtration, the dissolved cellulose was determined by oxidation with potassium dichromate, using a modification of the method of Bray and Andrews (B., 1923, 545 A; see also B., 1929, 89). The solubility increases with decreasing temperature, and under the conditions employed reaches a maximum at 0°. It is also highly dependent on the concentration of the sodium hydroxide solution, and is a maximum at about 9 wt.-%. Under these optimum conditions a solubility of 92.9% was found for an ordinary viscose. From unpublished results of X-ray investigations it is found that the unit cells and the micelles are smaller, more distorted, and less oriented in viscose rayon than in natural fibres such as hemp, cotton, etc. In ordinary viscose the micellar arrangement is regular in the direction of the fibre axis, but random in the other two directions. Some of the hydroxyl groups of the glucose chains are not mutually attracted, hence the valency forces between the micelles are weak and the adsorption of alkali by these groups (or by the micelles) is greater and results in gelatinisation as well as dispersion of the micelles. The greater solubility of the artificial silk in the alkali solution is considered to indicate that the structure of the fibre is less oriented, and the tensile strengths of the fibre in the dry and wet states are also less. B. P. RIDGE.

**Nuera viscose silk.** H. E. FIERZ-DAVID and A. BRUNNER (Helv. Chim. Acta, 1930, 13, 1320; cf. B., 1930, 235).—A reply to Geiger (B., 1930, 1145).

H. BURTON.

**Use of nitrogen tetroxide in nitration of cellulose.** L. A. PINCK (Ind. Eng. Chem., 1930, 22, 1241—1243).—It is shown that nitric acid may be replaced by nitrogen tetroxide in the nitration of cellulose, the method being highly reproducible. The addition of traces of nitric acid to the reaction mixture increases the degree of nitration and reduces the decomposition of the cellulose and its nitric esters. The effects of variation of experimental conditions are tabulated. T. T. PORTS.

**Cataphoresis in nitrocellulose solutions.** E. A. LANTZ and O. A. PICKETT (Ind. Eng. Chem., 1930, 22, 1309—1311).—The cataphoresis was observed by passing a stream of the solution of nitrocellulose through the solvent in which the electrodes were immersed. By illuminating the cell the differences in refractive index of the solution and the solvent could be used to trace the downward path of the solution. The voltages applied were between 90 and 180 volts and the electrodes were 1 cm. apart. The results showed that the nitrocellulose



carries a negative charge, but in some cases the charge is so small as to be nearly negligible. All other factors being equal, the more concentrated solutions showed the greater charge. The solvents used were acetone, ethyl acetate, ethyl acetate-toluene, butyl and amyl acetates. The effect in the solvents decreased in the order given. Solutions made from low-viscosity types of nitrocellulose showed less charge than those prepared from high-viscosity types. When solutions were set aside for several weeks the charge was greatly reduced, but in some cases the charge could be restored by shaking. Addition of potassium iodide in small amount to a solution showing a definite negative charge greatly decreased it and at the same time brought about a lowering of the viscosity.

H. INGLESON.

**Sulphite waste liquors.** HOWARD.—See VII. **Viscose silk and corrosion.** CLOTWORTHY.—See X. **Rosin size.** CHEMNITZ.—See XIII. **Tannery waste.** LLOYD.—See XV. **Whitewater in paper and pulp mills.** MARX. **Wool-scouring effluents.** KING; SMITH.—See XXIII.

#### PATENTS.

**Treatment of gut, particularly in the manufacture of tennis and like stringed racquets.** E. S. BARRALET (B.P. 338,223, 20.8.29 and 19.6.30).—The gut is treated with a solution, in a volatile solvent, of a synthetic resin, *e.g.*, of the phenol-formaldehyde type, formed in the presence of a basic non-volatile condensing agent (caustic soda); the solvent is subsequently volatilised at a temperature below that at which the gut would be detrimentally affected (about 70°). F. R. ENNOS.

**Acylation of cellulose.** F. G. C. KLEIN (B.P. 313,538, 13.6.29. Ger., 13.6.28).—Cellulose is soaked in an aqueous solution of one or more esters which are substantially non-volatile and are not swelling agents for cellulose, *e.g.*, mono-, di-, or tri-acetin; after removal of the excess of saturating solution by centrifuging, the mass is freed from water by drying and then acetylated in the usual way. F. R. ENNOS.

**Acylation of cellulose or cellulose substances.** C. F. BOEHRINGER & SOEHNE, G.M.B.H. (B.P. 312,242, 22.5.29. Ger., 22.5.28).—In order to avoid general or local overheating, *e.g.*, in the acetylation of cotton, while retaining the fibrous structure, where temperatures above 30° are preferably avoided, a volatile substance which does not react with cellulose, but which may act as a catalyst (sulphur dioxide, hydrocarbons, etc.), is added to the reaction mixture and is allowed to evaporate during the reaction so as to lower the temperature.

F. R. ENNOS.

**Production of cellulose ester compounds.** A. S. MCDANIEL, ASSR. to CELLULOSE UTILITIES CORP. (U.S.P. 1,759,489, 20.5.30. Appl., 4.6.23).—Cellulose esters, while in the porous and substantially solid state, are dissolved by bringing them into intimate contact with vapours of a volatile uncombined amino-base (*e.g.*, an alkylamine) and subsequently treating with a relatively small proportion of an organic solvent; the mixture is stirred until the solution acquires the desired consistency, the amino-base being either eliminated by volatilisation or neutralised by a volatile acid, the excess of which is removed by evaporation. F. R. ENNOS.

**Cellulose ester composition [transparent photographic films].** KODAK, LTD., ASSEES. OF C. S. WEBBER and C. J. STAUD (B.P. 336,353, 16.8.29. U.S., 18.3.29).—Mixed esters of cellulose containing an acyl group and an  $\alpha$ -hydroxy- or  $\alpha$ - or  $\gamma$ -ketoacyl group, soluble in acetone or in 50–75% alcohol, are dissolved in a mixture of ethylene dichloride and an alcohol below C<sub>6</sub>, *e.g.*, in 4–8 pts. of ethylene dichloride (75–30 vols.) and alcohol (25–70 vols.), and the solution is evaporated to give a transparent film. C. HOLLINS.

**Precipitation of cellulose acetate solutions.** K. WERNER (B.P. 338,214, 15.8.29).—Separate streams of cellulose acetate solution and of the precipitating liquid, either of which may contain a bleaching agent, flow into each other through a centrifugal pump placed at their point of union, by which they are conveyed onwards in very intimate admixture. (Cf. B.P. 336,349; B., 1931, 16.) F. R. ENNOS.

**Manufacture of cellulose esters containing radicals of different organic acids.** I. G. FARBERIND. A.-G. (B.P. 338,798, 17.1.30. Ger., 25.2.29).—Cellulose is treated, in a diluent if desired, with an organic acid or its anhydride and with the halide of another organic acid in the presence of a tertiary base (pyridine), the quantity of which is less than that which is equivalent to the acid and acid derivatives present. F. R. ENNOS.

**Manufacture of cellulose derivatives [basic ethers].** SOC. CHEM. IND. IN BASLE (B.P. 318,255, 29.8.29. Switz., 31.8.28).—Alkali-cellulose is treated with an amino- or substituted amino-alkyl halide, the product being subsequently acylated or etherified, if desired, by known methods. The ethers or ether-esters may be converted into threads which have affinity for acid and direct dyes. Suitable basic etherifying agents are  $\beta$ -diethylaminoethyl chloride hydrochloride and ethyl- $\beta$ -chloroethylaniline. C. HOLLINS.

**Compositions comprising cellulose derivatives and solvents for use therein.** BRIT. CELANESE, LTD. (B.P. 315,279, 10.7.29. U.S., 10.7.28).—Solvents for organic derivatives of cellulose in the manufacture of films, lacquers, etc., which do not evaporate too quickly, consist of methylene chloride, together with an aliphatic or cyclic alcohol, or a polyhydric alcohol or an ether thereof, having b.p. above 100°. F. R. ENNOS.

**Spinning processes [for fibres] and products obtained thereby.** L. S. M. LEJEUNE and J. E. C. BONGRAND (B.P. 338,381, 25.11.29).—The fibres to be spun are impregnated on the spinning frame with solutions, suspensions, or dispersions of coagulable, polymerisable, or condensable elastic or plastic materials (viscose, synthetic resins, unvulcanised rubber latex) while in the roving stage, the twist for converting the roving into thread being given after the impregnation; the thread is subsequently suitably treated to coagulate, polymerise, or condense the material in the interior of the thread. F. R. ENNOS.

**Production of cuprammonium silk by the stretch-spinning process.** BRIT. BEMBERG, LTD. (B.P. 337,608, 8.11.29. Ger., 19.11.28).—The freshly spun thread is coagulated and stretched, while passing down a cone-shaped insertion in the spinning vessel,

by a stream of precipitant flowing in the same direction with gradually increasing velocity; the fresh precipitant, before coming in contact with the thread, is mixed with a part of the used precipitant which passes through holes in the cone-shaped vessel to the surrounding casing.

F. R. ENNOS.

**Preparation of artificial silk of high-tensile strength from viscose.** BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of NAAML. VENN. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 337,350, 25.7.29. Holl., 8.6.29).—Viscose is spun into a bath containing at least 30% of sulphonated aromatic hydrocarbons, *e.g.*, benzenesulphonic acid, or the reaction mixture used for their preparation.

F. R. ENNOS.

**Manufacture of artificial silk threads, films, etc. from viscose.** I. J. FRENKEL (B.P. 338,318, 21.10.29).—In order to prevent the evolution of noxious gases, 20% of nitrocellulose is added to the cellulose xanthate (calc. on the amount of cellulose in the latter) during the treatment with alkali, and the resulting viscose solution is spun into a bath which, in addition to the usual precipitant, contains up to 0.2% of iron salts in solution.

F. R. ENNOS.

**Washing of artificial silk.** I. G. FARBEININD. A.-G. (B.P. 337,610, 8.11.29. Ger., 8.11.28).—Before entering the spinning box, the thread passes down a guide funnel, where it is washed by a countercurrent of washing liquor, which enters at the lower part and leaves at the upper part of the funnel, the escape of liquor through the thread outlet being prevented or minimised by the injector effect of the flowing liquor, or by the injection of compressed air, or by the narrow dimensions of the outlet.

F. R. ENNOS.

**Conditioning of cellulosic fibre for manufacture of rayon.** G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,754,437, 15.4.30. Appl., 13.7.26. Renewed 28.6.29).—Pulp which disintegrates on soaking in sheet form in caustic soda solution (kraft or soda pulp) is given a preliminary beating, formed into waterleaf sheet, treated with caustic soda, and converted into viscose.

F. R. ENNOS.

**Production of artificial materials from solutions of cellulose or its derivatives.** H. DREYFUS (B.P. 338,490, 17.5. and 12.11.29).—Products of reduced or modified lustre are made by dry- or wet-spinning solutions of organic derivatives of cellulose together with an insoluble substance in finely-divided form, which is added to the spinning solution, to the cellulose derivative, or to the solvent in an amount not exceeding 5 wt.-% of the cellulose derivative; the insoluble substance, which is an ignited metallic salt or compound (barium sulphate, zinc oxide, a silicate, etc.), is first ground dry by mechanical means, graded, and finally ground wet in the presence of a dispersing agent and/or protective colloid to yield particles of uniform diameter (0.0005 and 0.0025 mm.).

F. R. ENNOS.

**Treatment of wood cellulose.** ZELLSTOFFFABR. WALDHOF, and A. BERNSTEIN (B.P. 337,445, 29.7.29).—A high-value cellulose containing (1) 96–99% or (2) 90–95% of  $\alpha$ -cellulose is produced by treating bleached or unbleached wood cellulose with (1) 5% or (2)  $2\frac{1}{2}$ –5%

solution of caustic alkali or alkaline carbonate in the presence of a bleaching agent (chlorine) at 12° or lower, and afterwards washing first with alkali and then with water.

F. R. ENNOS.

**Treatment of paper pulp.** INTERCONTINENTAL RUBBER CO., Assees. of G. H. CARNAHAN (B.P. 338,389, 4.12.29. U.S., 4.12.28).—After removal of non-cellulose encrusting materials, the pulp is hydrated by subjecting it repeatedly to a squeezing and rubbing action, in the presence of water, between rubber surfaces of the tumbling elements in a tube or a ball mill.

F. R. ENNOS.

**Preparation of a raw material [from peat] suitable for artificial silk.** A. BOSSE (B.P. 337,360, 25.6.29).—Peat is boiled in an autoclave with dilute ammonia solution under pressure, acidified, and, after a time, washed until neutral; it is then covered and kept in contact with a bleaching agent (chloride of lime) in water, washed, treated with sodium bicarbonate solution, washed, and pressed. The resulting raw material may then be esterified etc. as desired.

F. R. ENNOS.

**Production of plastic compositions.** BRIT. CELANESE, LTD. (B.P. 317,443, 1.8.29. U.S., 16.8.28).—Plastic compositions containing organic derivatives of cellulose, suitable, *e.g.*, for laminated glass, are rendered more plastic by soaking in water until they have absorbed 1–6% of their weight of water. The bath for soaking may also contain solvents or plastifiers for the cellulose derivatives.

F. R. ENNOS.

**Pulp drying** (B.P. 338,547).—See I. **Proofing of wallpaper** (B.P. 336,244).—See VI. **Treatment of liquors** (U.S.P. 1,754,207–8).—See VII. **Insulating material** (B.P. 316,146).—See XI. **Transfer compositions** (B.P. 338,611 and 338,673). **Rosin solutions** (U.S.P. 1,759,526). **Plastic threads etc.** (B.P. 319,371).—See XIII. **Treatment of skins etc.** (B.P. 337,377).—See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Dyes suitable for dyeing synthetic-tanned leather.** G. E. KNOWLES (J. Soc. Leather Trades' Chem., 1930, 14, 562–567).—Basic dyes are readily fixed by synthetic-tanned leather, since the synthetic tannin is a good mordant for them. Acid dyes can be divided into four groups, according as they dye synthetic-tanned leather full, fairly full, medium, or weak shades, respectively. Two or more basic groups are present in the molecules of those acid dyes which yield full or fairly full shades on synthetic-tanned leather. Amongst a table of suitable dyes are: (for full shades) Soluble Blue, Acid Violet 4R, Erioviridine, Acid Violet 5BN; (fairly full) Wool Green S, Nigrosine, Acid Milling Red G; (medium) Acid Red S, Bordeaux Extra, Orange R and II, Azo Yellow, Resorcine Brown; (weak) Eriofloxine 2GI and 6BI, Benzyl Bordeaux, Naphthol Yellow S. The direct cotton dyes which give the fullest shades on synthetic-tanned leather contain both basic and acid groups, *e.g.*, Benzo Fast Scarlet 4BS, Carbide Black E, and Direct Blue 2B.

D. WOODROFFE.

**Chlorinated diphenyl.** PENNING.—See III.

## PATENTS.

**Fibrous product and its manufacture.** F. T. LAHEY (B.P. 337,359, 19.6.29).—Fibrous material, freed from extraneous matter, is mordanted, *e.g.*, by an aqueous solution of ammonium sulphate, tannates, etc., and treated with a dispersion of rubber latex and a gelatinous mixture containing a curing agent and other compounding ingredients such as gums, starches, sugars, etc. After forming into the desired shape, the product is dried, with or without pretreatment with suitable impregnating material, or further coagulated with lactic acid and dried, or coagulated and vulcanised.

F. R. ENNOS.

**Manufacture of artificial threads and filaments [of reduced lustre].** COURTAULDS, LTD., and C. DIAMOND (B.P. 338,269, 16.9.29).—Filaments which have been dry-spun from a solution of an organic ester of cellulose containing a small proportion of an animal or vegetable oil (arachis oil) are boiled with an aqueous soap solution, washed, and dried.

F. R. ENNOS.

**Proofing of leather, textiles, wallpaper, wood, etc. against fungoid infections.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 336,244, 1.5.29. Cf. B.P. 334,876; B., 1930, 1167).—Hydroxy-di- or -tri-arylmethanes, *e.g.*, the product from formaldehyde and 2 or 3 mols. of *p*-chlorophenol, or the tetrabrominated product from *cyclohexanone* and 2 mols. of phenol, are applied as fungicides.

C. HOLLINS.

**Hydro-extractor (U.S.P. 1,766,310).**—See I. Pigments for coloured surfaces (B.P. 317,763).—See XIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Chamber [sulphuric acid] process. XXI. Electric conductance method.** M. MATSUI, T. OKAMOTO, and T. NODA (J. Soc. Chem. Ind., Japan, 1930, 33, 446—450B).—The conductivity of nitrous vitriol is proportional to the "nitre" content, but is affected also by the sulphuric acid concentration. In order to effect an automatic control of nitre feed by the concentration of the nitrous vitriol it is therefore necessary to dilute the latter to  $d$  1.3 and pass an air current through it, the evolved gases being collected in a water tube the resistance of which is recorded. A series of trials with nitrous vitriol of known nitrosity gave a smooth resistance-concentration curve. In works' practice a current measurement with constant voltage might be made to operate an automatic nitric acid feed control (cf. Jap. P. 88,062).

C. IRWIN.

**Potassium salts from insoluble minerals.** G. CALCAGNI (Annali Chim. Appl., 1930, 20, 522—527).—Treatment of leucite in the cold with higher oxides of nitrogen gives potassium and aluminium nitrates. The latter salt is converted by heating into nitrogen oxides and alumina, from which aluminium is obtained, and the residual porous silica has marked adsorbent properties.

T. H. POPE.

**Use of anhydrite (calcium sulphate) in the production of ammonium sulphate.** Report III of the Ammonia Sub-Committee. P. PARRISH (Gas J., 1930, 192, 736—739).—The costs of manufacturing

ammonium sulphate by the sulphuric acid and anhydrite processes are compared. It is shown that the latter method may result in a saving of not less than 8s. 8d. per ton, allowing for cost of carriage of anhydrite to London. Experiments on the conversion of ammonia into ammonium bicarbonate are described, and it was found that these two reactions are best carried out in separate vessels and not simultaneously in one vessel. It is concluded that the production of ammonium sulphate from anhydrite is a conventional chemical works' operation, and that the nature of the process necessitates a medium-size scale of operations; its application in gasworks producing less than 30—40 tons of sulphate is therefore considered undesirable.

C. B. MARSON.

**Treatment of waste sulphite [pulp] liquors.** G. C. HOWARD (Ind. Eng. Chem., 1930, 22, 1184—1185).—Waste liquor is treated with lime to give a precipitate consisting mainly of calcium sulphite, which is used for the preparation of fresh cooking acid. The supernatant liquor treated with a further quantity of lime causes precipitation of lignin compounds which may be used as fuel. The process is arranged to be continuous, and the resulting final effluent is claimed to have a low biochemical oxygen demand and to be less toxic than the usual sulphite waste liquors.

T. T. POTTS.

**Effect of copper and lead ions on the rate of decomposition of hydrogen peroxide at various acidities.** H. W. RUDEL and M. M. HARING (Ind. Eng. Chem., 1930, 22, 1234—1237).—The rate of decomposition of a 30% aqueous solution of hydrogen peroxide at 32° has been studied in presence of copper sulphate and of lead chloride to which sulphuric acid and sodium hydroxide were added. Although the effect of copper ions is to accelerate the decomposition, the increase in  $p_H$  changes the form in which the copper is present in the solution. This leads to the production of maxima and minima in the curves which represent decomposition and  $p_H$  changes for a given copper content. Lead ions exert an inhibitory action on the decomposition. An attempt is made to provide explanations of the phenomena observed.

H. INGLESON.

**Properties and uses of helium.** W. E. SNYDER and R. R. BOTTOMS (Ind. Eng. Chem., 1930, 22, 1189—1191).—The thermal conductivity of helium is nearly six times that of air, which fact renders it valuable as a heating or cooling medium, especially as a cooling medium for electrical equipment. For airship work its greater density as compared with hydrogen is set off by the diffusion of the latter gas through the envelope being 50% more rapid. Its low solubility renders a helium-oxygen mixture suitable for caisson work. Its inertness renders certain metallurgical applications possible, and, together with its low density, renders it suitable for the grinding and grading of inflammable materials. The gas may also replace the use of vacuum in evaporation and in food containers. A diagram of a helium extraction plant operating in two stages is given. No external refrigeration is used. Portable plants have also been constructed for repurifying the gases in airships.

C. IRWIN.

**Viscosity data.** GENEREUX.—See I. Ammonia from coal carbonisation. EASTWOOD and COBB.

**Products from wood-distillation.** SCHOFIELD. **Ammonia and hydrogen sulphide from coal gas.** PARKER. **By-product ammonia.** HOLLINGS and SMITH.—See II. **Filter cloth.** GRADL.—See V. **Chrome-iron in nitric acid plant.** BELLINGER. **"Black-bronzing" of brass.** KELLNER and CURTIS. **Metals from waste.** HINCHLEY.—See X. **Cottrell units.** CAMP.—See XI. **Schweinfurt-green.** THÜRMER. **Cadmium pigments in lacquers.** SIEBERT.—See XIII. **Tannery waste.** LLOYD.—See XV.

## PATENTS.

**Manufacture of sulphuric acid of high purity.** II. HOWARD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,764,309, 17.6.30. Appl., 22.4.27).—Sulphuric acid formed in a number of gold or platinum tubes charged continuously with sulphur dioxide and water at opposite ends flows through a manifold into a cascade of 15–25 quartz or pyrex glass goose-necked flasks in which it is blown with hot, filtered air to remove sulphur dioxide. The blown acid, after cooling if necessary, is collected in glass carboys. L. A. COLES.

**Concentration of sulphuric acid [recovered from acid sludge from the purification of oils etc.].** F. E. KIMBALL (U.S.P. 1,764,210, 17.6.30. Appl., 9.11.27).—A cylindrical evaporator contains a vertical partition wall provided with openings partly or totally submerged in the acid under treatment. Inlets for the dilute acid and for furnace gases and an outlet for the concentrated acid are situated on the one side and an outlet for the waste gases on the other side of the partition wall. The waste gases pass through a compartment in which they are mixed with cold air and thence into an electrical precipitator, the recovered acid being returned to the evaporator. L. A. COLES.

**Production of nitric acid from ammonia.** N. CARO and A. R. FRANK (B.P. 338,566, 13.8.29).—Oxides of nitrogen obtained by the catalytic oxidation of ammonia and subsequent removal of water by condensation are highly compressed in a turbo-compressor constructed, *e.g.*, of V2A steel, for conversion by condensation and absorption into concentrated nitric acid; the residual gases are allowed to expand to normal pressure in an expansion turbine coupled with the turbo-compressor. (Cf. B.P. 337,847; B., 1931, 63.) L. A. COLES.

**Treatment of residual liquors. Production of cooking liquor which contains sodium hydroxide.** L. BRADLEY and E. P. MCKEEFE, ASSRS. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,754,207—8, 8.4.30. Appl., [A] 2.3.21, [B] 20.10.26. Renewed [A] 26.6.28).—(A) Residual liquors from the treatment of cellulosic materials by an alkaline process are treated with sodium hydrogen sulphate to remove organic matter, which is separated and washed; the resulting solution is concentrated to separate some of the sodium sulphate, and treated with a calcium compound, the acid radical of which forms a sodium salt causticisable with lime (calcium phosphate, fluoride, sulphite), in the presence of sufficient acid to bring about the reaction between the sodium sulphate and the calcium salt to give a precipitate of gypsum and a solution of the sodium salt, which latter is then causticised by addition of lime.

In (B) the cooking liquors are concentrated, carbonised, and leached to yield a solution containing sodium carbonate, sulphide, and carbon, which is partly causticised with lime and, after separation of the lime mud and part of the suspended carbon, is further causticised with excess of lime; the separated lime mud and carbon from the second operation (which contains excess of lime) is used for treatment of another batch of liquor in the first step of the process, whilst the lime mud and carbon from the first operation is returned for use again. F. R. ENNOS.

**Production of ammonium sulphate by the treatment with gypsum of an ammoniacal solution used for the scrubbing of gases.** UNION CHIM. BELGE SOC. ANON. (B.P. 338,373, 22.11.29. Fr., 17.5.29. Addn. to B.P. 307,037; B., 1929, 472).—In the process described in the prior patent, precipitation of the ammonium sulphate is caused to take place in a part of the apparatus where the warm ammoniacal solution is mixed with a large volume of colder solution saturated with ammonium sulphate. The cooling of the ammoniacal solution is effected in repeated operations, each involving only a slight fall of temperature, so that precipitation in the cooling part of the plant is avoided.

W. J. WRIGHT.

**Treatment of natural alkaline waters or brines to obtain therefrom commercially useful substances.** J. G. MILLER and A. C. HOUGHTON (U.S.P. 1,759,361, 20.5.30. Appl., 25.4.29).—The brine is cooled to 5° so as to separate crystalline sodium carbonate with a small amount of crystalline sodium sulphate, which are removed from the mother-liquor. To prevent a high concentration of sodium hydrogen carbonate in the mother-liquor and consequent precipitation of colloidal silica, sodium hydroxide is added to the brine before refrigeration. The avoidance of the silica precipitation ensures the retention in the mother-liquor of any colouring matter. W. J. WRIGHT.

**Preparation of calcium cyanamide or magnesium cyanamide or mixtures containing the same.** N. CARO and A. R. FRANK (B.P. 338,023, 4.12.29. Ger., 6.12.28. Addn. to B.P. 279,811; B., 1929, 53).—The water formed in the reaction in the prior process is removed continuously by the addition of, *e.g.*, carbon or carbonaceous material, in which case the metal oxides can be used instead of the carbonates, or of phosphorus pentoxide, the use of which yields a product of improved fertilising properties. These substances may be mixed with the other constituents, or may be placed behind the actual reaction space. L. A. COLES.

**Production of anhydrous aluminium halide from a double halide of aluminium and ammonium.** C. G. MINER (U.S.P. 1,754,797, 15.4.30. Appl., 28.6.26).—Aluminium nitride is treated with hydrochloric acid gas at about 1000° to form a double halide of aluminium and ammonium. This is caused to react in the liquid, solid, or gaseous state with phosphorus pentachloride or sulphide at a raised temperature, the aluminium chloride being separated from the phosphorus compound by volatilisation. W. J. WRIGHT.

**Manufacture of aluminium chloride.** H. BLUMENBERG, JUN. (U.S.P. 1,764,501—2, 17.6.30. Appl., [A]

14.4.27, [B] 12.2.30).—A mixture of aluminium sulphate with (A) an alkali or, preferably, ammonium chloride, or (B) an alkaline-earth chloride and carbonaceous material, is heated at a temperature such that the aluminium chloride produced by double decomposition is removed by distillation.

L. A. COLES.

**Production of metallic hydroxides and other metallic compounds.** R. S. CARRERAS (B.P. 338,178, 9.7.29).—A dilute solution of a substance such as sodium chlorate is saturated with carbon dioxide and electrolysed, the anodes being of the metal of which it is desired to produce the hydroxide. The chlorate solution is continuously regenerated, the exhausted electrolyte being drawn off from the bottom and replaced by a corresponding amount of fresh solution into which carbon dioxide is injected.

W. J. WRIGHT.

**Manufacture of finely-divided zinc oxide.** A. CAREMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,792, 7.8.29).—A zinc salt solution is added in slight excess to an alkali carbonate solution, both solutions being less than 1.5*N*, and the precipitated zinc carbonate is removed, washed, ground, and sifted, and calcined below red heat.

L. A. COLES.

**Treatment of [removal of iron from] lyes from chlorinated burnt pyrites.** A. CAREMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,822, 8.8.29).—The lyes, after precipitation of copper by the addition of iron, are treated with calcium chlorate to oxidise the iron, which is then precipitated with lime to render the liquor suitable for the recovery of zinc, cobalt, etc.

L. A. COLES.

**Preparation of solutions of gold and silver [for gilding or silvering].** A. MOZER (B.P. 338,383, 27.11.29, Ger., 7.1.29).—The metal is dissolved in a 2% solution of iodine in a 11–12% solution of an alkali halide, preferably potassium iodide. The resulting solution may be used for electroplating or for coating by dipping.

A. R. POWELL.

**Highly active adsorbent and catalytic mass.** F. STORWENER and E. MÜNZING, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,760,289, 27.5.30, Appl., 6.10.27, Ger., 13.10.26).—A heterogeneous gel is washed and, while moist, subjected to a process of mechanical homogenisation, which consists in grinding or kneading, or forcing it under high pressure at high velocity through one or more pipes of small diameter; it is then dried.

W. J. WRIGHT.

**Carriers for catalysts.** IMPERIAL CHEM. INDUSTRIES, LTD., F. A. F. CRAWFORD, and W. A. P. CHALLENGER (B.P. 337,761, 3.5.29).—The gelatinous precipitate obtained by the addition of an acid or, e.g., ammonium carbonate to a suspension of kieselguhr in an alkali silicate solution is used as a carrier for platinum, vanadium compounds, etc. The catalyst may be applied after drying, or the gel may be stirred with, e.g., a solution containing ammonium vanadate and potassium hydroxide and the mixture dried and granulated or worked into pellets.

L. A. COLES.

**Dry fire-extinguishing compositions.** D. J. BLOCK (B.P. 319,372, 21.9.29, U.S., 22.9.28).—The tendency of sodium bicarbonate to cake on storage is removed by the addition of small quantities of, e.g.,

magnesium stearate, magnesium carbonate, and tricalcium phosphate.

L. A. COLES.

**Production of carbon dioxide.** P. BROTHERHOOD, LTD., H. M. DUNKERLEY, and CARBON DIOXIDE Co., LTD. (B.P. 338,369, 19.11.29).—The flue gases from the limestone retorts are used to generate steam in a waste-heat boiler, this steam being returned to the retorts and mixed with carbon dioxide. The mixture of steam and carbon dioxide is employed to operate a prime mover, being subsequently passed to a condenser in which the steam is condensed, and from which the carbon dioxide is drawn off by a pump or compressor.

W. J. WRIGHT.

**Separation of gaseous mixtures** (B.P. 338,497).—See I. **Hydrogen** (B.P. 336,635).—See II. **Treatment of ores containing copper etc.** (B.P. 338,556). **Acid-resistant nickel alloy** (U.S.P. 1,753,904).—See X. **Manufacture of per-salts** (B.P. 318,154).—See XI. **Fertiliser** (B.P. 338,644).—See XVI. **Removing ammonia from air** (U.S.P. 1,767,057).—See XXIII.

## VIII.—GLASS; CERAMICS.

**Commercial glass transmitting ultra-violet light.** P. DAVIDOVITCH (J. Opt. Soc. Amer., 1930, 20, 627–641).—For the manufacture of glass transmitting ultra-violet light the raw materials must be free from iron and titanium, and the amount of oxygen in the furnace flames must be kept constant. Phosphate glasses are unsuitable, on account of their high cost and poor resistance to the atmosphere. The transparency of commercial UV-glass diminishes on illumination and on heating.

C. W. GIBBY.

**Shade numbers and densities of eye-protective glasses.** W. W. COBLENTZ and R. STAIR (J. Opt. Soc. Amer., 1930, 20, 624–626).—The shade numbers of eye-protective glasses are reduced to a standard formula of densities and transmissions.

C. W. GIBBY.

**Examination of antique glass.** E. BERL [with F. BENDER] (Chem.-Ztg., 1930, 54, 913–914).—A glass goblet, which had become opaque through age and had developed thin surface flakes, gave on analysis: 72.8% SiO<sub>2</sub>, 19.3% (K<sub>2</sub>O + Na<sub>2</sub>O), and 5% CaO; the flakes contained 73.4% SiO<sub>2</sub>, 18.6% (K<sub>2</sub>O + Na<sub>2</sub>O), and 5.48% CaO. This close approximation in the relative compositions indicates that atmospheric conditions, which appreciably affect glass of high alkali content, had not been responsible for the deterioration. Confirmation for this lies in the fact that the ground portions, usually specially susceptible to atmospheric influences, showed no attack. It is concluded that the effects were caused by strains in the external surface, developed by irregular cooling in manufacture, the absence of internal strain being due to the use of an interior mould. Old glass is best protected by a coating of cellulose lacquer, but the effects of strains can be prevented only by re-annealing.

W. J. WRIGHT.

**Behaviour of refractory materials in continuous vertical retorts.** Report 21 of the Refractory Materials Joint Sub-Committee. A. T. GREEN (Gas. J., 1930, 192, 720–724).—Data are given which show the deleterious influences affecting the life of refractory materials in continuous vertical retorts. The

precautions to be taken in the heating and cooling of a setting are discussed. Visual examination of the retort brickwork indicates the amount of flaking and spalling, the nature and amount of slag attack, the behaviour of cement in the joints, and the probable causes of any holing. Variation from the original alinement may occur, due to the high swelling pressures of certain coals; flaking of the retort often takes place at 10–12 ft. from the top, whilst slagging reactions occur in the lower zones. Slagging reactions are noticeable in all vertical retorts, the extent depending on the nature of the inorganic constituents, the design of the retort, and the manipulation of the retorts, particularly with respect to the temperature of working and degree of steaming; slagging causes erosion, failure of the brickwork, and "sticking" of the charge. The tendency to flake is increased by the formation of scurf and by the present method of removal, which causes localised high temperatures, and by high and variable flue temperatures. Spalling often occurs where the cement from a joint has been eliminated, the corner of the brick then spalling away, whilst rapid cooling of the retort also increases the risk of spalling; close-fitting sections requiring but little cement should be used and care taken in cooling a retort. The conditioning of hot retorts by hot patching mixtures has received little attention, and it is necessary that such mixtures do not contain appreciable quantities of fusible constituents. It is concluded that the zonal nature of a vertical retort might justify the use of different types of refractories and cements according to the zone of the retort. C. B. MARSON.

**Furnace-testing refractories for slag erosion.** B. W. STROMBERG (Chem. & Met. Eng., 1930, 37, 685–686).—A laboratory furnace 12 ft. × 5 ft. × 6 ft. holding 30 test bricks has been designed to reproduce works' conditions without elaboration. A temperature of 1550° can be maintained steadily by an oil burner combined with a turbulent powdered-coal burner, the coal flame impinging on the test bricks and providing the slagging test. Addition of fluxes or stiffeners to the coal burned gives a wide field of slag variation for tests, and fluxes can be used for accelerating the test process. High-grade fireclay bricks eroded about 1 in. in 48 hrs. at 1450°, silica bricks about  $\frac{3}{4}$  in., whilst silicon carbide bricks were hardly attacked. Means are provided for heating the outside of the bricks only, as representing boiler conditions, when heat flows and temperature gradients are to be measured. E. A. RYDER.

#### PATENTS.

**[Production of] laminated glass.** H. A. GARDNER (U.S.P. 1,762,513, 10.6.30. Appl., 23.5.27).—The binder contains a toluenesulphonamide-aldehyde resin (cf. U.S.P. 1,564,664; B., 1926, 202), and if desired a nitro-cellulose or an oxycellulose compound. L. A. COLES.

**Uniting materials [e.g., glass plates etc.] by adhesives or cements.** CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 314,379, 18.6.29. Ger., 26.6.28).—Non-porous articles are cemented by sheets or strips of rubber, canvas, paper, cellulose, etc. coated with polymerisation products of vinyl esters, preferably the acetate (cf. B.P. 271,090; B., 1928, 793). L. A. COLES.

**Manufacture of moulded glassware. Manufacture of glass in the form of sheets or plates.** CHANCE BROS. & Co., and (A) R. BROWN, (B) A. L. FORSTER (B.P. 339,484 and 339,535, [A] 3.12.29, [B] 23.1.30).

**Manufacture of laminated glass.** F. B. DEHN. FROM LIBBEY-OWENS GLASS CO. (B.P. 338,988, 30.8.29).

**Moulds for manufacture of articles in silica glass or any other vitreous material which is fusible with difficulty.** QUARTZ & SILICE (B.P. 339,197, 14.2.30. Fr., 30.10.29. Addn. to B.P. 338,456).

**Tunnel furnace (U.S.P. 1,768,486).**—See I. Plastic compositions (B.P. 317,443).—See V.

#### IX.—BUILDING MATERIALS.

**Manufacture of Portland cement and cupriferrous iron from slag.** J. A. BRADBURY (Chem. Eng. Min. Rev., 1930, 22, 443–449; 23, 27–29).—Attempts on a laboratory scale have been made to utilise an available dump of 5 million tons of blast-furnace slag by smelting it in an arc furnace so as to produce a slag of similar composition and properties to Portland cement and also a copper-bearing iron. Magnesite brick formed the most satisfactory lining in spite of severe corrosive attack with considerable reduction to metallic magnesium which was recovered in the flues in re-oxidised form. High coke consumption was experienced and reduction to a point where the slag was of cement composition was accomplished only when the tapped slag contained calcium carbide, which decomposed on granulation leaving soluble lime, the sugar-soluble figure of 3.5% being considered equivalent to normal cement. When cooled slowly in the furnace the slag spontaneously subdivided itself into an impalpable powder without any setting properties, which, however, appeared in varying degree if the slag was chilled more rapidly and pulverised. Water-granulation of the hot slag aided the removal of calcium carbide and the hydration of free lime without deleterious effects. Iron produced under highly reducing conditions favoured the presence of silicon and carbon and was in consequence coarse-grained and brittle; the following analysis indicates the nature of the metal: Fe 88.8%, Cu 0.95%, Ca nil, Mg 1.1%, Si 5.6%, C 3.05%, P 0.5%, S 0.04%. It is probable that the power costs would be an adverse factor to larger-scale working. C. A. KING.

**Magnesium oxide cements. III. X-Ray examination of basic magnesium chlorides.** W. FEITKNECHT (Helv. Chim. Acta, 1930, 13, 1380–1390; cf. B., 1927, 300).—The X-ray diagrams of basic magnesium chlorides containing 2.3–7 mols. of hydroxide per mol. of chloride show none of the lines of the magnesium hydroxide diagram, from which it is concluded that in these compounds the hydroxide is not present as a separate phase (cf. Maeda, B., 1926, 587). Various crystalline species may be present, depending on the ratio of hydroxide to chloride, and with at least two of these the solid hydroxide has a limited miscibility. The same crystals have been detected in Sorel cement after setting, and here also no evidence of the presence of mechanically admixed hydroxide has been obtained.

It would therefore appear that in the setting process the oxide is converted directly into basic chloride without the intermediate formation of hydroxide. In cement with a high chloride content the crystals first formed may undergo slow transformation into a more stable form. The decrease in hardness of hardened cement on desiccation is accompanied by changes in crystalline form. R. CUTHILL.

**Causes of bad agreement between strengths of cements determined as pure cements and in sand mortars.** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 425—427 B).—Numerous tests have shown that the tensile strength of cement is modified by the proportion of water added, and that for each cement there is an optimum cement-water ratio. In the case of pure cement it is decreased by fineness of division, but with sand mortars finer grinding increases the strength. As both these factors operate in the reverse direction in the two cases (pure cements and mortars), the divergence in tensile strengths under the two conditions of testing is natural. C. IRWIN.

**Dental cements.** O. RUFF, C. FRIEDRICH, and E. ASCHER (Z. angew. Chem., 1930, 43, 1081—1087).—Analyses of various dental cements in use are given. The powder consists of either zinc oxide or alumina and silica, or of a mixture, and the fluid with which it is mixed is phosphoric acid or a solution of acid zinc phosphate. All depend for their setting on the production of a phosphate gel, the silicate cements having no hydraulic properties *per se*. The plastic qualities depend on the fineness of division of the powder and the viscosity of the liquid. The setting time and compression strength of each cement were also determined. The authors have investigated new cements in which various oxides formed the powder and silicofluorides the liquid. Good results were obtained with zinc oxide or alumina and zinc fluosilicate. The zinc oxide cement gave quicker setting and greater final strength. Neither is equal to cements now used in these respects, and they are more brittle. The reaction of formation is  $\text{ZnSiF}_6 + 2\text{ZnO} = 3\text{ZnF}_2 + \text{SiO}_2$ . The setting of these cements was studied microscopically and it was concluded that the essential quality of a satisfactory cement was capacity of gel formation. Another new cement is prepared by heating to 1400° 1 mol. of lanthanum oxide ( $\text{La}_2\text{O}_3$ ) and 2 mols. of silica and mixing with phosphoric acid or acid zinc phosphate. The qualities of this cement are equal to those of standard dental cements. There is a tendency to give slight acidity to aqueous extracts. The addition of beryllium oxide prevents this, but causes contraction on setting. C. IRWIN.

**Wood waste.** SCHOFIELD.—See II.

#### PATENTS.

**Rotary [cement] kiln.** R. D. PIKE (U.S.P. 1,766,453, 24.6.30. Appl., 6.5.26).—In a dry-process cement kiln, of which the length is at least 30 times the principal diameter, a preheating zone is formed in an enlarged upper end, which is subdivided by corrugated radial and cylindrical partitions. Dust carried over by the induced draught is settled and returned by a dust pump to the lower end of the enlarged zone.

B. M. VENABLES.

**Manufacture of hydraulic mortar and the constituent parts thereof.** W. REISMANN, R. H. SCHLOSS, and W. H. EVERS (B.P. 338,664, 3.10.29).—A 1:3 mixture of a binder, comprising 92 pts. of finely-ground, granulated, foundry pig-iron slag and 8 pts. of burnt magnesite, with fine aggregate (*e.g.*, sand, cinder, sawdust), is worked up with 50 pts. of water-glass (*d* 1.21) per 100 pts. of binder and sufficient water to yield a homogeneous mortar. L. A. COLES.

**Preparation of bituminous emulsions and the like.** C. G. J. LEFEBVRE and E. E. F. BERGER (B.P. 319,663, 18.9.29. Fr., 25.9.28).—In order to enable an emulsion of bitumen in soapy water to be mixed with a powdered stone filler without breaking down the emulsion, 1—3% of an alkaline salt of a weak acid, *e.g.*, sodium carbonate or stearate, is added to the emulsion at the time when it is to be used. A. B. MANNING.

**Manufacture of bituminous material for application to road surfaces.** SOUTH METROPOLITAN GAS CO., H. PICKARD, and H. STANIER (B.P. 335,668, 23.7.29. Addn. to B.P. 316,897; B., 1929, 803).—A dispersion of 1—10% of coal in tar, made at about 300°, is mixed with 30—60 wt.-% of finely-divided mineral matter. The dispersion may contain not more than 10% of added pitch. A. B. MANNING.

**Removal of the slipperiness from road surfaces.** I. G. FARBERIND. A.-G. (B.P. 337,928, 11.10.29. Ger., 12.10.28).—The roads are sprinkled with organic solvents capable of dissolving oils and fats and of dissolving or swelling rubber (*e.g.*, petroleum fractions, coal-tar distillates), which may be used in aqueous suspension or emulsion and in conjunction with soaps, soda, wetting agents, sand, etc., after which they are brushed and rinsed with water. L. A. COLES.

**Apparatus for spraying liquid material [*e.g.*, road-surfacing preparations].** B. HORNE (B.P. 339,354, 7.9.29).

**Emulsifier** (B.P. 338,496).—See I. Bituminous emulsions (U.S.P. 1,752,449).—See II. Proofing of wood etc. (B.P. 336,244).—See VI. Carbon dioxide (B.P. 338,369).—See VII. Floor coverings (B.P. 318,239). Coloured surfaces (B.P. 317,763). Floor-covering material (B.P. 317,375).—See XIII.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Developments in fuel economy at Skinningrove [ironworks].** F. BAINBRIDGE (J. Iron & Steel Inst., 1930, 121, 97—119).—Economies in fuel consumption have been made by bringing into balance the outputs of the various gas-producing departments and by making available large quantities of surplus gas for use in the production of steel. All the stoves and boilers are equipped with automatic burners and coke-oven gas is fed to the open-hearth furnaces through water-cooled burners. The cleaned blast-furnace gas and surplus coke-oven gas are mixed with producer gas in predetermined proportions by means of simple diaphragm-type meters and electrical regulators. These modifications in the lay-out of the gas plant and the utilisation of the gases produced have resulted in a saving of 32,000 tons of coal per annum. A. R. POWELL.



**Interactions of gases and ore in the blast furnace.**  
**II. Carbon deposition at 450° and its influence on the ore reduction: equilibria between gases and ore at 650° to 1000°.** W. A. BONE, L. REEVE, and H. L. SAUNDERS (J. Iron & Steel Inst., 1930, 121, 35—81; cf. B., 1927, 484).—In the reaction between blast-furnace gases (33% CO and 67% N<sub>2</sub>) and native hæmatite the deposition of carbon, due to the reversible reaction  $2\text{CO} \rightleftharpoons \text{C} + \text{CO}_2$ , begins at about 275° and proceeds at optimum velocity at 450°. Iron, ferrous oxide, and ferrosoferrous oxide are all powerful promoters of the reaction up to about 650°, and at 450° there appears to be no limit to the progress of carbon deposition in the presence of any of the promoters; above 700°, however, the deposition becomes inappreciable until about 90% of the ore has been reduced, when it again becomes of importance. In the blast furnace the deposited carbon may impregnate the ore granules, in which case this carbon is a much more powerful reducing agent than carbon monoxide at 750°, or it may envelop the ore granules, in which case it is only a poor reducing agent. For carbide formation the presence of free iron in a system containing both oxides of carbon and an ore in the course of reduction is necessary. The maintenance of partly reduced ores for considerable periods above 750° usually causes sintering. Matsubara's ratios for the CO/CO<sub>2</sub> equilibrium at 750—975° in the systems  $\text{Fe}_3\text{O}_4 + \text{CO} \rightleftharpoons 3\text{FeO} + \text{CO}_2$  and  $\text{FeO} + \text{CO} \rightleftharpoons \text{Fe} + \text{CO}_2$  have been confirmed, and measurements have been made of the rate of approach to equilibrium over the 30—65% ore-reduction range for two different types of hæmatite ore.  
 A. R. POWELL.

**Physical chemistry of steel-making.** C. H. HERTY, JUN. (Met. & Alloys, 1930, 1, 883—889).—A survey of a research in progress in America on the action of oxygen on molten steel. The solubility of ferrous oxide in steel has been measured at various temperatures under a variety of slags. As part of a study of the non-metallic inclusions present in steel treated with deoxidisers, the constitution of some of the more fluid slags in the binary and ternary systems formed by this oxide with manganous oxide, silica, and alumina has been investigated by thermal and microscopical methods on slags made in the induction furnace. Viscosity measurements at raised temperatures have been made by means of the torsion viscosimeter on lime-silica slags. Minimum viscosity appears to correspond to the compound  $\text{CaO-SiO}_2$ . Lowering of the carbon content of steel has been shown to be followed by an increase in the content of ferrous oxide. In a study of the physical properties of clean and dirty steels it was found that impact values decrease as the content of inclusions becomes greater, but that ultimate tensile strength, yield stress, elongation, reduction in area, and the fatigue range are more profoundly affected by the nature and habit of the inclusions than by their amount. Among the analytical methods developed is an electrolytic process for separation of non-metallic matter.  
 E. H. BUCKNALL.

**Conditions of formation and decomposition of cementite.** P. PINGAULT (Compt. rend., 1930, 191, 1007—1008; cf. B., 1928, 818).—Pure cementite, pre-

pared by the action of sodium cyanide on electrolytic iron filings at 650°, is quite stable in a vacuum up to 1000°; slight decomposition occurs at 1075°, complete at 1175°. Cementation of pure iron does not occur when heated in sodium cyanide at temperatures up to 900°, provided air is absent. With this is compared the fact that grey cast iron heated at 675° in sodium cyanide exposed to the air shows an outer zone from which all carbon has been burned away. On electrolysis sodium cyanide at 650° with soft steel electrodes, with a current of 1 amp./dm.<sup>2</sup> in 40 hrs. the anode is hardened, the cathode unchanged; with 20 amp./dm.<sup>2</sup> there is no hardening, but corrosion of the anode occurs; with 60 amp./dm.<sup>2</sup> in 15 hrs. the anode shows a loss of weight of 2.5 g./cm.<sup>2</sup>, the detached powder being slightly oxidised cementite.  
 C. A. SILBERRAD.

**Single-sheet or thin-pack normalising, or heat treatment *versus* box-annealing of [steel] sheets.** R. WHITFIELD (J. Iron & Steel Inst., 1930, 121, 131—146).—High-quality steel sheets suitable for deep drawing are much more easily and cheaply produced by normalising at 570—600° in single sheets or thin packs in a slightly reducing atmosphere in a three-zone reverberatory-type annealing furnace than by the older method of box-annealing. In the first-named method the preheating, soaking, and cooling operations are all carried out under carefully controlled conditions to produce a sorbitic structure which imparts high ductility and elastic limit to the metal.

A. R. POWELL.

**Carbon loss and diffusion in the malleabilising process [for iron castings].** E. TOUCEDA (Met. & Alloys, 1930, 1, 815).—Eight blocks, each  $\frac{3}{4}$  in. thick, of cast iron have been subjected to a malleabilising treatment of 7 days' duration, according to normal (American) practice. The original carbon contents ranged from 1.99% to 2.66%. Analysis after treatment has shown a decrease of approx. 0.35% in the average carbon contents of the blocks. Millings taken from successive layers have also been analysed. The results indicate a very definite zone of decarburisation to a depth of  $\frac{1}{8}$  in., followed by a marked increase in carbon content. The composition at the centre of the blocks is almost unchanged by the treatment.

E. H. BUCKNALL.

**Chromium-copper structural steels.** J. A. JONES (J. Iron & Steel Inst., 1930, 121, 209—217).—The mechanical properties of a series of steels containing 0.3% C, 0.5—1% Mn, up to 1.5% Cr, and up to 1.2% Cu have been determined after normalising at different temperatures and after slowly cooling. The tests show that addition of chromium only to a plain manganese steel with up to 1% Mn has little effect on its value as a constructional material, but a marked improvement is produced by 0.5—1.2% Cu in addition to about 1% Cr; with about 1% Cu the resistance to atmospheric corrosion is increased and the sensitivity to the rate of cooling after normalising is diminished. The mechanical properties of the steel with 0.3% C, 0.5% Mn, 0.9% Cr, and 1.2% Cu compare well with those of the best manganese-silicon structural steels and excel those of the usual structural steels containing a high manganese

content only; the above chromium-copper steel has an elastic limit of 23 tons/in.<sup>2</sup>, a yield point of 28 tons/in.<sup>2</sup>, a tensile strength of 40.8 tons/in.<sup>2</sup>, an elongation of 31%, a Brinell hardness number of 182, and an Izod impact figure of 55 ft.-lb.

A. R. POWELL.

**Chrome iron in nitric acid plant.** E. ST. P. BELLINGER (Chem. & Met. Eng., 1930, 37, 691-692).—In proportion to the chromium content, the corrosion-resisting properties of chrome steel are much higher between 12 and 18% Cr, an optimum economic chromium content also being reached at 18%. More than 0.12% C or above 1% Si (probably) reduces the resistance to corrosion, but a small amount of silicon is essential. The low-carbon alloys resemble iron or mild steel and have been applied successfully in nitric acid plants. Experimental corrosion data covering the above facts is tabulated. Generally, cast materials corrode more than worked. For minimum-corroding chrome irons adequate and carefully controlled annealing is essential. Prolonged heating above 315°, or heating above 930° and air cooling, reduces corrosion-resistance, but the latter property can be restored by re-annealing.

E. A. RYDER.

**Initial corrosion rate of steels.** H. O. FORREST, B. E. ROETHEL, and R. H. BROWN (Ind. Eng. Chem., 1930, 22, 1197-1199).—The initial corrosion rate of steel samples was determined by pickling with 10% hydrochloric acid for 15-20 min., washing with oxygen-free water, and then rotating in water of known oxygen content for a given time. The decrease in oxygen content of the water was measured. Curves were obtained which suggest that the initial rate of corrosion of all steels, stainless or ordinary, is the same. In the case of chromium or chromium-nickel steels the rate of corrosion falls off very rapidly, action ceasing altogether in a few minutes. Blank tests indicated that the small oxygen loss found was not due to adsorbed hydrogen, and the results support the protective-film theory.

C. IRWIN.

**Corrosion of pipes. 1st Rept. of Corrosion of Pipes Sub-Comm., Inst. Gas Eng.** (Gas J., 1930, 192, 725-729).—Experiments on the use of various materials such as paints, waterproof tape, and cement, with the object of preventing electrolytic corrosion, are described. It is suggested that protective coatings must be carefully applied, otherwise protection against corrosion may be achieved only at the expense of intensified local electrolysis.

C. B. MARSON.

**Pickling of pipe using commercial inhibitors.** E. L. CHAPPELL and P. C. ELY (Ind. Eng. Chem., 1930, 22, 1201-1203).—The efficiency of various inhibitors proposed for use in pipe-pickling was determined by measuring the hydrogen evolved in a given time from a given quantity of 4% sulphuric acid containing the inhibitor acting on a  $\frac{1}{2}$ -in. length of  $\frac{1}{2}$ -in. pipe. Each inhibitor will have an optimum concentration, a slightly lower one being usually employed in practice. It is emphasised that some inhibitors prolong pickling time unduly, and others may have an adverse effect on subsequent treatment of the metal, such as zinc coating by the hot-dip process.

C. IRWIN.

**Dissolution of scale in pickling steel.** E. L.

CHAPPELL and P. C. ELY (Ind. Eng. Chem., 1930, 22, 1200-1201).—It was found that in a tank in which pipes were pickled in sulphuric acid, less than 5% of the scale removed was in the undissolved state, this residue consisting of iron silicates. The rate of action on scale and on iron of the acid is approximately the same in the absence of an inhibitor. The latter does not affect the rate of dissolution of scale. On the other hand, scale removed from a pipe and treated with acid in a laboratory appears to dissolve more slowly than scale in contact with a pipe.

C. IRWIN.

**Corrosion problems in the viscose silk industry.**

H. R. S. CLOTWORTHY (Ind. Chem., 1930, 6, 445-447).—Vessels which come into contact with cellulose xanthate solutions, and vessels in which carbon disulphide is stored under water, should be made of stainless steel, or plated with chromium or nickel. In the spinning operations the external surfaces of the pumps for the viscose solutions are liable to be attacked by the acid spray from the coagulating liquid, and here again plating is suggested. Tantalum jets, ebonite filters, glass godets and funnels, and Bakelite spinning boxes are used. There is no satisfactory metal for construction of the arms carrying the funnels, nor for the guides for the rods supporting these arms, but phosphor-bronze is the most resistant to corrosion. In the finishing process glass, ebonite, and ebonite-covered steel are largely used. Stainless steel would be preferable to steel for storage tanks. A less breakable substitute than the ebonite lining of vessels for sodium hypochlorite liquor and hydrochloric acid is desirable. The hanks of silk are hung on chromium-plated tubes to dry.

D. K. MOORE.

**Cupola melting of bronze.** E. R. DARBY (Met. & Alloys, 1930, 1, 812-813).—Copper-tin, copper-tin-lead, and other alloys have been melted in a cupola fired with pitch coke, a fuel containing little sulphur or ash and over 99% C. The crucible of the cupola held 500 lb. of molten bronze, and the charge was melted at the rate of 2500 lb./hr. Loss of lead was about 8% of the original content. Loss of zinc was high and phosphorus was almost completely eliminated.

E. H. BUCKNALL.

**Deposition of black copper oxide on brass.** H. L. KELLNER and H. A. CURTIS (Ind. Eng. Chem., 1930, 22, 1321-1324).—In the "black-bronzing" of brass the metal is dipped into an ammoniacal solution of basic copper carbonate. Aqueous ammonia was agitated with varying amounts of basic copper carbonate over long periods (up to 80 hrs.), and it was found in every case that the undissolved residue changed in character, the colour passing from the original light green to brownish-black. This change was the more rapid the lower was the ratio of ammonia to basic copper carbonate in the system. The results of subsequent analyses seem to show that the basic copper carbonate is decomposed when in contact with aqueous ammonia, leaving copper oxides or hydroxide in the residue. By replacing a portion of the ammonia by ammonium carbonate, a satisfactory black-bronzing bath could be made by using about half the normal amount of basic copper carbonate, thus cheapening the process. The deposit,

although appearing continuous and opaque to the unaided eye, is translucent when viewed under the microscope. Application of X-rays failed to show any crystal structure in the deposit. During the process the brass becomes deeply etched. H. INGLESON.

**Rapid determination of zinc in brass alloys.** THEWS and HARBISON (Chem.-Ztg., 1930, 54, 947—948).—In order to determine the zinc content of a charge of molten brass made freshly from pure zinc and copper so that necessary adjustments can be made in the composition before casting, a small sample ingot is cast and drilled, 0.2 g. of the drillings is dissolved in nitric acid, the solution made ammoniacal, acidified with acetic acid, and treated with potassium iodide, and the liberated iodine titrated with thiosulphate. For accurate results the reagents are added in standard quantities from burettes and the thiosulphate is standardised against previously prepared standard brass samples. The zinc is determined by difference within 0.5% and the test takes only 7—8 min. after a little practice.

A. R. POWELL.

**Thermal conductivity of the system copper-nickel.** G. F. SAGER (Rensselaer Polytech. Inst., 1930, Bull. No. 27, 48 pp.).—Measurements have been made of the thermal and electrical conductivities of copper, nickel, and of alloys of the two over the complete range of compositions, up to a maximum temperature of 743°. Sp. heats have been determined by the method of mixtures. The Lorenz ratio for copper is practically constant from 30° to 500°; that for nickel is almost the same at room temperature, but increases markedly with temperature. The values obtained are higher than those of previous workers. The ratios for the alloys are higher than those for copper and pass through minima at about 300°.

C. W. GIBBY.

**Uses of aluminium in the chemical industry.** S. J. RALPH (Ind. Chem., 1930, 6, 459—464).—Because of the difficulty of obtaining sound castings, wrought aluminium of 98—99% purity, the use of which has been rendered possible by the development of a satisfactory method of welding, is preferred for large plant. Aluminium-copper and aluminium-silicon alloys have good casting properties, but are less resistant to corrosion than is pure aluminium, but a modified silicon alloy containing 0.05% Na casts well, shows good resistance to corrosion, and has a greater tensile strength and elongation than the normal silicon alloy of similar composition.

D. K. MOORE.

**Extrusion of bismuth wire.** J. G. THOMPSON (Met. & Alloys, 1930, 1, 826—827).—Results are recorded of tests made by the Bureau of Standards in collaboration with the Cerro de Pasco Copper Corporation. Billets of commercial bismuth (99.99% Bi) have been extruded at temperatures between 0° and 100° by means of a press which is described in detail. Dies 0.012, 0.025, and 0.031 in. in diam. were made of hardened carbon steel, with an angle of entrance of 45° and a short length of parallel orifice. Pressure was applied to the plunger by means of a testing machine arranged for compression. Wires 20 ft. long were produced from sound billets, but no success was obtained with powdered bismuth in the press. The wire could

be formed into coils of small diameter, but could not be bent sharply without fracture and was not amenable to cold-drawing. E. H. BUCKNALL.

**Determination of platinum in platinum ore.** W. R. SCHOELLER (Analyst, 1930, 55, 550—553).—The platinum is completely precipitated as ammonium chloroplatinate and the impure precipitate is re-treated on the principle of separating into (a) a major and (b) a minor fraction, the latter containing the residue of the platinum in less pure condition. The original precipitate is redissolved by the action of a stream of chlorine on the suspension in boiling water, the solution partly evaporated after addition of just enough ammonium chloride (10% solution) to combine with the platinum, and the liquor is diluted with an equal bulk of half-saturated ammonium chloride solution and left to cool. The coarsely crystalline precipitate which forms contains most of the platinum and iridium, free from other metals. The filtrate from this precipitate and from the previous ammonium chloride precipitate are then treated for the separation of the fraction b. Details are given for the treatment of platinum minerals, and results should agree within 0.001 g. Pt. Palladium is tested for by means of the dimethylglyoxime test. The iridium is determined by the lead-fusion method. Rhodium (maximum amount present in the ore was 0.015 g. per 1.06 g. Pt) could not be detected in the final platinum precipitate after using this chlorine process.

D. G. HEWER.

**Materials [metals] for high-temperature service from the creep design viewpoint.** J. J. KANTER and L. W. SPRING (Met. & Alloys, 1930, 1, 880—882).—Resistance to scaling is essential in most high-temperature plant, especially in boilers and in oil-refinery apparatus. In the design of turbines, where the permissible deformation due to creep must not exceed 0.2% in 20 years, an accurate knowledge of the temperature at which detectable creep first sets in is essential. Further research on fatigue properties at intermediate temperatures and on the influence of temperature on the value of the Poisson ratio is necessary before designs can be made on theoretical grounds. The approximate temperatures permitting creep of various metals and alloys are tabulated.

E. H. BUCKNALL.

**Recovery of metals from waste materials.** J. W. HINCHLEY (Inst. Chem. Eng., Dec., 1930, 59—66).—Copper alloy scrap is usually freed from water and grease in a continuous roaster under reducing conditions. Briquetting facilitates the addition of fluxes. Melting methods fail to give satisfactory materials for the manufacture of a good bronze, owing to the presence of extraneous metals. Scarpa's electrolytic method employs the scrap bronze as anode plates. The copper is deposited in the cathode with a purity of 99.97%, whilst the tin is found in the slime, from which it is recovered by extraction with caustic soda. Processes for the recovery of tin from tin plate include electrolysis usually in caustic soda; the Goldschmidt chlorine process, which is the one most employed; and wet chemical processes dependent on caustic soda. The action of caustic soda on tin plate is very slow, but it is accelerated by oxidising agents. The author has

improved the litharge alkaline process, which now works in a continuous cycle, the loss of lead as insoluble oxide being avoided. The liquor, containing 4.42% Pb and 10.25% NaOH by wt., strips the tin from tinplate in a few seconds at 90°. Finely-divided lead is deposited and is removed from the plate by agitating in revolving drums. The sodium stannate solution is treated with caustic lime and the lead and regenerated caustic soda are treated with air in a tower for 20 hrs. It is advisable to remove solder and grease by pretreatment. A flow sheet for the process is given. The dissolution of lead in caustic soda by air-blowing in a special apparatus is effected at 45–50°. The air must, of course, be freed from carbon dioxide. Most of the plant is constructed of mild steel. Dumps of refuse near old works may sometimes be treated metallurgically for recovery of metal contents, after grinding. The author has investigated Mackay's process for the leaching of zinc-lead residues with dilute sulphuric acid. A solution of zinc sulphate was obtained containing small amounts of copper, silica, and iron, all easily removable, whilst the lead sulphate in the residue was removed by treatment with calcium chloride solution. C. IRWIN.

**Welding in the chemical industry.** F. LEVERICK (Ind. Chem., 1930, 6, 448–451).—The weld should have the same composition as the plates. Welding of stain'less steel causes a portion of it to be maintained at 800° for some time. This has a deleterious effect on the steel which should be removed by annealing at 1100°. In welding alloys of high nickel content, *e.g.*, monel metal, care should be taken that there is no stress on the weld at temperatures just below that of solidification. For chemical plant strapped welding is most useful, because it gives smoothness and strength. D. K. MOORE.

**Effect of various metallic sulphates on the throwing power of a chromium-plating bath.** L. E. SROUT and J. CAROL (Ind. Eng. Chem., 1930, 22, 1324–1325).—The baths were made with 250 g. of chromium trioxide per litre and proportions of zinc, cadmium, cobalt, and nickel sulphates in which the ratios  $\text{CrO}_3 : \text{SO}_4$  varied from 15:1 to 300:1. The electrolysis was carried out at 55°, using an anode of perforated platinum foil and two polished nickel cathodes placed on either side of the anode. The current density was 35 amp./dm.<sup>2</sup>, and voltages up to 14 volts were used. Mercurous and mercuric sulphates could not be used. Magnesium sulphate gave very poor deposits. The addition of all the sulphates to the chromic acid bath in the ratios  $\text{CrO}_3 : \text{SO}_4 = 200:1$  or  $150:1$  increased the throwing power. Cadmium sulphate used in these proportions gave the highest throwing power, whilst nickel and cobalt sulphates gave a deposit much more resistant to the action of hydrochloric acid. With the proportion of sulphates last mentioned the metals do not deposit at the cathode with the chromium. H. INGLESON.

**Electrodeposition of chromium-iron alloy.** G. FUSEYA and K. SASAKI (J. Soc. Chem. Ind., Japan, 1930, 33, 474–482 B).—Experiments were made to ascertain the best conditions for electrodeposition of iron containing more than 16% Cr. It was found, when

electrolysing chromium sulphate alone, that an acid content of 0.05–0.1 mol.  $\text{H}_2\text{SO}_4$  per litre was necessary for a deposit of bright metallic chromium to be formed. With less acid solutions chromium hydroxide was produced. Trials were then made with solutions of this acidity containing both chromium and iron, using copper plates as cathodes and magnetite rods as anodes. It was found that increasing the current density increased also the chromium content of the deposit and the (lower) current efficiency. Increasing the acidity gave the opposite effect, as also did a rise in temperature. It was found that the deposit always contained a small percentage of oxide. The best conditions existed when the concentration was 1–2.5 mols. Cr and 0.5 mol. Fe per litre, the temperature 15°, and the current density 8–15 amp./dm.<sup>2</sup> The deposit then contains 30–45% Cr, and the current efficiency is about 10%. X-Ray spectrograms showed that the deposit is a solid solution. It resists sodium chloride, ammonium chloride, ammonia solutions, and concentrated acetic acid when the base metal is completely covered. C. IRWIN.

**Three crystalline modifications of electrolytic chromium.** K. SASAKI and S. SEKITO (J. Soc. Chem. Ind., Japan, 1930, 33, 482–485 B).—The authors, while examining the chromium-iron alloy deposits of Fuseya and Sasaki (*cf.* preceding abstract) confirmed Ollard's statement that chromium may be deposited in either a hexagonal close-packed form or a cubic form, or as a mixture of the two. They obtained also another modification of the  $\alpha$ -manganese type, containing 58 atoms per unit cube. The values of  $d$  for the three forms are calculated as 6.08, 7.21, and 7.48, respectively. The two other forms are slowly changed into the cubic form at room temperature. C. IRWIN.

**Preparation of metal powders by electrolysis of fused salts. II. Thorium.** F. H. DRIGGS and W. C. LILLIENDAHL (Ind. Eng. Chem., 1930, 22, 1302–1303; *cf.* B., 1930, 668).—The electrolysis was carried out under the following conditions. A graphite crucible capable of holding 1 kg. of a melted 1:1 mixture of potassium and sodium chlorides was provided with a platinum anode and a molybdenum cathode 2.5 cm. wide and having an area of 50 cm.<sup>2</sup> The operating temperature was 750–775°, and after 60 g. of potassium thorium fluoride ( $\text{KThF}_6$ ), prepared by precipitation from the nitrate, had been dissolved in the bath, a current of 45 amp. was passed for 20 min. This cathode was then withdrawn and a fresh one inserted together with an additional 60 g. of the double fluoride. The deposited metal adhering to the cathode was washed free of soluble salts. The current efficiency of the process was 58%. The purified metal has been utilised for X-ray targets, and has been formed into sheet and wire. The metal is very soft, and bars of metal can be readily worked repeatedly without intermediate annealing. The metal contained 0.019% C, 0.046% Si, and 0.005% Fe as impurities. H. INGLESON.

**Technique of radiography [of metals] by gamma rays.** C. S. BARRETT, R. A. GEZELIUS, and R. F. MEHL (Met. & Alloys, 1930, 1, 872–879).—A technique for the detection of internal defects in metals is described.

X-Ray films are employed, the sensitivity being increased by placing thin screens of lead on each side of the film, as the secondary electrons emitted by the lead have more effect on the emulsion than have  $\gamma$ -rays themselves. The back of the film was protected from stray radiation by means of thick lead sheets. Fogging was less pronounced than in X-ray work. Radium emanation (sometimes radium) was used as the source of radiation.

E. H. BUCKNALL.

**Open-hearth combustion.** CHANDLER.—Sec II. **Helium.** SNYDER and BOTTOMS.—Sec VII. **Cupriferous iron from slag.** BRADBURY.—Sec IX. **Iron-chromium alloys.** FISCHER. **Cottrell units.** CAMP.—Sec XI.

#### PATENTS.

**Aluminothermic process for the generation of highest temperatures.** E. BLÜMNER (B.P. 338,468, 11.3.30).—Molten iron oxide and aluminium contained in two separate concentric chambers separated by a refractory wall are caused to interact by starting an aluminothermic mixture in proximity to a weak part of the wall so that the latter melts and allows the molten materials to come into contact. The operation is conducted in a closed, water-cooled, and refractory-lined vessel under a pressure of 200 atm. of an inert gas.

A. R. POWELL.

**[Heat-treatment] furnace [for wires etc.].** J. KURIK, Assr. to J. LORINCZ (U.S.P. 1,765,430, 24.6.30. Appl., 13.12.27).—A metal bath, *e.g.*, for annealing or tinning wires, is heated by an inverted reverberatory furnace, the earlier part of the flame being screened from contact with the bottom of the bath.

B. M. VENABLES.

**Treatment of air blast for furnaces.** G. MALYOTI (B.P. 338,244, 28.8.29).—The air is dried by means of calcium carbide and preheated to 350° before passing to the tuyeres of the furnace. The amount of acetylene formed in the drying is below the explosion limit and the slaked lime produced is useful for briquetting the flue dust.

A. R. POWELL.

**Manufacture of iron and other metals.** DAVIS STEEL PROCESS CORP., Asscs. of E. W. DAVIS (B.P. 315,760, 3.7.29. U.S., 17.7.28).—A mixture of ore, fluxes, and an excess of coal or other carbonaceous reducing agent is smelted in a reverberatory furnace under such conditions that the temperature in the smelting zone is above that at which graphitisation of the fuel and rapid carburisation of the reduced iron occur.

A. R. POWELL.

**Carburising of iron or steel.** R. G. GUTHRIE and O. J. WOZASEK, Assrs. to PEOPLE'S GAS BY-PRODUCTS CORP. (U.S.P. 1,768,317, 24.6.30. Appl., 30.3.29).—The metal is heated repeatedly for short periods at 800–1050° in an atmosphere of acetylene, or benzene or petrol vapour, the supply of which is interrupted when carbon begins to be deposited. During the intervals in the carburising treatment the metal is treated with a current of air, steam, or carbon dioxide to reactivate the steel surface.

A. R. POWELL.

**Refining of iron or steel.** F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 317,373, 13.8.29. Ger.,

14.8.28).—The molten metal bath is treated with water under pressure which is introduced by means of an inclined pipe terminating some distance below the surface of the bath. In this way the whole of the water is decomposed before it reaches the surface of the bath, and the resulting gases cause violent agitation and circulation of the molten metal below the usual layer of refining slag. The process causes a rapid removal of the phosphorus, sulphur, and silicon. A. R. POWELL.

**Manufacture of cast iron.** H. D. ELKINGTON. From MEEHANITE METAL CORP. (B.P. 337,844, 13.8.29).—Cast iron containing an alkaline-earth metal or its silicide, *e.g.*, calcium silicide, in insufficient quantity to produce a grey fracture is annealed above the Acl point (about 800°) for 12 hrs. and cooled in the air. The resulting metal has a high tensile strength with an elongation up to 7% and a reduction in area up to 10%.

A. R. POWELL.

**Production of composite metal castings.** W. ALBERTS and F. STEIN (B.P. 338,092, 21.3.30. Ger., 28.8.29).—Partition walls of electrolytic or Armco iron containing more than 99.8% Fe are used in making the castings.

A. R. POWELL.

**Treatment of malleabilised cast iron. Heat-treatment of metals [cast iron].** LINK-BELT CO., Asscs. of (A, B) C. F. LAUENSTEIN, (A) N. M. WATERBURY (U.S.P. 1,760,239 and 1,760,240, 27.5.30. Appl., [A] 29.12.26, [B] 7.11.27).—(A) The castings are annealed above 800°, preferably in a salt bath containing cyanides, until a considerable proportion of the carbon has been converted into cementite; cooling is effected at a controlled rate to prevent decomposition of the carbide and to avoid undue hardening. (B) The castings are annealed above 800°, quenched, and reheated at 650° to decompose martensite and troostite. A. R. POWELL.

**Protective treatment of metals [iron or steel].** A. ARENT (B.P. 338,221, 20.8.29).—The surface of the metal is scrubbed with a solution of antimony trichloride in benzene (2.5 lb./gal.), whereby it becomes coated with a layer of antimony which is tightly adherent, non-porous, and resistant to corrosion.

A. R. POWELL.

**Coating of metals [iron, with lead alloys].** E. R. MILLRING, Assr. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,760,603, 27.5.30. Appl., 21.7.23).—The metal to be coated is cleaned in a pickling bath, covered with a zinc chloride flux, and immersed in a bath of a super-cooled alloy of 12.5% Sb, 10% Bi, and 77.5% Pb, either directly or after first coating with an alloy of lead and 3% of phosphor-tin.

A. R. POWELL.

**Composition for and process of cleaning metal [iron or steel].** J. D. KLINGER and C. L. BOYLE (U.S.P. 1,752,746, 1.4.30. Appl., 20.6.29).—A solvent for grease and rust comprises a mixture of 35 vol.-% of 75% phosphoric acid, 15–20 vol.-% of the monobutyl ether of ethylene glycol, and 50–45 vol.-% of water.

A. R. POWELL.

**[Inhibitor for pickling baths for the] treatment of iron or steel.** K. B. THEWS and J. S. CARLITZ (U.S.P. 1,759,635, 20.5.30. Appl., 7.12.29).—The inhibitor is a substance of the general formula  $\text{NR}(\text{C}(\text{XR}')\text{NR}_2)$ , where R is hydrogen or an alkyl, aryl, or acyl

group, R' is the same or another group of one of these types, and X is sulphur, selenium, or tellurium;  $\psi$ -benzylthiourea is specifically mentioned.

A. R. POWELL.

**Manufacture of iron alloys. [Determination of the resistance of ferrous alloys to corrosion.]** VEREIN. STAHLWERKE A.-G. (B.P. 338,071, 24.1.30. Ger., 30.1.29).—The fatigue strength of the alloy is determined in the air and in a fine spray of water; the percentage reduction in the fatigue strength in the second case is a measure of the resistance of the alloy to corrosion.

A. R. POWELL.

**Steel alloy.** J. W. HEYMAN and S. L. ZAVON (MULTI-STEEL Co.), Assees. of J. T. WHITELEY (B.P. 337,919, 3.10.29. U.S., 24.1.29).—Claim is made for a steel containing 0.4–0.75% Mo, 0.1–0.3% Cr, 0.2–0.5% Si, and 0.5–1.0% Mn, to which is added just before pouring a mixture containing zirconium, titanium, and sodium dichromate in the ratio, respectively, of 0.5–1.5% : 0.1–0.3% : 0–0.1%, based on the weight of steel treated. In addition, 0.5–0.9% Cu and 0.1–0.35% Ni may be added to improve the strength and ductility.

A. R. POWELL.

**Production of iron-chromium alloys which are stable on heating.** SKODA WORKS, PLZEN, LTD. (B.P. 337,767, 5.7.29. Czechoslov., 18.4.29).—Alloys of iron with 20–40% Cr, 0.2–0.3% C, 1–15 (1–5)% Ti, and 0–15 (5)% Al are claimed. The alloys expand and contract regularly during heating and cooling.

A. R. POWELL.

**[Chromium-iron] alloys [resistant to scaling at high temperatures] and articles made therefrom.** (SIR) R. A. HADFIELD (B.P. 337,893, 11.9.29).—Alloys for electrical resistors and furnace windings comprise iron with 24–35% Cr, 3–5% W, 3–7% Al, and only small percentages of carbon, silicon, and manganese. With less than 4% Al the alloys can readily be forged, rolled into sheet, and drawn into wire. A. R. POWELL.

**Manufacture of iron and iron-nickel and iron-silicon alloys.** ASSOCIATED ELECTR. INDUSTRIES, LTD., Assees. of T. D. YENSEN and N. A. ZIEGLER (B.P. 338,409, 20.12.29. U.S., 18.1.29).—For the production of pure iron or of iron-nickel or iron-silicon alloys a charge is prepared in which the carbon and oxygen in the constituents are present in equal atomic proportions. The charge is then melted in an induction furnace under a pressure of less than 1 mm. Hg, whereby the whole of the carbon and oxygen is removed as carbon monoxide. The metal is allowed to cool at a rate between 50° and 300°/hr. in order to produce the best magnetic properties.

A. R. POWELL.

**Magnetic material [nickel-iron alloy].** G. W. ELMEN, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,768,238, 24.6.30. Appl., 12.1.27).—An alloy containing 78% Ni, 21% Fe, and 1% Si, heat-treated at 1100° and cooled at 5°/min., is claimed to have a high permeability at low magnetising forces. A. R. POWELL.

**Making or re-melting alloys of aluminium and iron.** BRITISH & DOMINIONS FERROALLOY, LTD., and J. W. BAMPFYLDE (B.P. 338,688, 21.10.29).—Iron is melted under 1–2% of fluorspar and, after skimming off the slag, 15–16% of aluminium is added together with

another 1% of fluorspar. By operating in an induction furnace sound castings free from inclusions may be obtained with only relatively small melting losses and small wear on the crucible. A. R. POWELL.

**[Nitrogenised aluminium-]steel alloys.** NITR-ALLOY, LTD., Assees. of H. A. DE FRIES (B.P. 338,655, 23.9.29. U.S., 9.10.28).—Claim is made for a nitrogen case-hardened steel containing 2.1–6% Al, 0.05–2% C, and 0.2–8% Si, Mn, Ni, Cr, Cu, Mo, W, V, Ti, or Zr, or a mixture of any or all of these elements. The preferred composition is 4% Al, 1.25% C, and 4% Si or one or more of the above elements. A. R. POWELL.

**Heat-treatment of nitrogenised alloy-steel articles.** R. SERGESON (B.P. 338,315, 18.10.29).—After nitrogenisation in the usual way at 450–540°, the articles are annealed at a temperature between 590° and the lower critical point of the steel, preferably at 650°, whereby the tendency of the case to chip or spall on bending is overcome. A. R. POWELL.

**Treatment of ores etc. containing copper and/or nickel.** E. A. ASHCROFT (B.P. 338,556, 20.8., 3.9., and 25.11.29, and 12.4.30).—The nickel or copper ore, which may be oxidised or sulphidic, is mixed, if necessary, with a sulphide ore of the same metals or of iron, and the mixture is treated at 400° with a current of oxygen or air enriched with oxygen, the gas stream being so regulated that the temperature of the mass, which is not rabbled or otherwise disturbed, is maintained at about 600°. Ferrous chloride or sulphate may be used instead of sulphide ores for admixture with oxidised copper or nickel ores. The roasted product is leached with water, which dissolves the greater part of the copper and nickel and gives a neutral sulphate solution almost free from iron; treatment of this solution with ammonia provides ammonium sulphate and nickel and copper oxides. A. R. POWELL.

**[Chromium-nickel-copper] non-ferrous alloys.** G. H. WHITEMAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 338,676, 14.10.29).—Up to 4% of chromium is substituted for a corresponding proportion of either nickel or copper in a 70 : 30 copper-nickel alloy; the preferred alloy contains 67.5% Cu, 30% Ni, and 2.5% Cr and is suitable for the manufacture of non-corroding condenser tubes, evaporators, etc. A. R. POWELL.

**Treatment of metals [melting 60 : 40 palladium-copper alloy].** C. H. ANDERSON, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,752,474, 1.4.30. Appl., 31.3.27).—The constituent metals are melted in a graphite crucible in an atmosphere containing hydrogen and/or carbon monoxide and the alloy is allowed to cool in the crucible, transferred to a lime, magnesia, or zirconia crucible, remelted in air, and, when evolution of gas ceases, cast into chill moulds. A. R. POWELL.

**Acid-resistant [nickel] alloy.** C. E. PLUMMER, Assr. to ELECTRO METALLURG. Co. (U.S.P. 1,753,904, 8.4.30. Appl., 3.11.28).—An alloy of nickel with 9–11% Si, 2.5–25% Cu, 1.5–2% Al, and less than 1% Fe is claimed. The preferred composition is 85% Ni, 10% Si, 3% Cu, and 1.5% Al. A. R. POWELL.

**Treatment of manganese [dioxide] ores.** J. H. BRENNAN, Assr. to ELECTRO METALLURG. Co. (U.S.P.

1,768,112, 24.6.30. Appl., 28.6.28).—Finely-ground ferruginous pyrolusite is mixed with sufficient silicon to reduce the iron to metal and the manganese dioxide to manganous oxide and the mixture is fired, whereby an alloy of iron with a little manganese, silicon, and phosphorus is obtained together with a manganese silicate slag almost free from iron and phosphorus.

A. R. POWELL.

**Extraction of tin from ores, alloys, scrap, etc.** SOC. D'ELECTRO-CHIMIE, D'ELECTRO-MÉTALLURGIE, ET DES ACIÉRIES D'UGINE (B.P. 338,149, 9.8.29. Fr., 22.6.29).—The process described in B.P. 217,900 (B., 1925, 926) is modified so that the ferrosilicon produced as a by-product contains more than 30% Si, and is therefore insoluble in hydrochloric acid. Any tin retained by the ferrosilicon can then be removed by digesting the ground material with acid or with stannous chloride. [Stat. ref.]

A. R. POWELL.

**Increasing the elongation and the bending and rolling capability of sheets, bands, wires, etc. consisting of aluminium alloys.** ALUMINIUM IND. A.-G. (B.P. 337,922, 4.10.29. Ger., 12.10.28).—Aluminium alloys of high tensile strength but low ductility are provided with a double coating of another aluminium alloy having a low tensile strength but good ductility and bending properties. The second alloy consists of aluminium with 1–4% Mn, 0.5–6% Mg, and up to 1% Sb, or with 0.3–1% Mg, 0.5–2% Si, and 0.5–2% Mn; in addition either alloy may contain up to 2% Pb.

A. R. POWELL.

**Production of coloured protective layers on the surface of aluminium alloys and articles made of aluminium alloys.** A. PACZ (B.P. 338,204, 16.8.29).—The articles are immersed for a short time in a hot (90°) solution containing 0.14% of chromic acid and about 2% of sodium carbonate. Different colours may be imparted to the coating by introducing small quantities of appropriate metal salts into the bath. A. R. POWELL.

**Recovery of lead and other metals from ores or other lead-bearing materials.** U. C. TAINTON (U.S.P. 1,759,494, 20.5.30. Appl., 19.9.27).—Lead carbonate or sulphate ore is ground in a 2–5% solution of sodium hydroxide containing 0.1% of sodium cyanide, and the filtered solution of sodium plumbite is electrolysed for the recovery of argentiferous lead sponge, which is melted and refined as usual. The spent electrolyte is agitated with lime to regenerate the sodium hydroxide for use again.

A. R. POWELL.

**Magnesium-covered calcium wire.** W. A. RUGLES, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,752,813, 1.4.30. Appl., 9.1.26).—Magnesium is sublimed in a vacuum and the vapour condensed on calcium wire. The treated wires are suitable for use as getters in radio valves etc.

A. R. POWELL.

**Preparation of zirconium.** W. P. KIERNAN, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,413, 27.5.30. Appl., 16.9.26).—A finely-ground mixture of zirconia, anhydrous calcium chloride, and calcium filings is heated at 950° in an iron-chromium-nickel alloy bomb fitted with a mild steel liner. The reaction product is extracted with a large volume of cold water to remove

calcium chloride, then with dilute nitric acid to remove the lime formed in the reduction. The resulting coarse zirconium powder is thoroughly washed, dried, and pressed into bars which are heated first at dull redness under a pressure of 0.01 mm. until all gas is completely removed, and then at a temperature just below the m.p. to effect complete sintering. The bars are forged at 900°, swaged, and drawn or rolled. A. R. POWELL.

**Manufacture of uranium metal and [molybdenum-alloy] product.** J. A. HEANY, ASSR. to ARCTURUS RADIO TUBE Co. (U.S.P. 1,759,454, 20.5.30. Appl., 22.10.25).—A mixture of molybdenum powder with up to 5% of uranium oxide is pressed into bars which are heated in hydrogen by means of a current equal to 90% of that which is sufficient to melt the bar. After cooling, the bar is forged, swaged, and drawn into wire which has a high electronic emission and is suitable for the filaments of radio valves.

A. R. POWELL.

**Production of metal [copper] sheets by electro-deposition.** H. M. WILLIAMS and R. G. SUMAN, ASSRS. to GEN. MOTORS RES. CORP. (U.S.P. 1,760,028, 27.5.30. Appl., 1.12.24).—Deposition is made on a rotating, horizontal, cylindrical cathode the cylindrical surface of which is made of chromium or an iron alloy containing more than 20% Cr. Copper does not adhere to these types of surface, so that the deposit may readily be stripped from the cathode.

A. R. POWELL.

**[Chromium] electroplating bath.** J. BEDO, ASSR. to PERMACHROME PROCESS CORP. (U.S.P. 1,754,075, 8.4.30. Appl., 5.7.28).—A bath containing chromic acid and sodium thiosulphate, preferably in the proportion of 50 : 1, is claimed.

A. R. POWELL.

**Electrolytic production or separation of tin.** L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 338,488, 16.5.29).—For the production of tin by electrolysis of alkaline stannite or stannate baths, using soluble anodes, the composition of the bath is so adjusted that the ratio of free alkali to tin is 1 : 5 with a minimum content of 1.5% of free alkali.

A. R. POWELL.

**Electrolytic production of aluminium.** VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 337,995, 14.11.29. Ger., 27.11.28).—The usual electrolytic furnace is provided with means whereby fresh supplies of alumina are automatically charged into the furnace at a rate equal to that at which it is reduced to metal in the bath.

A. R. POWELL.

**Manufacture of aluminium by electrolysis.** COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES, ET CAMARGUE (B.P. 338,084, 19.2.30. Fr., 15.3.29).—The bath comprises a mixture of aluminium fluoride and sodium or potassium carbonate in the molecular ratio 4 : 3 instead of cryolite. On fusion this salt mixture yields a solution of alumina in cryolite.

A. R. POWELL.

**Process and apparatus for varnishing wire.** INTERNAT. GEN. ELECTRIC Co., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 339,534, 22.1.30. Ger., 25.1.29).

**Heating with metal vapours** (U.S.P. 1,760,095).—See I. **Gilding and silvering solutions** (B.P.



338,333).—See VII. Cathodes for electrolysis (B.P. 318,154).—See XI. Coatings for tin-foil etc. (B.P. 338,340).—See XIII. Ore-conveyor belts (B.P. 338,536).—See XV.

## XI.—ELECTROTECHNICS.

**Magnetic and electrical properties of some iron-chromium alloys.** F. K. FISCHER (Rensselaer Polytech. Inst., 1930, Bull. No. 28, 32 pp.).—The magnetic properties and electrical resistances of iron-chromium alloys containing up to 20% Cr have been investigated after annealing at 1000°, annealing with subsequent baking at 500°, quenching at 900°, and quenching with subsequent baking. Annealing produces the best magnetic properties. The region of zero magnetostriction is 17.5—18.7 Cr; the properties of alloys in this range are least affected by quenching. Baking after annealing reduces the permeability. The electrical resistance of the alloys is 43—53 microhm-cm., varying 0.60 microhm-cm. per 1% Cr. The magnetic critical point varies between 675° and 750°, with an average value of 700°. The best alloy obtained contained 18.7% Cr and when annealed had a maximum permeability of about 2000 at approx. 6000 gauss. It has about one third the permeability and four times the hysteresis loss of Armco iron. An alloy in which the ratio Fe:Cr is 82.5:17.5 and containing 10% Ni is non-magnetic.

C. W. GIBBY.

**Carbon tubes in Cottrell units.** A. D. CAMP (Chem. & Met. Eng., 1930, 37, 676—677).—Carbon tubes used in Cottrell precipitators resist attack by strong corrosive agents. They have a high electrical conductivity, a low thermal expansion giving minimum breakage, and are subject to less distortion than lead tubes. They have been used successfully in gases containing phosphorus pentoxide and hydrogen fluoride from phosphate plants, sulphur dioxide from zinc and copper smelters, and in conjunction with drum-type concentrators for sulphuric acid. A variety of carbon impervious to liquids under hydrostatic pressure is required for further possible applications.

E. A. RYDER.

**Ultra-violet radiation in industry.** M. J. DORCAS (Ind. Eng. Chem., 1930, 22, 1244—1246).—The useful application of ultra-violet radiation in industry depends on the correct choice of wave-length as well as on the use of robust apparatus in which the conversion of electrical into active light energy is as high as possible. An illustration is given of a carbon arc lamp, in which this conversion into light energy of wave-length shorter than 4000 Å. is about 12%. Numerous industrial uses of ultra-violet radiation are mentioned.

H. INGLESON.

**Gasworks' effluent.** INST. GAS ENG.—See II. Chlorinated diphenyl. PENNING.—See III. Helium. SNYDER and BOTTOMS.—See VII. Formation of cementite. PINGAULT. Copper-nickel alloys. SAGER. Metals from waste. HINCHLEY. Chromium-plating baths. STOUT and CAROL. Electrolytic chromium. SASAKI and SEKITO. Electrodeposition of chromium-iron alloy. FUSEYA and SASAKI. Thorium. DRIGGS and LILLIENDAHL. Radiography by  $\gamma$ -rays. BARRETT and others.—See X. Cataphoresis in nitrocellulose

solutions. LANTZ and PICKETT.—See XIII.  $p_H$  of tanning extracts. BURTON.—See XV.

## PATENTS.

**[Depolariser for use in Leclanché-type] electric batteries.** C. DILGER (HAMBURGER BATTERIE-FABR. C. DILGER) and D. SCHMIDT (B.P. 338,765, 16.12.29. Ger., 22.3.29).—A non-deliquescent double salt of ammonia or an amine and zinc or a metal placed above zinc in the electrochemical potential series, e.g., the double salt of zinc chloride and methylamine or *p*-toluidine hydrochloride, is used in conjunction with pyrolusite.

J. S. G. THOMAS.

**Photo-electric [electrolytic] cell.** ARCTURUS RADIO TUBE Co., Assecs. of S. RUBEN (B.P. 317,837, 23.8.29. U.S., 23.8.28).—An anode comprising a metal, e.g., copper, coated with a non-porous homogeneous integral layer of a compound of the same metal, e.g., cuprous oxide, and an aluminium cathode are immersed in a transparent electrolyte composed of an aqueous solution of haloid salts or cobalt chloride. Alternatively, a pure iron cathode and an electrolyte consisting of an aqueous solution of sodium carbonate may be used.

J. S. G. THOMAS.

**Graphite electrodes for use in selenium cells.** TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 337,691, 22.1.30. Ger., 13.2.29).—Colloidal graphite is deposited upon the etched portion of the surface of a heated glass plate.

J. S. G. THOMAS.

**Electrodes [cathodes] for electrolytic purposes [manufacture of per-salts by anodic oxidation].** S. G. S. DICKER. From N.V. ELEKTROCHEM. IND. (B.P. 318,154, 23.8.29).—The use of cathodes formed of alloys composed of at least one metal which catalytically decomposes the electrolyte, but is not corroded thereby, together with, if desired, one or more metals which individually do not catalytically decompose the electrolyte, but which are corroded thereby, is claimed. Thus alloys containing two or more of the metals nickel, chromium, iron, silver, aluminium, and tin may be employed. A suitable cathode for the electrolysis of potassium carbonate to produce potassium percarbonate is composed of V2A steel.

J. S. G. THOMAS.

**Cathodes for electric-discharge devices.** INTERNATIONAL GEN. ELECTRIC Co., INC., Assecs. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 309,831, 15.4.29. Ger., 13.4.28).—A refractory metal filament, e.g., of tungsten or molybdenum, is slowly heated to 2200—2300° in an evacuated enclosure containing thorium acetylacetonate vapour, so that the vapour is decomposed; thorium oxide and carbon from which thorium is generated being produced on the wire.

J. S. G. THOMAS.

**Electron-emitting cathodes for electric-discharge devices.** GRAMOPHONE Co., LTD., W. F. TEDHAM, and F. ELLINGTON (B.P. 339,057, 16.10.29).—An electron-emitting coating containing, e.g., barium or caesium is deposited upon a core or layer of aluminium having an oxidised surface. If desired, the layer of aluminium may be deposited on a tungsten core.

J. S. G. THOMAS.

**Preparation of electron-emission material for electric vacuum-tube devices.** WESTINGHOUSE

LAMP Co., Assees. of M. N. FREDENBURGH (B.P. 315,801, 18.7.29. U.S., 18.7.28).—An intimate mixture of alkaline-earth carbonates is prepared by adding a soluble carbonate, *e.g.*, ammonium carbonate, to a solution of soluble compounds of barium and strontium, more especially the hydroxides, at about 50°. J. S. G. THOMAS.

Collecting electrode for electrostatic precipitators. NAT. CARBON Co., INC. (B.P. 337,389, 29.7.29. U.S., 14.11.28).—Smooth cylindrical carbon chambers, preferably in the form of extruded tubes made of a 2:1 mixture of coke flour and tar and baked at 900–1400° are employed. J. S. G. THOMAS.

Apparatus for electrical precipitation of solid or liquid particles from gases. LODGE-COTTRELL, LTD., and L. LODGE (B.P. 337,642 and 338,739, 29.11.29).—(A) Apparatus comprising a pivoted or otherwise movable frame formed of vertical bars having laterally projecting brackets at intervals along their front and back faces, metal plates or louvres attached to the brackets for passage of deposited matter, a pivotal suspension situated at the upper end of the frame, and a bumping bar which, when struck endwise, causes the frame to swing, is claimed. (B) A discharge electrode comprising rigid, parallel, horizontal rods, tubes, or strips interconnected by suspensory links so that the electrode can readily be folded for transport and assume the vertical position when hung from its upper edge, is claimed.

J. S. G. THOMAS.

Apparatus for electrical precipitation of suspended material from gases. LODGE-COTTRELL, LTD. From INTERNAT. PRECIPITATION Co., INC. (B.P. 337,685 and 338,796, 14.1.30).—(A) A collecting electrode comprising a number of relatively light, closely spaced elements of semi-conducting material, *e.g.*, concrete, cement, or terra-cotta, through each of which a metallic conducting and supporting member extends centrally, and a common supporting means for all the elements, is claimed. Relatively small head room is required above the collecting electrode chambers for moving the electrodes. (B) Electrodes of, *e.g.*, cement, concrete, terra-cotta, formed in sections, which are strung or mounted on metallic suspension members extending within the electrodes, are claimed. J. S. G. THOMAS.

Manufacture of electrical insulating material. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 316,146, 23.7.29. Ger., 23.7.28).—Layers of thread composed of paper, cotton, etc. of polygonal or flat cross-section are wound helically and caused to adhere by impregnation with a suitable binder.

J. S. G. THOMAS.

Installations for the irradiation of substances with ultra-violet light. N.V. PHILIPS' GLOEILAMP-ENFABR. (B.P. 318,616, 8.8.29. Holl., 8.9.28).—An electric-discharge tube and a vessel containing the substance to be irradiated, *e.g.*, milk or water, are detachably secured to a casing, and means are provided for pressing the tube and vessel together to form an irradiation chamber which, except for apertures for the supply and discharge of material, is entirely closed.

J. S. G. THOMAS.

Apparatus for determination of the concentration of blood, milk, and other suspensions by

means of electric current. A. SLAWINSKI (B.P. 338,928, 1.6.29).—Two tubes having their axes respectively vertical and horizontal are filled with the suspension, inserted in two arms of a Wheatstone bridge, and the ratio of the electrical conductivities of the suspensions in the tubes is determined after suspended matter has been deposited. The height of sediment in the vertical tube is also determined. Apparatus for making the necessary measurements is claimed.

J. S. G. THOMAS.

Electric accumulators. SOC. ANON. DES ACCUMULATEURS-MONOPLAQUE (B.P. 337,375, 29.4.29. Fr., 5.2.29).

[Cooling of] electron-discharge devices [for high-power transmission]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of M. A. ACHESON (B.P. 339,559, 21.2.30. U.S., 21.2.29).

[Adjustable] apparatus for X-ray investigation. SIEMENS-REINIGER-VEIFA GES. F. MEDIZIN. TECHNIK M.B.H. (B.P. 339,212, 3.3.30. Ger., 1.3.29).

Grinding apparatus (U.S.P. 1,766,447). Heating of liquids (B.P. 338,597). Distillation (U.S.P. 1,768,179). Liquid seal for gas chambers (B.P. 338,827).—See I. Pitch-coking [for electrodes] (U.S.P. 1,756,234). Carbon black etc. (B.P. 336,261).—See II. Hydroxides etc. (B.P. 338,178). Gilding and silvering solutions (B.P. 338,383).—See VII. Chromium-iron alloys (B.P. 337,893). Iron and its magnetic alloys (B.P. 338,409). Magnetic alloy (U.S.P. 1,768,238). Lead from ores etc. (U.S.P. 1,759,494). Magnesium-covered calcium wire (U.S.P. 1,752,813). Uranium-molybdenum wires (U.S.P. 1,759,454). Copper sheets (U.S.P. 1,760,028). Chromium-plating bath (U.S.P. 1,754,075). Tin (B.P. 338,488). Aluminium (B.P. 337,995 and 338,084).—See X. Treatment of tobacco (U.S.P. 1,754,117).—See XX.

## XII.—FATS; OILS; WAXES.

Cacao butter. VI. Extracted or expressed fat? H. P. KAUFMANN (Chem. Umschau, 1930, 37, 305–311; cf. B., 1930, 673, 430).—Preliminary. The properties of the extracted fats from cacao shell and cacao germ are reviewed. These fats differ considerably from true expressed cacao fat, and should be classed as adulterants of the latter, although the extracted fat from cacao tailings, shells, etc., after refining, may be suitable for edible purposes. The presence of such fat in admixture with the normal pressed fat cannot be detected by examination of the usual chemical characteristics.

E. LEWKOWITSCH.

Influence of the component glycerides of the soap base in the production of a white [milled] toilet soap when dried in a Proctor dryer. G. F. CALEY (Chem. Eng. Min. Rev., 1930, 22, [Chem. Sect.], 472–473).—The transparent appearance, characteristic of milled soaps made from soap that has been prepared by sudden chilling in a cooler and dryer, can be avoided by careful control of the proportions of solid and liquid fatty acids in the soap stock. E. LEWKOWITSCH.

**Surface tension of alkaline soap solution.** S. OGURI (J. Soc. Chem. Ind., Japan, 1930, 33, 471—474 B).—An improved form of Millard's apparatus (cf. B., 1923, 938 A) is described. The effect of mild alkalis in lowering the surface tension of soap solutions (drop number in pure benzene) is shown graphically.

E. LEWKOWITSCH.

**Determination of the water content of fats and soaps by the distillation method.** J. DAVIDSOHN (Chem.-Ztg., 1930, 54, 934—935).—Very good results were obtained by use of the apparatus prescribed in the Wizöff and D.V.M. standard methods. Identical results were obtained by the use of xylene or petroleum spirit with a lanoline cream or lubricating oil; for soaps xylene is recommended, but in this case as in that of butter it is essential to add oleic acid (10 g. to 10-g. test sample) in order to recover the full amount of water present.

E. LEWKOWITSCH.

**Oleic acid rancidity of fats. III. Catalytic influences in rancidification. IV. Behaviour of the stereoisomeric pairs, oleic and elaidic acids, erucic and brassidic acids.** K. TÄUFEL ([III.] Z. angew. Chem., 1930, 43, 1108—1112. [IV.] Chem. Umschau, 1930, 37, 281—284).—III. [With J. MÜLLER.] Certain differences are found in the apparent relative effect of various catalysts on the rancidification of oleic acid and its ethyl ester, when the degree of auto-oxidation is measured (a), by the intensity of colour developed in the Kreis test, or (b) by the increase in weight. Quinol and copper acetate appear as strong antioxidants; salicylic acid (a), iron chloride, and, to some extent, phenol (a) are positive catalysts, the apparent negative action of iron oleate being explained by the formation and loss of volatile products in (b), and in (a) by the rapid production of interfering aldehydes. The effect of catalysts is more pronounced in the case of oxidation of the oleic ester (cf. A., 1930, 891).

IV. [With E. SPIEGELBERG.] The configuration of the unsaturated acids influences the rate of oxidative change (cf. Kuhn and Meyer, A., 1929, 152). The rate of development of rancidity in the acids exposed to the air was measured; no rancidity could be detected in the two elaidic acids, whether exposed in the light or in darkness. Rancidity developed fairly soon in the case of oleic acid, and less rapidly with erucic acid (weight increase only after 5 months; no change in the dark under 6 months).

E. LEWKOWITSCH.

**Manipulation of the Mackey test [for oleines].** H. WOLFF and R. HEILINGÖTTER (Chem. Umschau, 1930, 37, 284—286). STIEPEL (*Ibid.*, 326—327).—In order to exclude the extra heating effect due to the formation of metal soaps during the Mackey test, it is recommended that the gauze cylinder should be made of platinum or brass wire. The authors prefer to pack the oiled cotton in the cylinder up to a height of 4—5 cm. only. A sample of oleine (90% of free oleic acid), free from metal soaps, so examined using platinum and brass gauzes, showed temperatures of 99° and 109° (118° after 3 hrs.), respectively, after 2½ hrs.

STIEPEL states that any metal soap present in a commercial oleine should not be removed before testing. A period of 1½ hrs. for the test is considered too short,

and the use of a brass gauze is deprecated; a paper container for the oiled wool is recommended.

E. LEWKOWITSCH.

**Fatty oil from the seeds of *Couepia grandiflora*, Benth., one of the so-called oiticica oils.** J. VAN LOON and A. STEGER (Chem. Umschau, 1930, 37, 337—340).—The dark violet or brown "couflor" kernels yielded on extraction with light petroleum 57.7% (41.3% on the whole fruit) of a viscous, pale yellow oil with an odour resembling that of tung oil. On long keeping the oil became a white buttery fat, melting to a clear oil again at 51°. The residual red-violet meal had a poisonous action on mice. The extracted oil had  $d_4^{20}$  0.9440,  $n_D^{21}$  1.5184,  $n_D^{40}$  1.5090 (supercooled oil  $n_D^{20}$  1.5002), acid value 0.92, saponif. value 188.3, Reichert-Meißl value 0.56, true iodine value 231, unsaponifiable matter 0.39, insoluble fatty acids 93.85% (having true iodine value 243, acid value 196.7, mean mol. wt. 285.2, m.p. 70—78°,  $n_D^{20}$  1.4998). The saturated fatty acids (11.63% of the oil, by Bertram's separation) had saponif. value 210, m.p. 56—57.5°, and consisted principally of stearic and palmitic acids (35 : 65).

E. LEWKOWITSCH.

**Kōryō oil from *Andropogon sorghum*, var. *vulgaris*, Hack. (Manchu).** S. UENO and N. KUZEI (J. Soc. Chem. Ind., Japan, 1930, 33, 452—456 B).—Manchurian grain yielded 3% of a pleasant-smelling brown pasty oil, which could not be bleached with Japanese acid clay, and had  $d_4^{20}$  0.9206,  $n_D^{20}$  1.4659, acid value 40.0, saponif. value 183.0, iodine value (Wijs) 114.0, unsaponifiable matter 5.43% (iodine value 68.4). The yellow-brown fatty acids deposited a small amount of solid acids at 16°, and had saponif. value 197.0, iodine value 119.5°; the liquid fatty acids (71.6%), and solid acids (28.4%, m.p. 53.9—54.6°) separated by the lead salt-ether method, had, respectively, iodine value 120, 13.1; saponif. value 193.7, 215.3. After acetylation the unsaponifiable matter yielded about 7.95% of hydrocarbons (m.p. 60.5—61.8°), steryl acetates (m.p. about 64°), and 19.6% of a fraction crystallising at 31.5°, having acetyl value 104.2, and containing higher aliphatic alcohols. The unsaturated fatty acids consisted of oleic and linoleic acids (in proportions about 2:1) and the saturated acids of palmitic acid with minute amounts (?) of myristic and stearic acids.

E. LEWKOWITSCH.

**Pyrolysis of vegetable oils possessing high acetyl values.** R. DELABY and R. CHARONNAT (Compt. rend., 1930, 191, 1011—1012).—Vacuum pyrolysis of commercial specimens of oil of raisin pips yields only traces of unsaturated aldehydes and 20% of mixed fatty acids (mean mol. wt. 303—339). The oil does not contain appreciable quantities of ricinoleic acid, which under the same pyrolytic conditions gives heptaldehyde and undecenoic acid, or any unsaturated hydroxy-acid giving a saturated aldehyde and an unsaturated acid by pyrolysis. Pyrolysis of the sodium soap of castor oil gives sebacic acid, methylhexylcarbinol, and methyl hexyl ketone; similar treatment of oil of raisin pips gave only a trace of an alcohol yielding an oily hydrogen phthalate. Vacuum pyrolysis of oil from the seeds of *Hevea brasiliensis* affords 50% of a product giving by fractionation 1% of aldehydic substances,

b.p. 80—140°/14 mm., and a large proportion of fatty acids, b.p. 200—230°/14 mm., mean mol. wt. 312.

C. W. SHOPPEE.

**New compounds produced during hydrogenation of fish oils. II. Hydrocarbons. II (contd.).** S. UENO and R. YAMASAKI (J. Soc. Chem. Ind., Japan, 1930, 33, 451—452 B; cf. Ueno, B., 1930, 1036).—The volatile products of hydrogenation were saponified and extracted with methyl alcohol: the crude hydrocarbons were purified by treatment with acetic anhydride and sulphuric acid, and by fractionation. The analysis and m.p. (−5° to −9°) of a compound  $C_{19}H_{40}$  indicate a branched-chain structure. E. LEWKOWITSCH.

**Japanese beeswax. I. General properties.** H. IKUTA (J. Soc. Chem. Ind., Japan, 1930, 33, 467—471 B).—Analyses of Japanese beeswaxes from various districts and various strains of bee are given and discussed. The general conclusions are drawn that the honey plants have no effect on the constants of the wax, and that differentiation between typical Japanese and European beeswaxes (e.g., ratio numbers 10—14 and 3·8, iodine values 11—14 and 7—8·5, respectively) depends on the different species of the bee. The constants of beeswax are modified by the use of artificial comb. Perfect separation of the wax alcohols and hydrocarbons could not be effected by Ley's method. E. LEWKOWITSCH.

**Wax in rice polishings.** U. TANGE (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 14, 275—277).—The wax was isolated (yield 0·03%) by the extraction of the rice polishings with boiling 95% alcohol. After ether extraction to remove fat, the wax was dissolved in benzene and precipitated by adding methyl alcohol. The wax had m.p. 81·5—82·5°, and was identified by saponification as melissyl cerotate. Cerotic acid has m.p. 78—79°, melissyl alcohol 86—87°, and its acetyl derivative m.p. 71—72°. B. W. TOWN.

**Viscosity and flash point of oil mixtures.** KADMER.—See II. **Emulsifying agents.** BENNETT.—See III. **Pigments for linoleum.** BODENBINDER.—See XIII. **Tannery waste.** LLOYD.—See XV. **Detection of extraneous fats in milk and butter.** CAMILLA. **Quality of ice-cream.** LUCAS and others. **Butter-fat in ice-cream.** CROWE. **Detection of sesamé oil in arachis oil.** BUNCE.—See XIX. **Wool-scouring effluents.** KING; also SMITH.—See XXIII.

#### PATENTS.

**Heat-treatment of fatty oils.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 339,048, 8.10.29. Ger., 8.10.28).—Light fuel hydrocarbons are prepared by heating fatty oils at temperatures above the b.p. (e.g., 500°) and at pressures below 45 atm. in the presence of hydrogen and a contact substance consisting of reduced iron, copper, and/or zinc on a pumice support. Decomposition of the fatty acids and hydrogenation over the catalyst may be effected separately in successive stages. E. LEWKOWITSCH.

**Refining of vegetable oils to be used for medical purposes.** H. HATAKEYAMA and H. WATANABE (B.P. 339,011, 12.9.29).—Free glycerin and the bulk of the free fatty acid are removed from the oil by washing, and the remaining traces of free fatty acids are esterified

by heating at 60—70°, in a current of carbon dioxide, with about 3% of an aromatic alcohol, e.g., benzyl or cinnamyl alcohol, the excess of which is removed by vacuum distillation. The product is a neutral oil ( $p_H$  about 7) suitable for use as a solvent for medical injection. E. LEWKOWITSCH.

**Lubricant product and its production.** R. R. ROSENBAUM (U.S.P. 1,752,309, 1.4.30. Appl., 1.10.27).—Castor oil which has been rendered soluble in mineral oil by heating, in the presence of some 10% of mineral oil, at about 345° for 6—8 hrs. under a vacuum of about 20 in., or metal soap prepared from such treated castor oil, are used as base in admixture with mineral or fatty oils. E. LEWKOWITSCH.

**Manufacture of esters [of fatty acids with polyhydric alcohols].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,276, 4.7. and 30.10.29).—The component having the lower b.p. is vaporised (if desired, at reduced pressure) and the vapour, with or without addition of carrier gases, is led into the other component (heated above the b.p. of the former) in presence of catalysts and/or fillers and/or in a cycle in such quantity or at such speed as to convert the second component substantially into foam. Glycerin vapour at 230°/20 mm. is passed into olive oil acids; or olive oil containing 10% of free acid is allowed to trickle down a packed tower in countercurrent to glycerin vapour under reduced pressure; or hydrogen and glycerin vapour at 230°/20 mm. is bubbled upwards through a porous base-plate into olive oil acids; ethylene glycol and carbon dioxide at 230° are passed similarly through soya-bean oil acids. C. HOLLINS.

**Oil compositions etc.** (U.S.P. 1,752,945—6).—See II. **Cleaning composition** (U.S.P. 1,752,746).—See X. **Linoleum** (B.P. 338,965 and 338,978).—See XIII.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Industrial paints; successors of white lead and anti-rust paints.** A. VILA (Chim. et Ind., 1930, 24, 1052—1067).—The history of white pigments is discussed with special reference to the development of lead-free materials intended to replace white lead. A comprehensive official examination of such pigments available on the French market in comparison with white lead was undertaken in 1926, large-scale tests of spreading power, opacity, durability, etc. under different conditions of application being carried out at Bellevue. These tests are described at length, and the results, together with photomicrographs of weathered films and general illustrations of the painted buildings, are given. A further series of normal and accelerated weathering tests on various industrial anti-rust paints in comparison with red lead paints is also described. An accelerated test, according to which painted aluminium plates were treated with sea-water and mercuric chloride solution, was devised. It is considered, from the results of this work, that satisfactory substitutes for white and red lead are available. S. S. WOOLF.

**Irregular results of accelerated [paint] tests.** P. NETTMANN (Farben-Chem., 1930, 1, 103—104).—The composite nature of paint films is indicated and

an attempt is made to illustrate this by means of an equation. Surface forces and thickness of coat, and the effects of temperature on these, are further influences acting against the possibility of obtaining uniform results from accelerated tests, even if such factors as volume, shape, wettability, and degree of grinding of pigment could be standardised. S. S. WOOLF.

**Manufacture and evaluation of Schweinfurt-green.** A. THÜRMER (Farben-Chem., 1930, 1, 114—116).—The preparation and properties of Schweinfurt-green (copper arsenoacetate) are described. The characteristics of this pigment depend on purity and regularity of particle size, and details of its evaluation by chemical analysis and by sieving and settling tests are given. S. S. WOOLF.

**Precipitating agents used in colour-lake manufacture.** A. W. C. HARRISON (Farben-Chem., 1930, 1, 105—108).—An account is given of insoluble agents and mordants functioning by occlusion, *e.g.*, green earth, artificial iron silicate, alumina; of soluble metal salts (true precipitants), *e.g.*, calcium acetate, lead nitrate, barium chloride; and of special synthetic agents, *e.g.*, Tamol NN and Katanol. S. S. WOOLF.

**Cadmium pigments in cellulose lacquers.** G. SIBBERT (Farben-Ztg., 1930, 36, 506—507).—Cadmium sulphides and selenides have exceedingly good light-fastness and the reds are non-bleeding, but they lack the opacity and brilliance of shade necessary for use alone in automobile lacquers. They can, however, be used in conjunction with other pigments remedying both these defects. On exposure they have a tendency to "chalk." S. S. WOOLF.

**Requirements of pigments for the linoleum industry.** H. G. BODENBINDER (Farben-Chem., 1930, 2, 58—61).—The desirable qualities of linoleum pigments are fastness to light; resistance to cleaning agents, heat, and acid; freedom from oxidation catalysts and from soluble iron salts. Each of these is discussed and a general description of linoleum manufacture is given. S. S. WOOLF.

**Liquid absorption by pigments of different types of organic liquids.** I. Relation of liquid absorption with a series of organic liquids to interfacial tension of these liquids against water. F. E. BARTELL and A. HERSHBARGER (Ind. Eng. Chem., 1930, 22, 1304—1309).—The volumes have been measured of various organic liquids and of water absorbed by a series of pigments with which the liquids do not react. These liquid-absorption values of a series of liquids having zero angles of contact with polar solids increase linearly with increase in interfacial tension of the liquids against water, but decrease linearly with this interfacial tension when the non-polar solid, carbon, is used. When the air-dried powders are heated and evacuated the liquid-absorption values are practically the same as those obtained with the untreated powder. These values with a given solid and a given liquid are approximately proportional to the sp. surface of the powder. H. INGLESON.

**Matt varnishes.** H. DECKERT (Farben-Ztg., 1930, 36, 453, 504—505).—A general dissertation on the attain-

ing of matt effects by application of suitable varnishes, as opposed to processes involving the mechanical rubbing down of glossy surfaces. The theory of reduction of transparency is discussed and details of the preparation of matt varnishes by incorporation of waxes and by use of metallic compounds (particularly aluminium hydroxide and its compounds with fatty and resin acids) are given. S. S. WOOLF.

**Kinetics of the baking process of oil varnishes.** R. H. KIENLE (Ind. Eng. Chem., 1930, 22, 1370—1373).—The velocities of reaction of the baking process of a typical oil varnish at 275°, 295°, 315°, and 335° in atmospheres of air, oxygen, and nitrogen were determined in a special baking oven, details of which are given. Films baked for intervals of time representing all stages from "initial" to "completely deteriorated" were examined spectrophotometrically, the percentage of light of wavelength 6600 Å. reflected being used as a relative measure of degree of bake. From the data, reaction velocity isotherms are given, and temperature coefficients and heats of activation are also quoted. The slowest reaction proceeding in the baking process corresponds to a unimolecular reaction, but others undoubtedly occur. Industrial applications of the results are indicated, the possibility of calculating the time required for any industrial baking process from the results at other temperatures, and the significance of removal of vapours from the varnish film being discussed. S. S. WOOLF.

**"Blooming" of varnishes.** H. WOLFF (Farben-Ztg., 1930, 36, 505—506).—The results obtained by Krumbhaar (cf. B., 1931, 33) are stated to be in concordance with Wolff's own results, exception being taken, however, to the "crystal formation" postulated. With sufficient magnification, such "crystals" are seen to be a fine honey-combed surface pattern. The "blooming" tendency of a film is considered to be inversely proportional to its ability to swell, films with poor swelling power being permanently distorted by an adsorbed water layer, whilst others recover. This hypothesis is shown to correlate all the facts propounded by Krumbhaar, when a further type of "blooming," due to the rapid surface-swelling of films with high swelling power, is also admitted. S. S. WOOLF.

**Manufacture of rosin size.** F. CHEMNITZ (Chem.-Ztg., 1930, 54, 915).—A process in which rosin is treated in a steam-heated autoclave with a mixed solution containing sodium carbonate and sodium hydroxide is described. W. J. WRIGHT.

**Photo-activity of colophony.** G. NORKIN and B. SHADIN (Chem. Umschau, 1930, 37, 340—341).—Samples of Russian and American colophony exhibit photo-activity in a degree increasing with the surface area. Thus powdered rosin has a more marked action than small pieces, and the plate is unaffected by exposure to the surface of fused rosin. E. LEWKOWITSCH.

**Optical rotation of the resins.** I. Amber. II. Copals. III. Optical rotation as a criterion of purity of compressed amber. C. PLONAIT (Chem. Umschau, 1930, 37, 321—323, 323—326, 342—344).—I. The optical rotation of extracts of amber and copal can be used for discrimination, even when other methods fail. The powdered amber, mixed with ignited sand,

was extracted with ether, and the extract evaporated to dryness; the weighed residue was dissolved in ether or dioxan (0.2—1 g. per 10 c.c.) for polarimetric examination. All extracts from crude or compressed ambers were strongly dextrorotatory ( $[\alpha]_D +20.5 - 29.1^\circ$ ) in ether, benzene, alcohol, or dioxan.

II. [With S. LOEBMANN.] The rotations of copals varied according to their source; solutions in dioxan or Wolff's solution were prepared from the ether extracts of the gums, or by suspension. Of the African copals, Zanzibar copal had  $[\alpha]_D -25^\circ$  to  $-30^\circ$ , Congo copal  $-24^\circ$  to  $-34^\circ$ , Benguela  $-3^\circ$  to  $-21^\circ$ , whilst Angola copal had  $[\alpha]_D +11^\circ$  to  $+12^\circ$ , and  $-1^\circ$  to  $-2^\circ$ , Brazilian and Manila copals had  $[\alpha]_D +11^\circ$  to  $+23^\circ$  and  $+31^\circ$  to  $+53^\circ$ , respectively, East Indian  $+32^\circ$  to  $+37^\circ$ , and kauri copals varied from  $-1^\circ$  to  $+23^\circ$ . The copal oil and residues from cracking show rotations in the same direction as the respective original copals and in many cases of the same magnitude.

III. The proportion of copal present in a mixture with compressed amber cannot be estimated from solubility data. The optical rotations of dioxan extracts, however, afford a reliable criterion of the purity of ambers, since the values for mixtures of copal and amber lie between the figures  $[\alpha]_D -28^\circ$  to  $-34^\circ$  and  $+22^\circ$  for pure copals and amber, respectively. In many cases the quantitative composition of the mixture can be determined. E. LEWKOWITSCH.

**Phenol resinoids in oil varnishes.** V. H. TURKINGTON, R. C. SHUEY, and W. H. BUTLER (Ind. Eng. Chem., 1930, 22, 1177—1180).—Recently developed types of phenol condensation products are soluble in drying oils without the addition of rosin or any inactive ingredients. The drying times, hardness, durability, kauri reduction values, and alkali resistance of tung oil varnishes made on Bakelite XR254 and on ester gum, and of mixtures of these, were determined. The results, which are tabulated and graphed, show the beneficial effects of the phenol resinoid even when used in relatively small proportions. S. S. WOOLF.

**Chlorinated diphenyl.** PENNING.—See III. **Pulp industry by-products.** GRIFFITH. **Cataphoresis in nitrocellulose solutions.** LANTZ and PICKETT.—See V. **Corrosion of pipes.** INST. GAS ENG.—See X. **Tannery waste.** LLOYD.—See XV.

#### PATENTS.

**Production of protective coatings.** BRIT. CELANESE, LTD. (B.P. 316,984, 7.8.29. U.S., 7.8.28).—Surfaces containing cellulose acetate or other derivatives of cellulose are treated with a synthetic resin coating, e.g., of the phenol-aldehyde and/or -ketone type, which is converted subsequently into the insoluble and infusible state by known means. S. S. WOOLF.

**Coating especially adapted for filming tin and like foil.** STANIOLFABR. BURGDORF A.-G. (B.P. 338,340, 1.11.29. Switz., 9.10.29).—Such coatings comprise a solution of nitrocellulose in ethyl lactate and alcohol, an alcoholic shellac solution, and castor oil, amyl acetate being added if desired. S. S. WOOLF.

**Transfer compositions.** P. MEYER (B.P. 338,673, 11.10.29).—Compositions comprising water-soluble dyes and water-soluble soap, e.g., hard or grain soap, are

applied to paper, preferably by spraying, the proportion of dye being relatively large for carbon paper and small for single transfers. S. S. WOOLF.

**Transfer and composition therefor.** W. S. LAWRENCE, and KAUMAGRAPH Co. (B.P. 338,611, 27.8.29).—A marking composition comprising resinous material, a high percentage of an ethylated or benzylated cellulose derivative, a blown oil, and colouring matter is applied to a paper base, from which it is releasable under the action of heat, the materials being dissolved in solvents of high or low b.p. to permit the transfer to be printed on the base at high or low temperatures, respectively. S. S. WOOLF.

**Manufacture of pigments and surfaces coloured therewith.** SOC. CHEM. IND. IN BASLE (B.P. 317,763, 20.8.29. Switz., 21.8.28).—Sulphonated disazo and polyazo dyes (cf. B.P. 274,130; B., 1929, 317) which contain the residue of a diazotised 1:8-aminonaphthol-sulphonic acid as initial component, and the alkali salts of which yield a precipitate with calcium ions, are precipitated by interaction with sparingly soluble compounds of magnesium, an alkaline-earth metal, or a heavy metal, which contain the metal in a form capable of reaction. The products are sparingly soluble pigments, fast to light and to weather. Such pigments may be precipitated on a surface by spraying dye solution on the surface in which the metal compounds have been incorporated. The process is applicable to the colouring of stone, brickwork, etc. S. S. WOOLF.

**Nitrocellulose solvent and coating composition.** ATLAS POWDER Co., ASSEES. OF D. CARNEGIE, JUN. (B.P. 311,739, 15.5.29. U.S., 15.5.28).—Solvent mixtures containing not more than 25 vol.-% of a true solvent for nitrocellulose, e.g., ethyl, butyl, or amyl acetate, and 75% or over of a diluent comprising mixed petroleum hydrocarbons which evaporate at about the same rate as the solvent are claimed. [Stat. ref.] S. S. WOOLF.

**Finishing of surfaces with cellulose nitrate lacquers.** H. C. MOUGEY, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,753,616, 8.4.30. Appl., 29.7.25).—Adherent undercoats containing drying oil, gum, and pigment are applied to vehicle-body or other surfaces, and a main colour coating of pigmented cellulose nitrate lacquer is applied, the undercoats (when properly air-dried, or baked at  $40-260^\circ$ ) being only slightly softened by the lacquer. If desired, an additional final coating of lacquer containing a greater proportion of thinner than does the main colour coat may be applied. S. S. WOOLF.

**Paint and varnish remover, and its manufacture.** C. ELLIS, Assr. to CHADELOID CHEM. Co. (U.S.P. 1,759,154, 20.5.30. Appl., 11.1.26).—Compositions comprising wax, a protective colloid therefor, e.g., nitrocellulose and soap, a wax precipitant consisting of isopropyl alcohol with at least one fifth of its volume of acetone, and a wax solvent of the benzol series (acting also as a water eliminator) are claimed. Cracked isopropyl alcohol obtained by passing the constant-boiling mixture of isopropyl alcohol and water, containing a small percentage of *tert.*-butyl alcohol, through a cracking zone is preferred as wax precipitant. S. S. WOOLF.

**Manufacture of oxidised resin [colophony].** A. S. RAMAGE (U.S.P. 1,752,693, 1.4.30. Appl., 12.1.26).—Colophony in solution in a non-aromatic hydrocarbon of the olefine or naphthene type is subjected to the action of ozonised air. The oxidised resin is substantially insoluble in the original solvent, and may be precipitated by addition of further quantities of the latter or of the condensed volatile product recovered during the oxidising treatment. The precipitated resin is purified by dissolving it in an aromatic hydrocarbon, *e.g.*, benzene, and reprecipitating it in the cold with an aliphatic hydrocarbon, after which it is collected on a filter, washed, and dried. The product has m.p. 121°, and lower iodine and saponif. values than rosin.

S. S. WOOLF.

**Preparation of rosin solutions.** A. THIRIET and P. DELCROIX, Assrs. to PROC. NAVARRE (U.S.P. 1,759,526, 20.5.30. Appl., 21.6.26. Ger., 10.4.26).—A cold solution of caustic soda of concentration less than 10 g./litre is passed directly over roughly ground rosin in suitable apparatus, a solution for use in sizing paper etc. being thus obtained without the intermediate stage of making a concentrated rosin soap.

S. S. WOOLF.

**Production of resins.** J. M. WEISS (B.P. 338,845, 4.7.30. U.S., 10.7.29).—Substances of the type of coumarone and indene are polymerised in the presence of a polymerising agent comprising a mixture of an absorbent earth, *e.g.*, fuller's earth, and an acid sulphate of a tervalent metal, *e.g.*, iron. After washing the treated material with alkali, the unpolymerised naphtha is recovered by distillation, whereby a relatively pure resin of light colour is obtained.

S. S. WOOLF.

**Manufacture of synthetic resins.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. V. ADAMS (B.P. 310,854, 1.5.29. U.S., 1.5.28).—A polyhydric alcohol and a polybasic acid or its anhydride are condensed with an organic compound of a multivalent metal, *e.g.*, cobalt linoleate, aluminium stearate, or ferric lactate, until a resinous compound has been formed. The metallic constituent may be introduced by first combining a metal, *e.g.*, calcium, with either the alcohol or acid component and then completing the reaction by adding the remaining constituent. [Stat. ref.]

S. S. WOOLF.

**Manufacture of phenol-carbohydrate resin.** J. McINTOSH, Assr. to CONTINENTAL-DIAMOND FIBRE Co. (U.S.P. 1,753,030, 1.4.30. Appl., 22.1.24. Renewed 27.1.30).—Starch is condensed with a phenol in the presence of a catalyst, *e.g.*, sulphuric acid, and the intermediate condensation product is heat-cured in the presence of a hardening agent, *e.g.*, hexamethylene-tetramine, yielding hard, lustrous, infusible, insoluble resins.

S. S. WOOLF.

**Manufacture of artificial resins, and compositions and articles containing them.** BRIT. CELANESE, LTD. (B.P. 317,456, 16.8.29. U.S., 16.8.28).—Toluenesulphonamide is heated at 200–260° in the absence of water with an aldehyde or a compound containing a reactive methylene group, preferably in equimolecular proportions, and, if desired, 5–10% of urea on the weight of sulphonamide, and a plasticiser, *e.g.*, diphenylolpropane, xylylmethylsulphonamide, tri-

acetin. The product is suitable for use in conjunction with cellulose acetate or other cellulose derivative, in plastic masses, coating compositions, etc.

S. S. WOOLF.

**Liquid or plastic compositions comprising derivatives of cellulose and articles derived therefrom.** BRIT. CELANESE, LTD. (B.P. 317,454, 16.8.29. U.S., 16.8.28).—Compositions comprising cellulose acetate or other organic cellulose derivative, a synthetic resin condensation product of a toluenesulphonamide and an aldehyde in the presence or absence of a small proportion of urea, and one or more plasticisers, *e.g.*, triacetin, diphenylolpropane, xylylmethylsulphonamide, suitable for use in films, coatings, moulded articles, etc. are claimed.

S. S. WOOLF.

**Manufacture of plastic masses, threads, films, or coatings.** I. G. FARBENIND. A.-G. (B.P. 319,371, 21.9.29. Ger., 22.9.28).—Binding agents which are insoluble or only sparingly soluble in water, *e.g.*, natural or artificial resins, drying oils, rubber, or cellulose derivatives, are finely dispersed in water with the aid, if desired, of suitable emulsifying or wetting agents, *e.g.*, water-soluble colloids, and made into the required shape. The shaped mass is then treated with an agent capable of dissolving or swelling the dispersed binding agent, *e.g.*, a slow-drying nitrocellulose lacquer, a mixture of benzene and alcohol, etc., the shape being substantially retained.

S. S. WOOLF.

**Compositions comprising derivatives of cellulose and articles derived therefrom.** BRIT. CELANESE, LTD. (B.P. 317,457, 16.8.29. U.S., 16.8.28).—The use of cellulose acetate or other thermoplastic derivative of cellulose, together with 1–25% of its weight of synthetic resins obtainable by the condensation of aldehydic or ketonic substances with phenols, or of sulphonamides with aldehydes, in the production of artificial filaments, films, coatings, etc. is claimed.

S. S. WOOLF.

**Linoleum and its manufacture.** ARMSTRONG CORK Co., Assees. of E. CLAXTON (B.P. 338,965 and Addn. B.P. 338,978, [A] 29.8.29, [B] 30.8.29. U.S., [A] 15.10.28, [B] 8.8.29).—(A) An anti-coagulant, *e.g.*, rosin, is added to the oil in order to delay thickening, so that complete oxidation of the oil may be effected during the treatment in mechanical oxidisers (cf. B.P. 305,656; B., 1930, 1037). About 1% of boric acid, or certain organic or inorganic compounds, preferably antioxidants, are added during the later stages of linoleum manufacture, in order to accelerate coagulation of the oxidised oil during the stoving or calendering processes. (B) A coagulant with delayed action, *e.g.*, boric acid in a dehydrated form, preferably metaphoric acid, or urea, is added to the linoleum cement or mix.

E. LEWKOWITSCH.

**Flexible masses adapted for use as floor coverings.** ARMSTRONG CORK Co., Assees. of E. J. PFEFFER (B.P. 318,239, 20.7.29. U.S., 31.8.28).—The cement or binder, consisting of the product obtained by heating a polyhydric alcohol (*e.g.*, glycerol) and a polybasic acid or its anhydride (*e.g.*, phthalic anhydride) with a fatty acid (*e.g.*, tung oil fatty acids), is mixed in the fluid condition with fillers and thickened under the heating



conditions incident to the shaping operations; further stoving is unnecessary. E. LEWKOWITSCH.

**Floor-covering material and the like.** E. I. DU PONT DE NEMOURS & Co. (B.P. 317,375, 14.8.29. U.S., 14.8.28).—An impregnated felt base, a linoleum, or other suitable base material is coated with a paint, the vehicle of which is a modified polyhydric alcohol-polybasic acid resin containing 40–75% of oil, or oil or resin acids, *e.g.*, linseed or tung oil acids, rosin, etc. S. S. WOOLF.

**Manufacture of lead pigments.** SWISS INVENTIONS SYND., LTD., and A. V. BLOM (B.P. 339,357, 7.9.29).—See U.S.P. 1,736,066; B., 1930, 156.

**Machines for grinding the materials used in manufacture of printing ink, paints, etc.** H. SMITH (B.P. 339,482, 2.12.29).

**Printers' ink** (U.S.P. 1,767,089).—See I. **Solutions of cellulose derivatives** (B.P. 315,279). **Spinning of fibres** (B.P. 338,381).—See V. **Laminated glass** (U.S.P. 1,762,513).—See VIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Utilisation of waste rubber.** E. B. BUSENBURG (Inst. Chem. Eng., Dec., 1930, 56–58).—Fibre-free scrap rubber may be ground and used as a compounding ingredient, but is only a filler. Fibre, if present, may be removed mechanically by air separation, but this gives incomplete results. It may also be destroyed by heating with 15% sulphuric acid or by cooking under pressure with 6% caustic soda. The latter treatment plasticises the rubber at the same time and is the most widely used. Its drawback is that it is difficult to wash the rubber free from alkali, and its insulating qualities are therefore defective. The dried, plasticised scrap is blended and refined to crush the lumps, and metal is removed by straining. The refiners are special mills with rollers running at different speeds, a number working in series. Analyses of reclaimed rubber are given. It is not equal to crude rubber, as the original fillers are still present. It breaks down more rapidly than crude rubber during masticisation and helps the breakdown of the latter. C. IRWIN.

#### PATENTS.

**Manufacture of sheets or impregnations of rubber from rubber latex.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. B. F. F. CLARKE (B.P. 337,946, 21.10.29).—A gas-permeable vessel of any desired shape, fitted with a gas-inlet tube so that it can be filled with carbon dioxide under pressure, is immersed in latex which may have been compounded. A film of rubber becomes coagulated on the external surface. Material of any kind intended to be impregnated with rubber may be wrapped round the exterior of the gas-permeable vessel before immersion. D. F. TWISS.

**Vulcanisation accelerator.** H. LECHER and W. ZIESER, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,766,755, 24.6.30. Appl., 9.5.29. Ger., 18.5.28).—See B.P. 312,069; B., 1930, 959.

**Spinning of fibres** (B.P. 338,381).—See V. **Fibrous product** (B.P. 337,359).—See VI. **Plastic threads etc.** (B.P. 319,371).—See XIII.

#### XV.—LEATHER; GLUE.

**Some physical properties of leather.** II. BRADLEY (J. Soc. Leather Trades' Chem., 1930, 14, 516–523).—Absorption and desorption curves, both experimental and theoretical, for leathers are compared. The usual form of water-penetration test is considered to be of little value. The effect of temperature and the humidity of the atmosphere on the water-permeability of leather is discussed. Other factors examined, which affect the quality of the leather, are the diffusion constant, the water-immersion test, and the air-permeability. The vapour-adsorption and water-absorption constants of a leather are used to derive the adsorbing area of four vegetable-tanned curried leathers, the values for which vary from 44.8 to 84.6 cm.<sup>2</sup>/g. D. WOODROFFE.

**Improved methods of determining the wear resistance of leather.** U. J. THUVAU (J. Soc. Leather Trades' Chem., 1930, 14, 602–623; cf. B., 1929, 925).—A description is given of minor improvements in the author's wear-testing machine and of other types of machine for testing the wear-resistance and hardness of leather. The wear-resistance of a copper plate of known composition, which has been heated at 600° for 15 min., is taken as the standard (100) and leathers are compared with it. Comparative tests have been made on various leathers, using the different machines. The wear-resistance of sole leather was found to have increased by hammering, by precipitating the unfixed tanning material in the leather, and by rendering it waterproof. One sample (waterproofed) showed a wear-resistance greater than that of the best leather-substitute soles. The wear-resistance is also increased by swelling the pelt with acids or synthetic tannins prior to tannage. D. WOODROFFE.

**Biochemistry of soaking and liming [of animal skins].** VI. **Further effect of gaseous environment on soaking.** E. R. THEIS (J. Amer. Leather Chem. Assoc., 1930, 25, 442–460; cf. B., 1930, 627).—Calf-skin and goat-skin pieces were soaked, as before, under aerobic and anaerobic gaseous environment, respectively. More ammoniacal compounds were formed under aerobic than under anaerobic conditions, but less volatile fatty acids. The sulphur compounds in the skin were more affected under anaerobic conditions, which also caused considerable deamination. Curves are given showing the behaviour of both goat- and calf-skin when soaked in gaseous environments of hydrogen, oxygen, nitrogen, carbon dioxide, and air, respectively. D. WOODROFFE.

**Use of sulphide lime liquors in the sterilisation of dried hides infected with anthrax.** IV. **China hides.** (Miss) D. J. LLOYD and (Miss) M. E. ROBERTSON (J. Soc. Leather Trades' Chem., 1930, 14, 641–657).—Infected hides were treated with a lime liquor containing 1.6% Na<sub>2</sub>S.9H<sub>2</sub>O in 3% of lime at 37° for 8 days, after which no anthrax was found, but the hides were damaged. Good results have followed when the disinfecting solution was 1% Na<sub>2</sub>S.9H<sub>2</sub>O in slaked lime of 1.2% concentration, the hides being soaked for 8 hrs. at 37° and then for 4 days at the ordinary temperature. D. WOODROFFE.

**Standardisation of hide powder.** V. Calculation of "official" results from the data submitted by members of the Hide Powder Committee. H. G. BENNETT (J. Soc. Leather Trades' Chem., 1930, 14, 537—540; cf. B., 1930, 1165).—It is proposed that the Chauvenet criterion for rejecting abnormal results should be officially adopted for the work of the Hide Powder Committee. The probable error,  $p$ , of a single observation is given by  $p = 0.6745 \sqrt{\Sigma x^2 / (n + 1)}$ , where  $\Sigma x^2$  is the sum of the squares of the deviations from the mean. Values deviating from the mean by more than  $p$  should be rejected. D. WOODROFFE.

**Control of the  $p_H$  value of the official hide powder.** A. JAMET (J. Soc. Leather Trades' Chem., 1930, 14, 540—542).—The  $p_H$  value of the aqueous extract of hide powder is altered by filtration through powder. This is avoided by Meunier and Chambard's method (cf. B., 1926, 798), which is favoured by the author in preference to the Official International method. D. WOODROFFE.

**Application of the Atkin-Thompson method for acidity to vegetable-tanned calf leather.** H. B. MERRILL and R. G. HENRICH (J. Amer. Leather Chem. Assoc., 1930, 25, 511—518).—Three samples of leather, the weights of which form a geometrical progression, e.g., 1, 3, and 9 g., were each treated with 100 c.c. of 0.1N-potassium chloride, and after 24 hrs. each solution was filtered and the  $p_H$  value determined electrometrically. From the percentage of water in the leather, which was also determined, and the several sample weights, a dilution coefficient was calculated. The logarithms of the dilution coefficients were plotted against the  $p_H$  values obtained and from the straight line thus derived the  $p_H$  value was noted at zero dilution. Data are given for eight leathers tanned with vegetable tanning liquors containing sulphite-cellulose waste-liquors. The leathers gave Procter-Searle figures varying from 0.65 to 2.9% of free mineral acid, whilst the  $p_H$  values as determined by the above method varied from 2.90 to 5.00. D. WOODROFFE.

**Deterioration of vegetable-tanned [bookbinding] leather on storage.** R. F. INNES (J. Soc. Leather Trades' Chem., 1930, 14, 624—640).—From the analyses of a number of sound and rotted bookbinding leathers from libraries in industrial and non-industrial areas it is shown that the rotted leathers contained more free mineral acid than the sound portions from the same books, and that they all contained more than 3% of sulphuric acid, which must have been absorbed from the atmosphere. Several sound leathers were found which also contained > 3% of sulphuric acid, so that this is not the only factor causing decay. The rotting was more serious among leathers stored in industrial atmospheres. The deterioration was proportional to the amount of acid which had been absorbed by the leather, but it was also influenced by the nature of the tanning material. The fibres of the sound leathers were well separated, whereas those of the rotted leathers were badly separated. The grease content of some commercial bookbinding leathers was low (1.3—2.5%), and the degree of tannage 45—119. Most of the bookbinding leathers examined failed to comply with the recom-

mendations of the Society of Arts' Committee in respect to tanning material and the use of mineral acid.

D. WOODROFFE.

**Preservation of leather bookbindings.** R. W. FREY and F. P. VEITCH (U.S. Dept. Agric., 1930, Leaflet 69, 8 pp.).—The leather should be treated with a mixture of oils and greases or purified petroleum jelly, applied by hand. Various recipes are given. Leather bindings which have started to decay may be well oiled and subsequently treated with a cellulose nitrate lacquer free from gums or resins, but containing a high percentage of castor oil. After the application of a coat of oil dressing to a binding, it should remain in a warm place (38—45°) for 1—2 hrs. and the treatment should be repeated next day. D. WOODROFFE.

**Action of oxygen on tanning materials.** J. JÁNY (Collegium, 1930, 453—460).—A tan liquor, which contained much non-tans, after treatment with sulphur dioxide to reduce any quinones present, was found to lose none of its tanning properties. Further experiments with tan liquors, alone and in contact with hide powder, showed that the amount of oxygen absorbed by the tannins was the same, thus disproving the oxidation theory of tanning. The amount of oxygen absorbed by different tan liquors under normal conditions was very small, but there was a sudden increase at  $p_H$  9. The amount of oxygen absorbed by dextrose in tan liquors is negligible. D. WOODROFFE.

**Proposals for revising the regulations for quantitative tannin analysis.** F. STATHER (Collegium, 1930, 480—493).—The results are not affected by the position of the dishes in an electric drying oven or by the method of drying. More than 1 cm. in depth of sand is necessary in the Koch extractor to avoid clogging, and a depth of 2—3 cm. should be used. The amount of tannin per litre of infusion is very important, as the greater the tannin content of the infusion, the lower is the figure obtained in analysis. The prescribed method for dissolving liquid extracts is unnecessary, and the amount of insoluble matter remains the same if the extract is dissolved by heating it with ten times its volume of water. Equal results for the moisture are obtained by dissolving solid extracts in 30 c.c. of water in silver basins as by the proposed official method, but constant weight is attained more quickly by the latter method. The results for insoluble matter were not affected by varying the vacuum pressure in candle-filtration. All solutions should be filtered according to the regulations, even though they be optically clear, since many of the latter will give insoluble matter by such filtration. Solutions should remain 2 hrs. before analysis, since the tannin content obtained by analysis varies with changes in temperature. D. WOODROFFE.

**Report of the British Section Committee on the determination of the  $p_H$  values and copper contents of tanning extracts.** I. Determination of the  $p_H$  of tanning extracts. D. BURTON (J. Soc. Leather Trades' Chem., 1930, 14, 597—601).—Accurate results for non-sulphited extract liquors, e.g., chestnut, are obtainable with the bubbling hydrogen electrode method. The hydrogen should be purified by passing it successively through alkaline potassium permanganate solu-

tion, alkaline pyrogallol solution, 0.1*N*-silver nitrate, a cotton-wool spray trap, and distilled water. The glass electrodes should be cleaned with chromic acid before use. The body of the electrode should be filled with a phosphate buffer solution having  $p_H$  7—8. The difference in potential between the buffer and a solution of known  $p_H$  is measured and then the latter is replaced by the tan liquor. Necessary precautions are (i) the standard buffer solution should be freshly made every fortnight, (ii) the battery and accumulators used should not be allowed to run down, (iii) the outside of the glass electrode should be wiped dry before each determination; (iv) the apparatus should be kept in a dry room at 17—20°.

D. WOODROFFE.

**Measurement of the  $p_H$  of a pure chestnut extract by means of the quinhydrone electrode.** A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 567—573).—0.5 G. of quinhydrone is shaken for 10 min. with 50 c.c. of the tannin solution, which is then transferred to a stoppered electrode tube, provided with a side tube, rubber connexion, and clip on one side, and a siphon and side tube on the other. The siphon is placed with its end dipping into a saturated potassium chloride solution. The Poggendorf compensation electrometric method of determining the *E.M.F.* is used. The maximum error is  $p_H \pm 0.04$ . The  $p_H$  was only very slightly increased by dilution.

D. WOODROFFE.

**Problem of tannery waste.** (Miss) D. J. LLOYD (Inst. Chem. Eng., Dec., 1930, 45—48).—Whilst the hair of many skins is a by-product of high value, a solution of hair products in sulphide liquor is run to waste. A process has been tried for its recovery and use as fertiliser. Waste lime is usually an unsaleable by-product. It might be dried, aerated, and reburned in central plants. The fat attached to the skin usually goes to the glue manufacturer with the collagen, but in some cases it is pressed out or extracted with solvents and sold for soap-making. A very serious loss of tan occurs in the sludge deposited from natural tan liquors consisting chiefly of chebulic and ellagic acids. The former is a tan and soluble in hot water. The latter is insoluble and is a total waste. Spent tan residues are used to some extent as a packing agent in the white lead industry. It has been used for the manufacture of coarse wrapping paper, but is generally burned. Scrap leather has found no satisfactory application. A certain amount of scrap chrome-leather is treated for chrome recovery with alkali. Vegetable leather scrap has been distilled or treated with sulphuric acid and sold as fertiliser. The tallow used in currying is affected by the process, and can only be sold at a low price for soap manufacture. Spent tan liquors contain colloidal matter, and may require precipitation before they can be run into the sewers.

C. IRWIN.

**Production of glue from chrome-tanned leather waste by the chromium hydroxide process.** C. STIEPEL (Chem.-Ztg., 1930, 54, 894—895).—Chrome-leather waste is detanned by treatment with lime, which also provides the material for an intensive liming process. The alkalinity is adjusted, the product is boiled, and the glue thus obtained is recovered. The yield is twice that obtained from wet glue pieces and the quality is equal to that of good hide glues. The glue

can be recovered from the prepared leather waste in 24 hrs., which is quicker than any other process for the recovery of glue from chrome-leather.

D. WOODROFFE.

**Blue mineral colouring matter in "pure" gelatin.** A. L. BACHARACH and G. N. GRINLING (Analyst, 1930, 55, 566).—A sample of gelatin (not English) was found to contain a blue dye showing the characteristic reaction with hydrochloric acid of ultramarine.

D. G. HEWER.

**Determination of insoluble matter in tanning extracts.** [Report of International Committee on tannin analysis.] E. STIASNY (J. Amer. Leather Chem. Assoc., 1930, 25, 460—486, and J. Soc. Leather Trades' Chem., 1930, 14, 542—562).—See B., 1930, 159.

**Pulp-industry by-products.** GRIFFITH.—See V. **Dyeing of tanned leather.** KNOWLES.—See VI. **Wool-washing effluent.** SMITH.—See XXIII.

## PATENTS.

**Treatment of animal skins and pelts.** W. SAILER (B.P. 337,377, 30.4.29).—Concentrated sulphite-cellulose waste-liquors or ligninsulphonic acid are treated for 2 hrs. with compounds splitting off oxygen, e.g., perborates, persulphates, hydrogen peroxide, and then with 6—10% of chlorine and/or bromine; the product is used for tanning purposes.

D. WOODROFFE.

**Treatment of products having a basis of protein materials.** SOC. D'APPLICATIONS ET DE RECHERCHES SCIENTIFIQUES ET INDUSTRIELLES (B.P. 338,015, 25.11.29. Fr., 24.11.28).—Products having a protein base, e.g., ossein, albumin, casein, etc., are treated with the thiosulphate of an alkali or alkaline-earth metal in the presence of an acid or salt, e.g., alum, capable of decomposing the thiosulphate and liberating colloidal or finely-divided sulphur within the protein mass.

D. WOODROFFE.

**Treatment of leather, particularly for ore-conveyor belts.** A. McLENNAN (B.P. 338,536, 19.8.29).—Leather, preferably chrome-tanned, is immersed in a mixture of carbon disulphide and petrol for 24 hrs., dried, and impregnated with rubber solution. Its surface is then cleaned, buffed to raise the fibre, and treated with one or more coats of a solution containing gutta-percha, antimony sulphide, or other suitable cold-vulcanising agent, and carbon disulphide or carbon tetrachloride or mixtures of these.

D. WOODROFFE.

**Substitute for egg-yolk [for leather manufacture].** O. RÖHM (B.P. 337,524, 5.9.29. Ger., 6.12.28).—Hides are treated with 10% of their weight of a tanning liquor consisting of oil (20 pts.), sodium glycerophosphate (5 pts.), or a salt of one or more esters of di- or poly-hydric alcohols with phosphoric acid, except lecithins and phosphatides, gum arabic (2 pts.), a suitable oil emulsifier, e.g., methylcellulose, water (73 pts.), in addition to meal, alum, and salt.

D. WOODROFFE.

**Extraction of glue and gelatin from chrome-[tanned] leather.** BRIT. GLUES & CHEMICALS, LTD., and R. B. DREW (B.P. 338,584, 17.8.29).—An intimate mixture of chrome-leather waste and an excess of an alkaline-earth, e.g., magnesia or baryta, is placed on a grating in a digester and hot water is allowed to percolate repeatedly through it at a rate which enables

the liquor to dissolve sufficient alkali to maintain a  $p_H$  of 8–8.5. Alternatively, chrome-leather waste may be repeatedly extracted with a solution of an alkaline-earth having  $p_H$  8–8.5 at 90–105° until the latter is charged with glue or gelatin; the liquor is then drawn off. D. WOODROFFE.

**Apparatus for tanning skins, hides, etc.** E. G. WILSON (B.P. 339,252, 23.8.29).

**Proofing of leather** (B.P. 336,244).—See VI.

## XVI.—AGRICULTURE.

**Physical chemistry of cultivated soils. III. Adsorption and redox-potential. IV. Chemical energy in surface and subsoils.** R. A. HERZNER (Z. Pflanz. Düng., 1930, 18A, 249–264, 264–274; cf. B., 1930, 735).—III. For the measurement of reduction-oxidation (R-O) potentials, bare platinum electrodes are preferable.  $E_h$  potentials of water-saturated soils should be measured within 1 hr. of the preparation of the soil sample, since after 2–3 hrs. there is a steady decline in recorded values, reaching a limiting value in 6–10 days. Except in strongly acid soils R-O potentials vary with the  $p_H$  value. In acid soils variations in R-O potential and adsorption potential are of a similar nature. Numerically, the greatest  $E_h$  values were found in acid soils and the least in alkaline soils. The rate of oxidation in neutral and weakly alkaline soils is very great and decreases with increasing acidity. Electro-dialysis considerably alters the  $p_H$  and adsorption potential, but not the  $E_h$  of soils. In all soils examined  $E_h$  values were positive.

IV. High energy values of soils, as measured by R-O potentials and adsorption potentials, correspond with low humus contents, high capillary activity, and high dispersion. Considerable potential differences exist between soils and subsoils. Application to soils of artificial fertilisers alters their adsorption potentials, but has little effect on the R-O potential.  $E_h$  values of subsoils are practically constant owing to their small bacterial numbers. A. G. POLLARD.

**Distribution of iodine in some climatic soil types.** J. BECK and K. SCHLACHT (Z. Pflanz. Düng., 1930, 18A, 274–281).—The iodine content of soils decreases as podsolisation proceeds. In forest soils and podsols the iodine content is greatest in the (illuvial) B horizon, that of the  $A_1$  horizon being below that of the parent rock. A. G. POLLARD.

**Relation between various methods for determining the lime condition of soils and the  $p_H$  value and base-fixing zone.** S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1930, 18A, 281–291; cf. B., 1930, 1123).—Differences between the results of lime-requirement methods is ascribed to the fact that each records the lime necessary to produce in soil a  $p_H$  value within a specific zone. A. G. POLLARD.

**Rapid colorimetric method for determining the  $p_H$  of soils.** S. KÜHN (Z. Pflanz. Düng., 1930, 18A, 309–314).—Soil suspensions are quickly and effectively cleared by shaking with Merck's "barium sulphate pro-Röntgen," and the colorimetric method may be utilised satisfactorily. Results so obtained agree more closely with hydrogen electrode measurements than do those obtained with the quinhydrone electrode. A. G. POLLARD.

**Silicic acid content and the accuracy of chemical analysis of plants.** A. RIPPEL, G. BEHR, and H. WIANGKE (Z. Pflanz. Düng., 1930, 18A, 336–341).—During the ashing of highly silicious plant material (e.g., horsetail), silicates are formed (notably of calcium and magnesium) which are not broken up by evaporation with hydrochloric acid. By very cautious ignition this fixation is minimised, but not prevented. For accurate work decomposition with hydrofluoric acid is essential. A. G. POLLARD.

**Composition of grass from plots fertilised and grazed intensively in 1929.** J. G. ARCHIBALD (J. Agric. Res., 1930, 41, 491–501).—Analyses have been made of grass from nine 8-acre pasture plots under intensive manuring and grazing. The fertiliser treatment decreased considerably the dry-matter content of the grass and the crude fibre of the dry matter. All other constituents determined were increased, particularly nitrogen. Acre recovery of all nutrient constituents was increased, nitrogen being nearly double that recovered on control plots; the effect of withholding nitrogen from one plot was very marked. A comparison of results with climatic factors indicates that rainfall has most effect on seasonal fluctuations in grass composition. E. HOLMES.

**Wood carbolineum as an insecticide, from the physico-chemical viewpoint.** S. JENČIČ (Arh. Hemiju, 1930, 4, 176–194).—The insecticidal value of wood carbolineum used for spraying fruit trees is a function of the stability of the emulsions produced.

R. TRUSZKOWSKI.

**Tannery waste.** LLOYD.—See XV. **Tuberculous milk.** WOOD.—See XIX. **Wool-washing effluent.** SMITH.—See XXIII.

## PATENTS.

**Treatment of seeds to secure uniform germination.** WARREN-TEED SEED CO. (B.P. 337,601, 1.11.29. U.S., 15.11.28).—Loss of colour of seeds during the soaking process described in B.P. 335,102 (B., 1930, 1083) is prevented by the use of a colour-fixing solution, e.g., alum (1–10 g. per gal. of water) or copper sulphate, in which the seeds are soaked at 15.5–52°.

A. G. POLLARD.

**Production of a fertiliser containing urea-calcium nitrate.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 338,644, 16.9.29).—A mixture of practically anhydrous calcium nitrate and urea (e.g., 1:4 mols.) is melted at 148–150°; the product may be atomised hot, crushed when cold, or granulated, and its stability may be increased by spraying it with oils.

L. A. COLES.

**Cyanamides** (B.P. 338,023).—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Effect of sodium bisulphite on the polarising power of sugars and use of sodium bisulphite in sugar analysis.** Y. TOMODA and T. TAGUCHI (J. Soc. Chem. Ind., Japan, 1930, 33, 434–441 B).—According to the concentration, sodium bisulphite markedly lowers the polarising powers of dextrose, xylose, arabinose, galactose, and lactose, slightly lowers that of maltose, and scarcely affects those of mannose, sucrose, raffinose, dextrin, and levulose. The combination of dextrose

and sodium bisulphite does not follow the mass-action law exactly. As the polarising power of dextrose can be reduced to  $+0.5^\circ$  to  $-0.5^\circ$  V., using a 200-mm. tube, and that of laevulose is scarcely affected, a mixture can be analysed with the saccharimeter with an error of 0–6.5%. The same method is applicable to maltose–dextrose mixtures, honey, hydrolysed starch, and similar products.

L. J. HOOLEY.

**Commercial production of glucose.** A. E. WILLIAMS (Ind. Chem., 1930, 6, 495–499).—A suspension of starch in dilute hydrochloric acid is heated in a converter by steam under 50 lb. pressure. To lessen the time of conversion and to prevent undue formation of reversion products, the quantity of acid is reduced to a minimum and the pressure kept correspondingly high. In the preparation of liquid glucose the conversion is arrested at an early stage, but for solid and for crystal dextrose the dextrin content of the liquor is reduced to 5 and 2%, respectively. The liquor is almost neutralised by soda ash, decolorised by animal charcoal, filter-pressed, and 48% of the water content removed under vacuum in a Kestner falling-film evaporator. To obtain monohydrate and anhydrous crystals the syrup is seeded at 35 and 25°, respectively, with the corresponding crystals, whilst by cooling to 12° no seeding is required. The crystals are separated by centrifuging. Solid dextrose is prepared by concentrating the syrup in two stages and allowing it to solidify as a homogeneous mass.

C. RANKEN.

#### PATENT.

**Aliphatic acids by fermentation** (B.P. 337,153).—*See* XVIII.

### XVIII.—FERMENTATION INDUSTRIES.

**Brandies and related products.** H. ZELLNER (Chem.-Ztg., 1930, 54, 925–928).—A series of analyses is given.

C. RANKEN.

**Swedish barleys; their cultivation, improvement, and brewing value.** C. WIRTH (Woch. Brau., 1930, 47, 483–486, 495–498, 505–508).

#### PATENTS.

**Manufacture of yeast.** C. LANGEMEYER (B.P. 337,947, 22.10.29. Ger., 24.10.28).—The keeping qualities of yeast which has been separated after the usual fermentation process are improved by storing it for several hours in an aqueous solution of potassium salts and albuminous substances until it is completely saturated. It is then pressed and mixed.

C. RANKEN.

**Treatment of brewing yeast.** N. MARESCU (B.P. 320,021, 30.9.29. Fr., 28.9.28).—Brewing yeast is debittered and rendered suitable for the manufacture of goods by alternate treatment with acids and alkalis—in particular phosphoric acid and ammonia—the salts of which are assimilable by the yeast.

C. RANKEN.

**Manufacture of alcohol.** F. E. LICHTENTHAELER (U.S.P. 1,759,122, 20.5.30. Appl., 26.1.24).—The alcohol-containing waste gases and vapours from the fermenters and from the distillation part of the plant are led back into a scrubber down which flows the unfermented wort prior to entering the fermentation vessel.

C. RANKEN.

**Production of aliphatic acids by fermentation.** DISTILLERS Co., LTD., and H. B. HUTCHINSON (B.P.

337,153, 30.9.29).—Volatile fatty acids are produced by the fermentation of very dilute solutions of sugars by thermophilic cellulose-fermenting organisms. During fermentation sugar solution is added continuously or intermittently so that the concentration of the sugars is retained under that at which non-volatile acids accumulate. The optimum  $pH$  for the growth of the organisms is maintained by the addition of alkali, and, if necessary, nutrients may be added. The fermented wash is withdrawn intermittently or continuously so that any desired concentration of fatty acid salt may be maintained in the mash.

C. RANKEN.

**Production of peroxidases from vegetable substances.** W. SAILER (B.P. 337,405, 25.4.29).—Vegetable substances containing peroxidases are allowed to ferment for several days in water; to such substances are added oxides, hydroxides, carbonates, or metals which do not act in this case as catalysts. After filtration, the liquid is stabilised by the addition of a preserving agent such as alcohol, benzoic acid, etc.

C. RANKEN.

### XIX.—FOODS.

**Examination of milk for tubercle bacilli.** D. R. WOOD (Analyst, 1930, 55, 544–549).—The method of detecting tubercle bacilli in milk by microscopical examination, though expeditious, is not reliable, nor is the cytology of tuberculous milk always helpful. The only safe method to detect tuberculous animals appears to be to test the milk of the cows either individually or in groups.

D. G. HEWER.

**Some problems connected with milk.** C. J. H. STOCK (Analyst, 1930, 55, 535–543).—Experiments on the rising of the fat in milk show that on remaining undisturbed for 20 min. the fat in “fresh” milk begins to rise, the rate being quicker with cooled milk, and in 1 hr. 20 min. the rise is much more definite. Collins’ rule, which states that there is a diminution of 0.24% of fat for every hour by which the interval between milkings is lengthened, was tested, but the results did not support the theory, and the production of milk must be looked on as a function not subject to mathematical control. The accumulation of fat in the upper layers of milk left undisturbed is accompanied by a marked increase of solids-not-fat, and there was no indication that the highest proportion of solids-not-fat was in the bottom layers of such milk.

D. G. HEWER.

**Detection of extraneous fats in natural milk and butter.** S. CAMILLA (Annali Chim. Appl., 1930, 20, 527–534).—By a modification of Marchand’s method and using a simple lacto-butyrometer, exact values, comparable with those given by Gerber’s method, are obtainable for the proportion of fat in milk. If the ethereal solution of the fat thus separated from milk or butter is kept at 18–19° for 15–20 hrs., absence of adulteration may be presumed if the solution remains clear. Separation of solidified fat from the solution, even in minimum amount, indicates addition of at least 10% of margarine or butter substitute. Such adulteration may be confirmed by microscopical examination of the separated fat. Photomicrographs are reproduced.

T. H. POPE.

**Gelatin in cream.** O. A. MENDELSON (Analyst, 1930, 55, 567).—Stokes’ test (addition of acid mercuric

nitrate followed after filtration by cold saturated picric acid) cannot be accepted as conclusive for the presence of gelatin in cream, as degradation products of milk-protein give the same action; it is suggested that the Hopkins test for tryptophan should be applied to the precipitate, since gelatin does not contain this amino-acid.

D. G. HEWER.

**Influence of sugar and butter fat on the quality of ice cream.** P. S. LUCAS, T. MATSUI, and O. E. MOOK (Michigan Agric. Exp. Sta., 1930, Spec. Bull. 201).—Increased fat content within the range 8–14% improved the flavour, body, texture, and resistance to melting of ice cream. Mixtures with the lower fat content became coarse on storage. Increasing milk solids-not-fat (6–12%) improved texture and increased the acidity without affecting resistance to melting. With more than 12% of solids-not-fat the mixtures were "sandy."

A. G. POLLARD.

**Testing ice cream for butter fat.** L. K. CROWE (Nebraska Agric. Exp. Sta., 1930, Bull. 246).—Various modifications of the Babcock test examined were unsatisfactory. The ice cream is melted below 26.5° to avoid melting the fat and strained; 9 g. are placed in a 10%, 18-g. cream bottle, 5 c.c. of a mixture of 90 c.c. of *n*-butyl alcohol and 10 c.c. of ammonia solution are added, and the mixture is shaken. 30 C.c. of a 1:1 mixture of sulphuric acid ( $d$  1.82) and 95% alcohol are now added, and the mixture is again shaken till all curd dissolves. The bottle is heated for 15 min. in a water-bath at 79–82°, occasionally shaken, and centrifuged for 5 min. If the fat column does not reach the graduations, hot water is added (above 82°) and the tube shaken and centrifuged, placed in a water-bath at 57–60° for 5 min., and the fat volume read after the addition of "glymol."

A. G. POLLARD.

**Detection and determination of sesamé oil when mixed with other edible oils, with particular reference to arachis oil.** E. H. BUNCE (Analyst, 1930, 55, 567–568).—To a mixture of 5 g. of the oil and 5 c.c. of concentrated hydrochloric acid in a separator are added 4 drops of a 1% alcoholic solution of furfuraldehyde, and after shaking for 2 min. and setting aside for 3 min. the acid layer and washings are made up to 50 c.c. in a Nessler glass and the red colour is compared with similarly treated standards. By this means sesamé oil may be detected to the nearest 5% up to 40%.

D. G. HEWER.

**Inversion of sucrose in the preparation of fruit preserves.** S. BERLINGOZZI and M. LIGUORI (Annali Chim. Appl., 1930, 20, 501–521).—During the various operations involved in the making of fruit preserves, the  $p_H$  does not vary appreciably, even on addition of citric acid, which has no accelerating action on the inversion of the sucrose present. The pectic substances exert no protective effect on the separation of crystalline sugar from concentrated solutions, but they definitely retard the inversion at temperatures near 100°. Deposition of sugar crystals never occurs when the degree of inversion of the sucrose lies between 25 and 75%, but is almost always observed when this degree reaches 90% and when the fruit pulp used is of inferior quality and poor in pectin. A combined polarimetric and refractometric method is given for determining approxi-

mately and rapidly the degree of inversion of sucrose in preserves.

T. H. POPE.

**Analysis and food value of some unusual Philippine fruits.** A. D. FRANCISCO and P. J. WESTER (Philippine J. Sci., 1930, 43, 655–663).—Eighteen fruits, including five kinds of banana, are discussed.

**Helium.** SNYDER and BOTTOMS.—See VII. **Cacao butter.** KAUFMANN.—See XII. **Wax in rice polishings.** TANGE.—See XII. **Use of sodium bisulphite in sugar analysis.** TOMODA and TAGUCHI.—See XVII.

## PATENTS.

**Milling of flour.** W. W. TRIGGS. From VITIMIN MILLING CORP. (B.P. 337,496, 16.8.29).—Air is drawn through the eye of the upper stone of a buhr mill and thence between the grinding surfaces of the stones, in order to cool the flour during grinding. The ground material is raised to a bolting separator and the tailings are reground in a second buhr mill, the products of the second grinding being passed through the blower and intimately mixed with the ground material from the first mill.

E. B. HUGHES.

**Heat-treatment of flour and wheaten stock.** D. W. KENT-JONES, C. W. CHITTY, and WOODLANDS, LTD. (B.P. 338,603, 23.8.29 and 22.4.30).—Heat-treatment apparatus is described in which the reaction period is considerably reduced by bringing the flour (etc.) into intimate contact with the heating surface. After treatment the flour is immediately cooled by a counterflow air system to avoid condensation of moisture.

E. B. HUGHES.

**Increasing the percentage of moisture in corn, fruits, and other vegetable materials.** A. E. JONSSON (B.P. 338,635, 9.9.29).—In conditioning wheat and other cereals before grinding, the moisture content may be increased by placing the cereal and the required amount of water in an airtight vessel, evacuating, and vaporising water by heating to 30–45°.

E. B. HUGHES.

**Production of butter.** E. G. N. and (Mrs.) E. S. SALENUS (B.P. 337,778, 7.5.29).—Whole milk is centrifuged and the resulting cream subjected to different degrees of vacuum during its passage to a receiver.

E. B. HUGHES.

**Preservation of alimentary substances.** U. GIOMI (B.P. 338,768, 17.12.29).—The food is packed in an inert atmosphere (*e.g.*, nitrogen) in a suitable container after a pretreatment consisting in the case of fruits of immersion in alcohol and subsequent drying, and in the case of meat and vegetables of treatment with superheated steam in the container.

E. B. HUGHES.

**Preservation of materials of animal origin.** G. BEISSER (B.P. 338,011, 23.11.29. Ger., 29.7.29).—Methods of introducing preservative (brine having  $d$  1.16 or over) into the carcasses or parts of the animal after *rigor mortis* has set in are described.

E. B. HUGHES.

**Storing and transporting fruit.** F. KIDD and C. WEST (B.P. 337,422, 31.7.29).—The atmosphere surrounding fruit is automatically controlled by having containers fitted with a membrane of suitable selective permeability with respect to carbon dioxide, oxygen, etc.; *e.g.*, rubber is much more permeable to carbon



dioxide than to oxygen and nitrogen. As this membrane is also very permeable to ethylene, this may be used to accelerate ripening. E. B. HUGHES.

[Cooking]-oven thermometers. THERMOSTATICS, LTD., and F. W. MILLER (B.P. 339,397, 30.9.29).

[Dough-producing apparatus for] making bread and the like. J. E. WILSON, S. MCCONNELL, and C. BROWN (B.P. 339,006, 10.9.29).

Freezing of fish. STERILEX, LTD., and A. E. SHERMAN (B.P. 339,393, 25.9.29. Addn. to B.P. 328,396).

Method of rapid chilling, and freezing and defrosting for the transportation and preservation of food products. M. T. ZAROTSCHENZEFF (B.P. 339,172, 20.1.30. Fr., 16.2.29).

Screening apparatus (B.P. 338,608).—See I. Irradiation installations (B.P. 318,616). Determination of concentration of milk (B.P. 338,928).—See XI. Coatings for metal foils (B.P. 338,340).—See XIII. Egg-yolk substitute (B.P. 327,524).—See XV. Brewing yeast (B.P. 320,021).—See XVIII. Purine bases from vegetable substances (B.P. 338,057).—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pharmacological and chemical studies of the cause of the so-called ginger paralysis. M. I. SMITH and E. ELVOVE [with P. L. VALAER, JUN., W. H. FRAZIER, and G. E. MALLORY] (U.S. Public Health Service Reports, 1930, 45, 1703—1716. Reprint No. 1396).—Samples of tincture of ginger which had produced paralysis in man gave similar symptoms in rabbits and calves, but were non-toxic to monkeys and dogs. Concentrates contained no free phenols, but yielded on distillation or alkaline hydrolysis phosphoric acid and 1% of the original of phenols, not definitely identified. The toxic substance is therefore probably tolyl phosphate, and this view is confirmed by pharmacological tests with tinctures to which 2% of this product has been added. H. E. F. NOTTON.

Quantitative colorimetric reaction for ergot alkaloids and its application in the chemical standardisation of ergot preparations. M. I. SMITH (U.S. Public Health Reports, 1930, 45, 1466—1481. Reprint No. 1390).—A mixture of 2 c.c. of an aqueous solution of the alkaloidal tartrate and 1 c.c. of 0.16M-*p*-dimethylaminobenzaldehyde in concentrated sulphuric acid (cf. van Urk, A., 1929, 832) is exposed to sunlight for 15 min. or to diffuse daylight for 0.5—2 hrs. and the blue colour is compared with that obtained from a standard. The results obtained in the comparison of two solutions of the same alkaloid are equal to those obtained by biological methods, but, since within the limits of accuracy of the determination (5—10%) equal mols. of ergotamine, ergotoxine, ergotinine, and ergotamine give equal intensities of colour, the method may not indicate accurately the physiological activity of mixtures such as are present in fluid extract of ergot. In testing the last-named the ethereal extract of the concentrated basified aqueous solution is washed with ammonia to remove a yellow colouring matter, after which the alkaloids are extracted by 1% aqueous tartaric acid. The presence in the original extract of histamine, tyramine, ergosterol, choline, acetylcholine,

tryptophan, or skatole does not affect the reaction, but oxidising and reducing agents both interfere. The reaction is effected principally by light of wavelength 300—400 mμ. The influence of time and intensity of illumination on the reaction is shown graphically. The optimum proportions of acid and aldehyde are those given above; smaller amounts of acid may be compensated by more intense radiation, but larger amounts of either produce a brown coloration. H. E. F. NOTTON.

Determination of morphine in opium and its preparations. L. DÁVID and I. NOVÁK (Pharm. Ztg., 1930, 75, 1419—1420, 1433—1435).—The general method of treatment with calcium hydroxide (cf. Debourdeaux, B., 1910, 1225; 1911, 1029; 1913, 988, 1126) is preferred to the ammonia method (Dieterich, B., 1887, 148, 566, 609). It is not possible quantitatively to decompose solutions of morphine in calcium hydroxide by means of carbon dioxide; ammonium carbonate is satisfactory. The following method is recommended. Opium extract (0.2 g.) is ground with the slaked lime from 2 g. of pure calcium oxide and the mixture rinsed with a solution of lead acetate (0.32 g. in 30 c.c. of water) into a stoppered 100-c.c. flask; after 2 hrs., during which it is frequently shaken, the solution is filtered through a funnel of specified dimensions and porosity (Schott's, No. 11c G/3—7) and the filter-flask and funnel are washed with 30—40 c.c. of water. The clear filtrate is treated with 3 c.c. of ammonium carbonate solution (see below) and evaporated to dryness on the water-bath; the dry residue, after further heating to drive off ammonia, is taken up in alcohol (20 c.c.), bone-charcoal (4 g.) added, and the mixture heated to boiling. After ½ hr. (with shaking) the solution is filtered through a funnel (11 GH) surrounded by a hot-water funnel, and filtrate and washings are titrated with 0.1N-hydrochloric acid, using lacmoid as indicator. Opium powder is treated similarly, 0.4 g. being taken for analysis; tinctures (5 g.) are evaporated to dryness and the residue is treated as above. The ammonium carbonate reagent is prepared by dissolving 19.6 g. of ammonium carbonate crystals in 21.3 g. of 10% ammonia solution and diluting to 100 c.c. R. CHILD.

Determination of essential oil in camomile leaves (*Matricaria chamomilla*, L.). M. BERGMANN (Pharm. Zentr., 1930, 71, 785—789).—The D.A.B. VI method, in which 200 c.c. of distillate followed by a further 100 c.c. are obtained, gives low results, as the leaves are not completely extracted. A simplified method, in which 1000—1200 c.c. of distillate are collected and the final salting out is avoided, gives more satisfactory results. Many investigations under varying conditions are described. E. H. SHARPLES.

## PATENTS.

Manufacture of *N*-monoacylated diaminoarsenobenzenes. I. G. FARBERIND. A.-G. (B.P. 336,454, 1.11.29. Ger., 1.11.28).—Diaminoarsenobenzenes, e.g., the 3:3'-diamino-4:4'-dihydroxy- and 4:4'-diamino-3:3'-dihydroxy-compounds, are monoacylated by treatment with 1 equivalent of acylating agent (acetic anhydride, benzoyl chloride). C. HOLLINS.

Preparation of easily soluble and stable organic aluminium compounds (gluconates). CHEM. FABR.



VORM. SANDOZ (B.P. 336,922, 10.2.30. Ger., 15.2.29).—Calcium or barium gluconate is treated with aluminium sulphate solution, or gluconic acid with aqueous sodium aluminate. Aluminium gluconate, obtained by filtration and evaporation in a vacuum or by precipitation with methyl alcohol, is soluble, stable to ammonia, and non-hygroscopic.

C. HOLLINS.

**Production of purine bases from vegetable substances containing them.** G. B. ELLIS. From C. F. BOEHRINGER & SOEHNE (B.P. 338,057, 7.1.30).—Purine bases, especially theobromine and caffeine, and the fats which accompany them, are extracted from plant materials (tea, cocoa, etc.) with liquid sulphur dioxide either at normal or raised temperatures. The raw material may be pretreated with other reagents such as dilute alkali solutions before extraction.

E. H. SHARPLES.

**Separating the poisonous constituents of tobacco smoke.** H. PÄFFGEN (B.P. 338,006, 19.11.29).—Constituents such as nicotine, pyridine, etc. are removed by passing the smoke through dry absorption media (activated carbon, silica gel) which have been pretreated with smoke from the combustion of tobacco or with other aroma-imparting substances. Ethereal oils may also be added to the absorption media, and in the production or activation of the latter the following substances may be incorporated: sodium hydroxide, carbonate, or chloride, cane sugar, tannic acid, barbituric acid, sodium hyposulphite, ozone, or substances hindering the oxidation.

E. H. SHARPLES.

**Reducing the poisonous effect of nicotine [in cured tobacco], and the product thereof.** A. J. PACINI, Assr. to C. M. RICHTER (U.S.P. 1,754,117, 8.4.30. Appl., 3.5.29).—Cured tobacco is subjected to irradiation with infra-red or ultra-violet rays.

E. H. SHARPLES.

**Specific for cancer.** H. LEITNER (B.P. 337,797, 8.7.29).—The gall-bladders of animals, preferably those (such as the genus *Lepus*) that are immune from cancer, are freed from gall and then extracted, at normal temperature, with a water-soluble solvent such as alcohol, and/or ether. The solvent-free extract is then dissolved in an aqueous solution of a potassium compound such as potassium chloride and the solution is used for injection.

E. H. SHARPLES.

**Oils for medical purposes** (B.P. 339,011).—See XII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photo-activity of colophony.** NORKIN and SHADIN. —See XIII.

PATENT.

**Transparent films** (B.P. 336,353).—See V.

## XXII.—EXPLOSIVES; MATCHES.

**"Penthrinite"; Stettbacher's universal explosive.** P. NAOUM (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 442—446).—The author refutes Stettbacher's claims concerning the brisance and efficiency of penthrinite. No lead-block tests or determinations of velocity of detonation were made, and had the compression tests been carried out on samples of 100 g. and 40 mm. diam. instead of 30 g. and 25 mm. very different results would

have been obtained. The alleged slight loss in sensitiveness of blasting gelatine and dynamites on storage has no practical significance. Apart from the fact that blasting gelatine is not commonly used, and that fresh explosives are usually employed, the author's tests on explosives, after 3 months' storage, gave the following results: lead-block tests of blasting gelatine (3 compositions), 575, 605, and 605 c.c., respectively, before storage, and 525, 515, and 510 c.c. after storage; corresponding figures for a 30/70 mixture of nitroglycerin and nitropentaerythritol were 510, 510. Compression tests on the blasting gelatines gave 29, 27, and 26 mm. and 30, 29, and 29 mm. before and after storage, respectively, the nitroglycerin-nitropentaerythritols giving 28—30 mm. in each case. Penthrinite is not only too expensive for industrial use, but also gives inferior results to certain other explosives. Ammonium-penthrinite gave 420 c.c. expansion, and at  $d$  1.31 had a detonation velocity of 5900 m./sec. The corresponding figures for blasting gelatine ( $d$  1.6) and an ammonium nitrate-nitroglycerin mixture ( $d$  1.46) were 500, 8000 and 400, 6950, respectively. As regards sensitiveness to shock with a 2-kg. fall-hammer, the distances found were 15—20 cm. for three penthrinite compositions, 40 cm. for picric acid and tetryl, and 10 cm. for 65% dynamite, penthrinite being therefore quite unsuitable for military purposes.

W. J. WRIGHT.

**High brisance [of explosives] in relation to blasting efficiency and chemical constitution.**

**I. Tetranitromethane and its mixture with toluol.**

A. STETTBACHER (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 439—441).—The preparation of tetranitromethane from acetic anhydride and fuming nitric acid on a laboratory scale is described. The process requires 10—12 days for completion, and the yield is only 56% of the theoretical; the method is unsuitable for industrial application. Tetranitromethane has f.p. 13°; other properties are given. A mixture of tetranitromethane and toluol (100 : 15.66) gives a heat of explosion of 1701.5 kg.-cal./kg., or 2474 kg.-cal./litre. This mixture is not readily ignited by a flame or by sudden heating, and burns quietly. On the other hand, its sensitiveness to shock is greater than that of nitroglycerin. The brisance of a 100/20.9 mixture, as tested by exploding on iron plates, is greater than that of "gelatine-penthrinite."

W. J. WRIGHT.

**Determination of potassium perchlorate in ammonium nitrate explosives.** C. BUNGE (Chem.-Ztg., 1930, 54, 915).—100 C.c. of an aqueous extract are mixed with 20—25 g. of powdered quartz, the mixture is evaporated to dryness and ignited, and potassium is determined in an aqueous extract of the ash by the perchlorate method, a correction being made for any potassium chloride or nitrate originally present in the explosive. For rapidly determining perchlorate alone in explosives containing only small amounts of other potassium salts and nitroglycerin, 5 g. of the explosive are mixed with 20 g. of powdered quartz, and the mixture is heated in a quartz crucible. The ammonium salts are thus decomposed, and the aqueous extract of the ash is analysed as before.

W. J. WRIGHT.

**Nitration of cellulose.** PINCK.—See V.

**XXIII.—SANITATION; WATER PURIFICATION.****Effect of pollution contributed to streams.**

J. K. HOSKINS and H. R. CROHURST (Ind. Eng. Chem., 1930, 22, 1340—1342).—Data collected during a re-survey of the Ohio River last year between Cincinnati and Louisville when compared with that obtained in 1914—1916 indicates that the river at Cincinnati has improved during this period except in regard to the amount of available dissolved oxygen. The results of application of the biochemical oxygen-demand test, however, in interpreting the effect on this river of a sewered population of 600,000 may be so influenced by errors in stream-flow measurement and in the oxygen-consumed test itself or by the oxygen-demand contributed by the run-off from the watershed as to lead to entirely erroneous conclusions. The most accurate analysis of the chemical, hydrometric, and sewered population data will be obtained under conditions of minimum stream-flow as in dry summer months or in winter when dry frosty conditions prevail.

C. JEPSON.

**[Sewage-]sludge aeration. I. Rate of disappearance of oxygen in sludge.** E. J. THERIAULT and P. D. McNAMEE (Ind. Eng. Chem., 1930, 22, 1330—1336).—In a series of narrow channels about a mile long, the bottom of which was covered with pebbles to imitate a small stream, a very high rate of purification of grossly polluted liquors was observed. This led to laboratory experiments in which the rate of oxygen demand of sludges and liquors from various parts of the system was obtained by direct determination of the oxygen absorbed when the sample was continuously aerated in a closed system. It was found that the oxygenation of sewage sludge conforms closely to equations of the unimolecular type, and from which velocity coefficients indicating the respective rates of satisfaction of the "immediate" and "first-stage" oxygen demand of the sludge can be deduced. The "immediate" demand is shown to depend largely on the degree of staleness or putrescibility of the sample, and the large initial purification obtained when sewage is "floculated" with a well-aerated sludge is probably due to a rapid adsorption of the organic matters by the sludge and not to actual oxidation. The mat of well-aerated sludge on the bed of small streams would appear to be responsible for the high degree of apparent biological efficiency.

C. JEPSON.

**Comparative rates of stream purification under natural and controlled conditions.**

H. W. STREETER (Ind. Eng. Chem., 1930, 22, 1343—1346).—Rates of oxidation in a highly polluted stream, such as the Upper Illinois River, appear to be fairly similar to those observed in an artificial stream carrying a mixture of Ohio River water and sewage of approximately equivalent intensity of pollution. The specific rate of oxidation in the upper reaches of the river was found to be about three times that of the lower, and is apparently due to the presence of readily oxidisable matter producing an "immediate" oxygen demand and also the demand of oxidisable material previously deposited or adsorbed by the plankton growth. The apparent rate of reduction in the biochemical oxygen demand of the supernatant stream as measured by the decrease in demand at successive

stations does not appear to represent the true rate of deoxygenation, which is the key to an accurate measurement of the re-aeration capacity of the stream and depends on the combined demand of the matter in suspension and solution, of deposits in various stages of oxidation, and the "immediate" demand of the stream itself.

C. JEPSON.

**Control of microscopic organisms in public water supplies, with particular reference to New York City.** F. E. HALE (J. New Eng. Water Works' Assoc., 1930, 44, 361—385).—In using copper sulphate for this purpose in large supplies of water the chief difficulty is to obtain a fairly even mixture of the salt throughout the water. Various methods employed and results obtained are described and discussed. The normal dosage is 1 lb. per million gals., but this is sometimes based on the top 10, 20, or 30 ft., according to the position of the organisms. Experience has shown that the organisms do not in course of time develop the power to resist copper sulphate and that the small amount which remains in the water is not deleterious to health. The presence of micro-organisms in the supply may sometimes be avoided by varying the depth of the point of draught. Chlorine is an effective agent in the control of microscopic organisms, but sometimes results in the production of increased tastes due to the liberation of distasteful oil from the destroyed organisms. In India an excess amount of caustic lime has been successfully employed in controlling the amount of *Cyclops*, which, by acting as an intermediate host for embryos of the guinea-worm, causes disease in man.

C. JEPSON.

**Water purification. IV. Effects of certain modifications in coagulation-sedimentation on the bacterial efficiency of preliminary water treatment in connexion with rapid sand filtration.** H. W. STREETER (U.S. Public Health Reports, 1930, 45, 1521—1536, 1597—1623. Reprint No. 1392; cf. B., 1926, 966).—Studies carried out with a fully equipped plant of 160,000 gals. per day capacity have shown that (a) improved bacterial efficiency was obtained by increasing the period of sedimentation following coagulation up to a maximum of 12 hrs.; (b) between the values 5.6 and 6.9 any variation in  $p_H$  value had little effect on the efficiency of the coagulation process, but the efficiency improved slightly below  $p_H$  5.5 and diminished sharply above  $p_H$  7.0; (c) double-stage coagulation with two separate stages of sedimentation gave consistently better bacterial removal than the single-stage treatment, but, with the same total amount of coagulant and the same total period of sedimentation, similar results were obtained by single- and double-stage coagulation when conjoined with two separate sedimentation stages; (d) increased bacterial efficiency was obtained by increasing the amount of coagulant up to 5 grains/gal.

C. JEPSON.

**Water supply of Stuttgart and the use of activated carbon [in water purification].** E. LINX (Gas- u. Wasserfach, 1930, 73, 985—992, 1016—1022, 1038—1045).—About 60% of the water supply of Stuttgart is obtained from a source at a distance of 100 km. In order to provide an emergency supply in case of unforeseen interruption of the main supply

a new water works utilising surface water from a lake in the town is in course of construction. This will use the "A.D.M." process of water purification, which involves super-chlorination of the water and removal of the excess of chlorine by means of activated carbon (cf. Adler, B., 1929, 700). Extensive tests have shown this process to be preferable to other modifications of the chlorination process. The construction of the works is described in full. One of the other sources of supply is to be similarly modified. A. B. MANNING.

**Whitewater in paper and pulp mills and its utilisation.** R. J. MARX (Inst. Chem. Eng., Dec., 1930, 24—29).—Whitewater is the effluent from the passage of the watery suspension of fibre over a closely meshed wire in a paper machine. Owing to the large volume of water required and the good quality necessary, the re-use of the water as well as the recovery of the fibre etc. is desirable. Settling methods for fibre removal were used first, but these are mostly of low efficiency. An early power-driven machine employed an endless belt travelling over a wire-covered drum. The Dorr save-all consists of a vertical tank with horizontal trays connected close to the vertical shaft, which carries one scraper in each chamber. The Oliver save-all is a horizontal revolving cylinder with a wire screen in which the deposited fibre forms a filtering medium. It has oscillating rakes and works under suction. The author prefers a gravity settler, in which the water is allowed to fall as drops on to a distributing surface. The impacts cause the separation of entrained air from the fibre, which thereafter settles readily in a large conical tank with bottom-discharge for solids.

C. IRWIN.

**Industrial wastes.** J. B. C. KERSHAW (Inst. Chem. Eng., Dec., 1930, 3—8).—It is now usual for oil-burning ships to pass waste oily water through settling tanks either on the ship or on special barges; the oil separated usually contains 1—2% of water. The treatment and disposal of domestic waste products by municipalities is discussed. The waste boiler-flue gases from the Battersea power station are to be treated in three stages with sprays in contact with iron and steel surfaces to promote catalytic action. In the last stage Thames water containing 20 pts. of calcium carbonate per 100,000 will be used and hot dry air added at the chimney base. The consumption of water will be 25 tons per ton of coal burned, and the time of contact 25—44 sec. Centrifugal sprays of a copper-silicon alloy will be used. The cost of this treatment is estimated not to exceed 1s. 6d. per ton of coal burned. C. IRWIN.

**Treatment of suint liquors from wool scouring.** A. T. KING (Inst. Chem. Eng., Dec., 1930, 9—15).—The present tendency in wool scouring is towards a lesser use of soap and soda and a greater use of suint liquor freed from grease. In the Duhamel process the grease-laden liquor is pumped to machines of the cream separator type. Silt cannot, however, be completely settled out before centrifuging, which is consequently inefficient and troublesome. The relation between the capacity of a scouring bowl and flow of liquor for a grease content of  $\frac{4}{5}$  of the equilibrium quantity obtainable in infinite time is calculated and shown by curves. The Barber patent jet is a mixing device in

which one fluid is forced by tangentially arranged jets into a thin film of the other fluid. It has been adapted to the separation of wool grease from suint liquors by supplying its two chambers with greasy liquor and compressed air, respectively. Five sets of 10 jets are used successively, in place of centrifuges, and the greasy froth is washed away from the surface of the treated liquor to heating tanks. The efficiency of removal is about 50%, as with centrifugal treatment. The silt is largely denuded of grease, and the autoclaved product contains only 1—5% of water. The aeration prevents the putrefaction of crude liquor on keeping. The overflow liquors are cracked with sulphuric acid in a similar jet machine.

C. IRWIN.

**Treatment and disposal of wool-washing effluent.** B. A. SMITH (Inst. Chem. Eng., Dec., 1930, 16—23).—The acid-cracking method of treating wool-washing effluent yields a hot-pressed cake containing up to 17% of fatty acids. It is used for the manufacture of cheap lubricants, it may be distilled for oleins and stearins, or used as leather dressing. The efficiency is about 50%. The sud-cake is gradually freed from grease by weathering and may then be sold for manure. The washing of wool by organic solvents would give an excellent grease recovery, but the danger of fire has prevented its adoption. The best method for the centrifugal separation of grease is the use of the Adams machine. This is an ordinary disc-type separator with an extended bowl hood and four jets placed in the periphery. The liquor is previously passed through settling tanks and a small double-screen filter. It is then heated to the highest temperature possible without frothing. The mud being the densest substance present is thrown to the walls of the bowl and washed out through the jets, and the grease emulsion and mother-liquor are separated as usual. Such of these liquors as are run to waste will, in England, still require acid cracking before passing to the sewers. The product of centrifuging is an almost neutral grease with a widening field of application.

C. IRWIN.

**Wood waste for heating and power.** WANS.  
**Disposal of gasworks' effluent.** INST. GAS ENG.—See II. [Sulphite liquor from] pulp industry. GRIFFITH.—See V. Waste liquors. HOWARD.—See VII. Tannery waste. LLOYD.—See XV.

## PATENTS.

**Purification of air [containing ammonia].** A. L. DOTTER, ASSR. to MINE SAFETY APPLIANCES Co. (U.S.P. 1,767,057, 24.6.30. Appl., 26.3.28).—A respirator or other apparatus for absorbing ammonia is charged with a dry, inert, highly porous solid (e.g., charcoal) which has been treated (by soaking, draining, and washing) with a strong inorganic (preferably hydrochloric) acid. The treated charcoal is stated to absorb much more ammonia than the sum of the quantity that the untreated charcoal would absorb and that necessary to neutralise the acid.

B. M. VENABLES.

**[Base-exchange] water-softening apparatus.** S. A. KRÜGER (B.P. 339,510, 21.12.29).

**Treatment of alkaline waters** (U.S.P. 1,759,361).—See VII. Irradiation installations (B.P. 318,616).—See XI.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

FEB. 13 and 20, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Waste-heat boilers with horizontal settings [in gasworks].** E. S. DAVIES (Gas J., 1930, 192, 861—864).—Of the heat used for carbonisation, 17.8% is recovered from the waste gases in regenerators by pre-heating the secondary air, and a further 19% by a Spencer-Bonecourt Kirke waste-heat boiler. The "N.O.C." process, in which electricity, at a voltage of a few millivolts, is passed, at the rate of 0.1 kw. through a closed circuit in which the boiler acts as a conductor, is cheaper than the usual water-softening methods and keeps the boiler tubes free from scale. Details of a test on the boiler are given.

D. K. MOORE.

### **Heat transfer in ammonia condensers. III.**

A. P. KRATZ, H. J. MACINTIRE, and R. E. GOULD (Univ. Ill. Eng. Exp. Sta. Bull., 1930, No. 209, 50 pp.).—Results of tests of a horizontal shell-and-tube and a multitube-multipass type condenser are recorded. For given water velocities through the condenser tubes lower values of transfer coefficients are obtained when the identical shells are connected in series than when in parallel. The actual total capacity developed is a function of the amount of condensing surface, the limiting condensing pressure, the temperature, and the amount of water circulated; fouling of the tubes greatly reduces the coefficients. The general equation for heat flow in condensers may be used, under the conditions employed, to predict the coefficients (within 10%) for the multitube-multipass ammonia condenser.

CHEMICAL ABSTRACTS.

**Electrofiltration.** PURI.—See XVI.

PATENTS.

### **Furnaces. Blocks or tiles for furnace walls.**

BABCOCK & WILCOX, LTD. From FULLER-LEHIGH Co. (B.P. 339,052 and 339,055, [A] 10.8.29, [B] 11.8.29).—(A) A furnace for burning powdered anthracite or other fuel has the lower part of the front and back walls inclined to form a V, the sloping sides being kept cool by water tubes or other means. The finely-divided fuel is admitted downwards from the upper part and the air upwards through a chain-grate at the apex of the V at a velocity sufficient to maintain the fuel completely in suspension, even that part that slides down the walls. It is intended that the particles of ash should frit together until they are heavy enough to remain on the grate and be removed by it. (B) Blocks of refractory material are dove-tailed and cemented into metallic blocks which are shaped on the back to accommodate water-cooling tubes. B. M. VENABLES.

**Rotary tube furnaces [having removable stirrer arms].** T. LANG, Assee. of METALLGES. A.-G. (B.P.

319,335, 20.9.29. Ger., 20.9.28).—The stirrer arms, which are air-cooled, project through openings in the side of the furnace which are lined with metal having a higher thermal expansion than has the furnace masonry.

A. R. POWELL.

**Regulating the temperature of flowing reaction mixtures.** HOLZVERKOHLUNGS-IND. A.-G. (B.P. 339,144 and Addn. B.P. 339,148, [A] 30.12.29, [B] 31.12.29. Ger., [A] 18.1.29, [B] 2.11.29).—In an apparatus for exo- or endo-thermic reactions between constituents of a gaseous or fluid mixture, the reaction zone is situated on one side of a heat exchanger and the temperature is kept constant and reasonably uniform throughout the reaction zone by continuously circulating through the other side of the exchanger the mixture before reaction at a greater rate than the flow required for consumption. The minor quantity for the reaction is tapped off the circulation just before it leaves the heat exchanger and passes through the reaction side in a countercurrent direction, and a corresponding quantity of fresh mixture is drawn in at the suction side of the circulating pump. The temperature is regulated by the rate of circulation and by the provision of a heater or cooler in the main circuit outside the reaction exchanger. In (B) the process may be applied to gases under pressure.

B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of [A] W. LER. EMMET and [A, B] B. P. COULSON, JUN. (B.P. 319,220 and 339,237, [A] 17.9.29, [B] 14.6.30. U.S., [A] 17.9.28, [B] 14.6.29).—(A) In a mercury boiler a heating element is composed of a group of double-concentric return tubes arranged almost vertically, the lower closed ends of which are heated by radiation, but, owing to the increased pressure due to the head of mercury, ebullition takes place only in the upper parts which are heated entirely by convection. Actually the inner tubes are double-walled (making three concentric tubes in all) with a hermetically sealed dead space between; the outer annulus of mercury is maintained at a uniform small thickness by projections on the middle tube. Baffles and solid displacers are also placed in the vapour drum to reduce the quantity of mercury locked up. (B) In a boiler of the type described in (A), arrangements for leading liquid mercury into the inner down-tubes and for reducing the quantity of liquid mercury locked up are described. B. M. VENABLES.

### **Dryers [for sheet materials, e.g., wall-board].**

(A, B) COE MANUFG. Co., Assees. of A. J. VANCE. (C) A. E. WHITE. From COE MANUFG. Co. (B.P. 314,013, 314,982, and 337,432, 29.5.29. U.S., [A] 21.6.28, [B] 7.7.28).—The sheet material is caused to progress continuously through a casing once only in a longitudinal direction, but preferably in several independently

\* The remainder of this set of Abstracts will appear in next week's issue.

supported stories with heating means such as steam coils under each. Air is caused to circulate longitudinally through the casing and back outside it, (A) in one circuit, (B) in several substantially independent circulations each traversing part of the length; the flow may be con- and/or counter-current in the dryer and steam may be admitted in the earlier stages. Each supply of circulating air is split not only by twin fans (entry and exit then being to or from both sides), but also by guides, so that the number of layers of air is one more than the number of storeys of goods. The return conduits are preferably above the casing and the circulating fans situated at the withdrawal end so that the dryer is maintained under slight vacuum and the return conduit under slight pressure, the minor quantity of saturated air or steam being vented out near the fan. Patent (c) refers mainly to the construction of the casing of the dryer. B. M. VENABLES.

**Rotary drums for drying purposes.** DEUTS. BARCOCK & WILCOX DAMPFKESSEL-WERKE A.-G., and A. W. KRAMLOWSKI (B.P. 338,265, 12.9.29).—An elaborate system of lifting and spreading shelves is described for the interior of a rotary drum dryer.

B. M. VENABLES.

**[Gear- and chain-driven] drying or evaporating apparatus of the heated-roller type [for milk, foodstuffs, etc.].** A. ST. J. GATES (B.P. 334,355, 6.8.29).—The rollers are provided with sprockets which are driven by the same chain and with a further sprocket which is connected to a suitable source of power through a worm reduction gear. A. R. POWELL.

**Cooling agents.** N. CARO and A. R. FRANK (B.P. 339,194, 6.2.30. Ger., 8.2.29).—Aqueous ammonia is used as a cold-conveying agent in processes involving the liquefaction or freezing of nitrogen oxides; the solution may contain also f.-p. depressants, *e.g.*, ammonium salts, metal nitrates or thiocyanates.

B. M. VENABLES.

**Grinder.** R. PRATER (U.S.P. 1,768,713, 1.7.30. Appl., 17.3.27).—The apparatus is suitable for "cracking" corn or similar purposes. It comprises a number of beaters increasing in diameter away from the feed end and driven by a horizontal shaft. The lower portion of the casing is perforated and conical, the upper portion cylindrical and imperforate. B. M. VENABLES.

**Mixing and/or grinding or crushing machines.** G. H. and P. S. HOWSE (B.P. 338,232, 23.8.29).—The apparatus comprises a rotating drum in which are suspended, loosely but in such a manner that their axes are always in line with the drum, a number of rollers which make contact with the lower part of the circumference of the drum or the material thereon. The rollers may be in sections and some or all helical so that good longitudinal mixing is produced.

B. M. VENABLES.

**Disintegrating, tearing, kneading, mixing, drying, or like machines.** DEBERAG DEUTS. BERATUNGSGES. F. DIE CHEM. U. METALLURG. IND. M.B.H. (B.P. 338,316, 19.10.29. Ger., 19.10.28).—The apparatus comprises a cylindrical casing provided with an inlet at one end and an outlet at the other so that a continuous process may be maintained, and, if desired, with a jacket for heating or cooling. A number of radial arms are

freely mounted on an axial rod within the casing and these are driven by a cylindrical cage-like member, which is rotated by power about an axis which is eccentric to that of the casing and preferably below it, the arms being threaded through suitable holes in the cage. The result is that the radial stirrers rotate at different speeds at different points (faster at the top) and the material is also squeezed between the cage and the casing and is prevented from sticking to the arms by the scraping action of the cage on them. Longitudinal motion may be produced by helical grooves on suitable surfaces.

B. M. VENABLES.

**Mixing and blending systems.** FULLER Co., Assees. of J. H. MORROW (B.P. 338,123, 8.8.29. U.S., 3.10.28).—Pulverulent materials are delivered, preferably by the Fuller-Kinyon air-borne conveying system, to a number of silos in rapid succession so that a number of thin layers are formed in each, and then the material is withdrawn from several silos or several layers simultaneously. The intervals and sequence of the transfer may be electrically controlled. B. M. VENABLES.

**Mixing apparatus.** J. A. PICKARD (B.P. 338,945, 1.8.29).—The apparatus is suitable for mixing powder or other material into a stream of liquid. The main conduit is provided with a stopcock across which is a by-pass. The by-pass route embodies isolating cocks and a feeding chamber, which is charged with powder when the cocks are closed and through which the liquid subsequently passes in an upward direction, the withdrawal of solid being observable through a window. There is also a sighting device in the main stream after the by-pass rejoins.

B. M. VENABLES.

**Mixing of gaseous fluids and solid particles.** BARCOCK & WILCOX, LTD. From FULLER-LEHIGH Co. (B.P. 339,056, 11.10.29).—The device is for destroying stratification in a stream of gas carrying solid particles, so that it may be divided into a number of streams of identical composition. The supply conduit of round or other ordinary section is changed to narrow rectangular and again to narrow rectangular at right angles to the first, all three cross-sectional areas being about equal. The subdividing means, *e.g.*, hinged flaps and separate conduits for each stream, are situated at the second rectangular section. B. M. VENABLES.

**Agitator, aerator, and mixer.** W. N. JONES and C. C. HUMPHREYS, Assrs. to ROTEX ENG. Co. (U.S.P. 1,765,338, 17.6.30. Appl., 14.11.28).—The apparatus comprises a unit, one or more of which may be stood in a tank of liquid. A motor at the top drives a propeller at the bottom by means of a vertical shaft. The propeller rotates within cut-out portions of vertical radial baffles. When aeration is desired the speed is raised sufficiently to break the surface of the liquid and air is drawn in through small pipes extending close to the blades of the propeller. B. M. VENABLES.

**Determining the degree of saturation of materials which have been immersed in liquids.** W. & T. AVERY, LTD., W. A. BENTON, and P. E. GOFF (B.P. 338,282, 26.9.29).—In a mass of material under treatment, *e.g.*, barley steeping in water, are embedded a number of frusto-conical baskets containing samples of

the material which are adapted to be placed in a centrifuge and then upon a weighing machine. If the baskets are adjusted to constant tare and a constant weight of dry sample is taken, the scale of the weighing machine may read directly in percentage of moisture.

B. M. VENABLES.

**Apparatus for expressing liquids from solids.** J. CRICHTON & Co., LTD., and J. CRICHTON (B.P. 338,850, 20.6.29).—The cylindrical wall of a press is formed of a helical band, the outlet for fluid being between the turns.

B. M. VENABLES.

**Preparation of adsorbent materials.** H. E. BIERCE (U.S.P. 1,752,721, 1.4.30. Appl., 9.5.28).—Clay containing 10–20% of moisture, and crushed to a fineness of from  $\frac{1}{2}$  in. to 200-mesh, is mixed with an organic acid, *e.g.*, oxalic acid, and heated to 90–120°, the product being pulverised. The addition of 1, 2.5, and 5% of oxalic acid to raw standard Utah clay increased its decolorising efficiency (when tested on a lubricating oil) from 100 to 108, 139, and 155%, respectively.

W. J. WRIGHT.

**Effecting continuous discharge of solid matters from centrifugal bowls with unperforated bowl wall.** AKTIEB. SEPARATOR (B.P. 339,566, 28.2.30. Swed., 4.3.29).—Mercury is placed in the bowl and forms a layer which prevents sticking of the sludge. The heavier liquid may be relied on to discharge the sludge, or a carrier liquid may be supplied through special channels in such a way as to swirl past the entrances of the discharge channels and to sweep along the surface of the mercury in an axial direction.

B. M. VENABLES.

**Sterilisation or like heat treatment of liquids.** BERGEDORFER EISENWERK A.-G. (B.P. 338,422, 9.1.30. Ger., 9.1.29).—In apparatus of the type in which the liquid (*e.g.*, milk) is heated and cooled by heat exchange with itself, external heat being supplied only to the high-temperature stage, the heating side of the first stage is preferably of the trickle type to facilitate removal of bad gases, whereas the other stages are of the closed-tube type. In any case several stages are used with means to by-pass one or more. Devices for separating or filtering may be connected between heat-exchange stages.

B. M. VENABLES.

**Continuous distillation.** N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,756,032, 29.4.30. Appl., 8.6.22. Renewed 17.5.28).—A series of still and rectifier units are operated at increasing temperatures; when required, *e.g.*, for the production of lubricating fractions from petroleum, the temperatures may be kept low by means of vacuum pumps. Each unit comprises a coil heater, rectification tower, and condenser for the lighter fractions, the last-named being collected in parallel. The discharges from the bottoms of the towers are reheated and passed mostly to the (respective) next tower, but some of the reheated oil is sent back to the lower part of the tower from which it came in order to regulate the temperature. Independent means for heating at the bottoms and for partial condensation at the top may be provided.

B. M. VENABLES.

**Vapour outlet for stills.** R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,767,196, 24.6.30. Appl.,

22.5.24).—The vapour is admitted to an internal outlet pipe through a longitudinal slit on the upper circumference; the slit is provided with lips leading to internal plates which catch moisture and lead it to the bottom of the outlet pipe, whence it drains back into the still.

B. M. VENABLES.

**Distributors for gases carrying solids in suspension.** BARCOCK & WILCOX, LTD. From FULLER-LEHIGH Co. (B.P. 338,327, 26.10.29).—A stream of, *e.g.*, air-borne coal dust is divided into several streams of even composition by passing it first through a bend which has a flat surface on the outside, which acts as a mixer, then through a fan-shaped piece (of conduit) to form a wide thin stream, after which it is divided by a number of oppositely tapered, narrower, fan-shaped pieces which lead the individual streams to round pipes.

B. M. VENABLES.

**Filtering apparatus for separating fibre and dust from air.** R. and J. GREENHALGH (B.P. 339,514, 24.12.29).—The apparatus comprises a number of bag filters with fixed dust-receiving chambers below and movable inlet headers above. The filters are divided into groups which can be shaken in turn by jarring the inlet header of a group; the group headers are connected to a main inlet header by means of collapsible pipes which are closed by pressing their sides together when shaking takes place.

B. M. VENABLES.

**Treatment of exhaust gases of internal-combustion engines to render them innocuous.** L. GAISMAN and S. J. PEACHEY (B.P. 338,933, 24.7.29 and 19.5.30).—Air under a sufficient pressure is admitted to the exhaust pipe and the mixture passed through a silencer or other chamber containing a catalyst able to cause oxidation of carbon monoxide, *e.g.*, a combination of nickel wire-gauze discs, palladised pumice, and asbestos, or a worm covered with a palladised material.

B. M. VENABLES.

**Dispersal of fog or mist.** H. A. LLOYD and F. A. WRIGHT (B.P. 339,295, 5.6.29).—A stream of heated air is produced by an electric fan surrounded by electric heaters.

B. M. VENABLES.

**Manufacture of vessels and other structures that are proof against chemical and atmospheric influences.** P. PICK (B.P. 339,320, 4.7.29. Ger., 23.7.28).—Hollow vessels are constructed from the elements described in B.P. 316,134 (B., 1931, 3). The edges of the metal are bared and joined by known means, and the bare joints are re-covered with the plastic material as before.

B. M. VENABLES.

**[Saddle-shaped] filling bodies for reaction and washing towers and the like.** E. BERL (B.P. 319,239, 30.8.29. Ger., 18.9.28).—The bodies are in the shape of a saddle having its breadth equal to its depth and its ends projecting slightly outwards.

A. R. POWELL.

**[Hollow cylindrical] filling bodies for use in distilling columns, absorption towers, reaction vessels, etc.** F. WEINREB and C. H. BUTCHER (B.P. 335,889, 28.6.29).—Both ends of the cylinders are rounded and smooth.

A. R. POWELL.

**Effecting heat exchange.** W. D. LA MONT and A. F. ERNST, Assrs. to LA MONT CORP. (U.S.P. 1,783,724,

2.12.30. Appl., 7.10.26).—See B.P. 278,704; B., 1929, 495.

**Apparatus and processes for treating materials.** C. W. VOGT, ASSR. to VOGT INSTANT FREEZERS, INC. (U.S.P. 1,783,864—6, 2.12.30. Appl., [A] 1.11.28, [B] 17.3.28, [C] 31.12.28).—See B.P. 330,887 and 332,577; B., 1930, 838, 887.

**Mixing apparatus.** A. B. and C. R. SMITH (U.S.P. 1,782,795, 25.11.30. Appl., 7.9.29. U.K., 22.3.29).—See B.P. 328,401; B., 1930, 645.

**Centrifugal apparatus.** R. M. BERLINE, ASSR. to SHARPLES SPECIALTY CO. (U.S.P. 1,784,510, 9.12.30. Appl., 7.5.29. Luxemb., 7.5.28).—See B.P. 311,248; B., 1930, 886.

**Filtering apparatus.** E. W. W. KEENE (U.S.P. 1,783,937, 2.12.30. Appl., 1.3.30. U.K., 8.11.28).—See B.P. 326,221; B., 1930, 444.

**Production of adsorbents [from flue dust].** H. B. RÜDER, ASSR. to LURGI-GES. F. WÄRMETECHNIK M.B.H. (U.S.P. 1,784,543, 9.12.30. Appl., 22.10.27. Ger., 11.11.26).—See B.P. 280,505; B., 1929, 8.

**Regeneration of adsorption material.** V. PANTENBURG, ASSR. to AMER. LURGI CORP. (U.S.P. 1,784,536, 9.12.30. Appl., 23.7.25. Ger., 12.11.24).—See B.P. 242,986; B., 1927, 287.

**[Boiler] furnaces.** JOHN THOMPSON WATER TUBE BOILERS, LTD., and H. E. PARTRIDGE (B.P. 339,374, 16.9.29).

**Operation of furnaces, particularly steam boilers.** I. ARBATSKY (B.P. 338,174, 10.6.29).

**Apparatus for removing sludge from water blown down from steam boilers.** FILTRATORS, LTD., and V. V. SAKS (B.P. 302,942 and 312,332, [A] 22.6.27, [B] 24.5.29).

**Refrigerating plants.** A.-G. BROWN, BOVERI, & CIE. (B.P. 318,597, 6.9.29. Ger., 6.9.28).

**Method of cooling and condensing in periodically working absorption refrigerating plants.** DEUTS. GASLÜHLICHT-AUER-GES. M.B.H. (B.P. 313,890, 18.6.29. Ger., 18.6.28).

**Subjecting materials to indirect heat** (U.S.P. 1,763,758).—See II. **Atomisers for liquids** (B.P. 324,565).—See VII. **Pulverulent products** (B.P. 338,463).—See IX. **Metallurgical furnaces** (B.P. 338,893).—See X. **Precipitation from gases** (B.P. 338,427 and 338,944; U.S.P. 1,766,421—2, 1,767,265, and 1,767,338).—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Coke breeze.** E. W. SMITH (J. Inst. Fuel, 1930, 4, 113—116).—The quantity of coke breeze formed in gasworks is greater with continuous than with static carbonisation. Semi-caking coal yields more breeze than a highly caking coal. Cleaning and fine-grinding of the coal reduce breeze production; over-coked and uncarbonised coke have the opposite effect. Coke breeze can be utilised in gas producers for blending with coal that is to be carbonised, and for steam-raising.

D. K. MOORE.

**Asphaltic bitumen, its properties and applications.** J. S. JACKSON (J. Oil Col. Chem. Assoc., 1930, 13, 304—312).—The greater part of the bitumen in use is prepared artificially by the removal of the more volatile constituents from asphaltic-base crude petroleum oils. Its preparation in the Trumble plant is described. The various grades of bitumen are usually classified according to their consistency, and this test and those for ductility and ring-and-ball m.p. are given. For the purpose of analysis bitumen may be separated into three classes of compounds: (a) oily constituents; (b) semi-solid, sticky, resinous bitumenes; (c) dark-brownish solid asphaltenes. "Blowing" of bitumen with air results in an increase in the m.p. and an alteration of the character of the bitumen. Typical figures for a representative range of asphaltic bitumens are tabulated and their commercial applications, such as the making of emulsions, roof felt, road tar, and in the electrical industries, are described. H. S. GARLICK.

**Determination of wax in pitch.** W. LITTLEJOHN and W. H. THOMAS (J. Inst. Petroleum Tech., 1930, 16, 814—824).—The effect of a number of variables on the determination of wax in pitch by the cracking-distillation method has been investigated and a standardised method suggested, in which 10 g. of a sample are distilled up to 500° in a 70-c.c. glass retort at the rate of a 15° rise in temperature per min. Pure acetone is added (35 c.c. per g.) to the weighed distillate, and the mixture is refluxed. The solution is cooled, held at —32° for 5 min., filtered as rapidly as possible, and washed with the minimum quantity of acetone. The wax is dissolved in light petroleum, the solution evaporated, and the wax redissolved in acetone (70 c.c. per g. of wax). Precipitation is effected at 0°, the wax is filtered, redissolved in light petroleum, the solution evaporated, and the wax content calculated on the original sample, the result being expressed as a percentage of wax determined at 0° (and/or —32°) precipitated from acetone. The m.p. also should be determined and recorded.

H. S. GARLICK.

**Creosote oil.** K. A. POPOV (III Coll. Art. Inst. Mat. Comm. Commun., Moscow, 1929, No. 8, 112, 13—39).—Creosote oil for impregnation of wood should contain not more than 8—10% boiling below 205°, and the final b.p. should not exceed 355°. Not more than 25% of soft pitch and 0.5% of free carbon should be present.

CHEMICAL ABSTRACTS.

**Industrial uses of fuel oil.** I. LUBBOCK (J. Inst. Fuel, 1930, 4, 90—105).—Descriptions of the application of oil-firing to glass furnaces, the ceramic industry, metallurgy, cement manufacture, and domestic appliances are given.

D. K. MOORE.

**Turbine oils.** N. I. TSCHERNOSHUKOV (Azerbaid. Neft. Choz., 1930, No. 7—8, 96—99).—Types of turbine oil sludges are described. The stability of such oils is best determined by Butkov's method (B., 1929, 119); the product must not require more than 1 mg. of potassium hydroxide per g., and should contain not more than 0.15% of sediment. CHEMICAL ABSTRACTS.

**Petroleum from Chusovskikh Gorodkov.** I. V. POSTOVSKI and V. G. PLJUSNIN (J. Chem. Ind. Russia,



1929, 6, 776—779).—The oil contained C 82.86, H 10.17, S 4.09, N 0.33, O 2.65%, and had  $d_{25}^{25}$  0.9545,  $E_{D}^{20}$  3.08.

## CHEMICAL ABSTRACTS.

**Total heat and sp. heat of a series of fractions of petroleum oil, and their relation to other properties.** H. R. LANG, R. JESSEL, and A. H. STEED (J. Inst. Petroleum Tech., 1930, 16, 783—813).—Five fractions were prepared from Miri benzine and kerosene and their sp. heat, total heat, and the variation of these with temperature were measured, as were also the mean mol. wt., refractive index, sp. gr., and expansion coefficient. The determination of sp. heat between 0° and 100° was made by the continuous-flow electric method. For temperatures above 100° an apparatus was devised, based on direct measurement of the fall of total heat of a stream of oil by cooling it with a stream of water, which can be used at pressures other than atmospheric for both the liquid and the vapour phases. The mean mol. wts. were determined by the cryoscopic method, using nitrobenzene as the solvent. The results show a direct linear relationship both to the sp. gr. and to the refractive index. A detailed discussion of the apparatus and results obtained is given.

H. S. GARLICK.

**Waste-heat boilers in gasworks.** DAVIES.—See I. **Ammonium sulphate and nitrate mixtures.** GLUUD and others.—See VII. **Asphalts and bitumens on rubber estates.** SUTCLIFFE.—See XIV.

See also A., Jan., 41, **Water-gas equilibrium constant** (EMMETT and SHULTZ). 44, **Gaseous combustion in electric discharges** (FINCH and PATRICK). **Ignition of carbon monoxide-oxygen mixtures** (BREWER and DEMING). 54, **Analysis of mixtures of hydrogen, methane, and ethane** (MULDERS and SCHEFFER). 60, "Fibrous lignites" and "fibrous coal" (GOTHAN and BENADE). **Occurrence of resin in Ruhr coals** (HOFFMANN and KIRCHBERG). **Condensation of hydrocarbons by electrical discharge** (LIND and GLOCKLER). **Production of liquid hydrocarbons from propylene** (MAILHE and RENAUDIE). 78, **Behaviour of benzenoid hydrocarbons in the Tesla discharge** (AUSTIN and BLACK).

## PATENTS.

**Separation of combustible material from its associated non-combustible material.** W. L. REMICK (U.S.P. 1,767,400, 24.6.30. Appl., 27.5.29).—Coal and slate are separated by a combination of gravity separation and flotation processes. A preliminary gravity separation is effected by washing in an upward current of water. The large coal is separated from the fines and small slate particles by means of a de-watering screen. The fines, slate particles, and separated wash-water are mixed with oil and air in a pump and returned to the washing tank through revolving jets. The oiled coal particles adhere to the large coal and are removed with them at the screen.

H. E. BLAYDEN.

**Coking retort oven.** J. BECKER, ASSR. to KOPFERS Co. (U.S.P. 1,755,383, 22.4.30. Appl., 16.5.21).—The heating walls of the coking chamber of a battery contain a group of horizontal combustion flues disposed vertically above each other and extending from side to side of the

chamber. Each group of flues works in conjunction with an inflow and an outflow regenerator also extending from side to side of the chamber and being parallel to the heating walls. Means are provided for reversal of the direction of flow of gases in the regenerators and flues in such a manner that all the flues of the battery operate as burning flues. Provision is also made for heating the battery by means of preheated air and producer gas etc.

H. E. BLAYDEN.

**Removal of distillation products from the lower part of coke ovens.** K. BEUTHNER (B.P. 337,278, 3.1.30. Ger., 3.1.29).—The door and fuel support which closes the bottom of a vertical coke oven has a hood consisting of a closed part and a part which is open on the inner side, the lateral opening of this part running in the direction of the middle longitudinal plane of the chamber. The opening is so arranged that the material to be distilled only partly fills the open part of the hood on account of its natural angle of repose, leaving free a space suitable for removing the distillation gases. At the end of the distilling period steam may be introduced into the coke oven through the open-hood part.

A. B. MANNING.

**Coke-oven heating flue.** C. E. UNDERWOOD and J. A. BEATTY, ASSRS. to BETHLEHEM STEEL Co. (U.S.P. 1,751,775, 25.3.30. Appl., 27.4.26).—The centre bricks at the ends of the heating flue walls of the oven have their outer faces flush with the terminal bricks of the side walls, and have on their outer side filler bricks which overlap the joints between the side walls and the centre bricks. Flash plates embrace the filler bricks and the terminal portions of the side walls.

A. B. MANNING.

**Chamber ovens for producing semi-coke and similar products.** SOC. GÉN. DE FOURN. À COKE, SYSTÈMES LECOCQ (B.P. 336,809, 28.10.29. Belg., 27.10.28).—Coal is coked in narrow metallic enclosures on either side of which are heating walls of refractory material. The walls of the enclosures are spaced from the heating walls so that they are heated only by radiation.

A. B. MANNING.

**Heat treatment of vegetable and mineral substances.** F. K. WOODROFFE (B.P. 336,657, 18.7.29).—The material is fed in at one end of a retort upon the bottom of which it is distributed by paddles which are given an oscillatory movement across and a longitudinal movement along the retort, so that the material is gradually conveyed from the inlet to the discharge end. The retort is preferably sector-shaped in cross-section, the bottom being formed of curved metal plates, and is adapted to be heated from below.

A. B. MANNING.

**Apparatus for subjecting materials to the action of indirect heat.** MCG. CLINE, ASSR. to WOOD PROCESS Co., INC. (U.S.P. 1,763,758, 17.6.30. Appl., 17.6.26).—The apparatus is suitable for the destructive distillation of sawdust etc. It comprises a number of superposed, hollow, annular shelves supported by a fixed central flue, the material being raked over the shelves in succession by arms projecting inward from a rotating outer shell. Heating of the shelves is effected by gases rising through spaces left between the wall of the flue

and the lower inner edges of the annuli, and radial conduits extending from near the outer circumference back to the flue permit the gases to leave the shelves. By dividing the central flue into a number of truncated cones with closed small (and lower) ends a form of stage-heating may be obtained. B. M. VENABLES.

**Apparatus for ascertaining the fractionating temperatures of fractionatable constituents of solid carbonaceous and other materials [e.g., ores].** J. W. H. AINSOW (B.P. 318,966, 2.9.29. Austral., 13.9.28).—The apparatus comprises a small, inclined tubular retort with suitable inlets, outlets, and thermometer pockets; it is also provided with an internal stirrer the shaft of which is rather below the axis of the tube, and with a setting to produce uniform heating. B. M. VENABLES.

**Reactivation of contaminated adsorptive carbon.** METALLGES. A.-G. (B.P. 313,154, 15.5.29. Ger., 8.6.28).—The carbon, with the addition of a binding medium if desired, is moulded into blocks, which are then treated, without external heating, with gaseous mixtures containing not more than 8% of free oxygen, e.g., mixtures of air and steam, at temperatures at which the impurities in the carbon are eliminated without any appreciable loss of carbon by combustion. The carbon may be treated with water, acids, or other solvents prior to reactivation. A. B. MANNING.

**[Destructive] hydrogenation of [bituminous] coal.** IMPERIAL CHEM. INDUSTRIES, LTD., R. HOLROYD, and C. COCKRAM (B.P. 337,028, 18.7. and 28.12.29).—Bituminous coal is heated at or just above the temperature of initial thermal decomposition (cf. Holroyd and Wheeler, B., 1929, 116), with or without hydrogen, and the product is then subjected to destructive hydrogenation. The first stage of the treatment may be carried out under pressure, hydrogen being added in sufficient amount to deoxygenate the coal and convert it into a fusible product. Both stages may be performed in the presence of an oil and of catalysts, particularly massive metal catalysts. A. B. MANNING.

**Destructive hydrogenation [of solid carbonaceous materials suspended in a liquid].** C. F. R. HARRISON, E. D. KAMM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,610, 16.7.29).—The total products of destructive hydrogenation are withdrawn from the reaction vessel and led to a hot catch-pot maintained at such a temperature, e.g., 350°, that the light oils and middle oils remain in the vapour phase, while the heavy oils are condensed or remain unvaporised, and are led back to the process without substantial loss of heat. When solid matter is present in the reaction products it may be separated, e.g., by centrifuging, before returning the oil to the process. A. B. MANNING.

**Destructive hydrogenation [of carbonaceous materials].** C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,991, 19.7.29).—Coals and oils are hydrogenated in the liquid phase under pressure in a vertical tower (cf. B.P. 308,995 and 309,258; B., 1929, 465, 457) wherein is mounted a massive catalyst of relatively large surface area. The catalyst may comprise a number of units each consisting of a number

of plates, assembled side by side, and mounted at right angles to the cross-section of the tower.

A. B. MANNING.

**Gas producers.** A. L. GALUSHA (B.P. 337,005, 23.7.29).—A gas producer is provided with an overhead feed bin (surmounted, if desired, by a storage bin) capable of holding fuel sufficient for a relatively long period of working. The producer is connected with the bin by means of hollow columns which open into the producer in such a way as to maintain a constant level of fuel bed therein and at the same time leave a sufficient gas space above the bed. A. B. MANNING.

**Gas-producer furnaces.** L. RAINCHON (B.P. 336,887, 13.1.30. Addn. to B.P. 255,329; B., 1926, 734).—A furnace in accordance with the prior patent has, on the front side, below the guide surface which conducts the coal from the hopper to the furnace, a primary air channel and an opening for cleaning purposes, and at the rear side, at a level below that of the discharge opening of the loading hopper, a secondary air channel, a second air-admission channel, and a cleaning opening which also serves the purpose of a channel for the admission of air. A. B. MANNING.

**Gas manufacture.** F. W. STEERE, ASSR. to SEMET-SOLVAY ENG. CORP. (U.S.P. 1,752,278, 25.3.30. Appl., 25.4.25).—The apparatus comprises a generator, a carburettor, a superheater, and a wash box in series, and also a checkerbrick chamber adjacent to and connected with the base of the generator. A gas conduit leads from the top of this heat-storage chamber to the connexion between the superheater and wash box. After air-blasting the fuel bed and burning the blast gases in the carburettor and superheater, water is injected into the top of the latter through water sprays and the steam so formed is passed through the carburettor and down through the fuel bed; the hot water-gas produced is passed through the heat-storage chamber and thence through the wash box to storage. An up-run may then be made by spraying water into the heat-storage chamber, and also into the bottom of the generator, and passing the steam so formed up through the fuel bed, the water-gas produced being passed through the carburettor, superheater, and wash box to storage. A. B. MANNING.

**Water-gas apparatus.** F. W. STEERE, ASSR. to YOUNG-WHITWELL GAS PROCESS CO. (U.S.P. 1,751,497, 25.3.30. Appl., 7.5.23. Renewed 6.8.27).—A water-gas set comprises a generator, carburettor, and superheater, connected in series by unobstructed passages. Another passage connects the bottom of the generator with the gas offtake leading to the wash box. A by-pass connexion is provided between this second passage and the main passage, and a jet which supplies steam for the down-run is mounted in the by-pass in such a manner that by its injector action a part of the water-gas produced is re-circulated through the superheater, carburettor, and generator, admixed with the steam. A. B. MANNING.

**Back-run method of operating gas plants.** G. E. WHITWELL, ASSR. to YOUNG-WHITWELL GAS PROCESS CO. (U.S.P. 1,752,223, 25.3.30. Appl., 26.4.23. Cf. U.S.P. 1,751,497; preceding abstract).—In the manu-

facture of carburetted water-gas a step is introduced in which a mixture of combustible gas and steam is passed successively through a single superheater, a single carburettor, and through the fuel bed of the generator, whereby water-gas is produced. A. B. MANNING.

**Heating of regenerator chambers in the continuous production of water-gas.** J. PINTSCH A.-G. (B.P. 318,174, 24.8.29. Ger., 29.8.28).—In the method employing as gasifying agent a current of water-gas and steam heated in an apparatus of the regenerator type, itself heated by the combustion therein of part of the water-gas produced, the temperature of the heating apparatus is regulated by the introduction of flue gases, or a mixture of flue gases and steam, by means of a blower which recirculates part of the combustion products from the heating apparatus.

A. B. MANNING.

**Production of water-gas.** E. S. ANDREWS. From J. PINTSCH A.-G. (B.P. 336,965, 22.7.29).—Finely-divided solid carbonaceous material is gasified in steam, or in a mixture of steam and water-gas, in such a way that the gasifying agent, preheated in a regenerator surrounding the reaction tube, is introduced into the latter at a number of points arranged progressively along the tube from the end where the fuel enters.

A. B. MANNING.

**Production of water-gas from pulverulent and fine-grained carbon.** METALLGES. A.-G., and C. MUELLER (B.P. 336,821, 4.11.29).—The fuel is gasified with oxygen admixed with steam, part of the gas produced being introduced into the current of oxygen and steam and burned therein before the thereby highly preheated gasifying medium reaches the fuel.

A. B. MANNING.

**Absorption process [for removing gasoline from natural gas].** P. T. SHARPLES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,766,195, 24.6.30. Appl., 4.4.25).—In an enclosed cyclic system a condensible hydrocarbon is absorbed in a suitable oil which is subsequently stripped of the absorbed hydrocarbon by distillation. A portion of the stripped oil is purified from contaminating material of a higher sp. gr. than that of the oil by centrifuging while hot; the by-passed oil is then returned with the main supply of oil to the absorption process.

H. S. GARLICK.

**Recovery of benzol and the like [from coal-distillation gas, water-gas, etc.].** P. T. SHARPLES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,766,196, 24.6.30. Appl., 20.7.25).—After the wash oil is debenzolised by distillation, it is centrifuged at 95–100° to remove suspended particles of finely-divided matter. The stripped and centrifuged oil is then returned to the scrubbing tower.

C. B. MARSON.

**Apparatus for treating asphaltic oils for production of asphalt.** M. R. L. MILLER (U.S.P. 1,766,446, 24.6.30. Appl., 8.1.27).—A metallic still jacketed with asbestos is provided with a heating furnace and internal cooling pipes submerged in the oil. A suitable cooling fluid such as gas or oil can be made to flow through these pipes, the flow being regulated by thermostatically controlled valves, and other pipes are arranged to blow air through the contents of the still. The still is heated

externally, and air is simultaneously blown through the oil. When the temperature of the oil reaches 127° the furnace is shut off and the supply of air continued to effect oxidation of the oil. Increase of temperature of the oil above that at which destructive distillation commences is prevented by the action of the valves mentioned above. The flow of air is increased from 300 to 700 cub. ft. per min over a period of 6–7 hrs. The whole process may occupy 18–70 hrs. according to the nature of the required product. During oxidation steam is led into the still above the oil to prevent ignition of oil vapours. The steam and oil vapours may subsequently be condensed. When high-gravity oils are to be treated, steam may be blown through the oil to effect a preliminary distillation, the product being then treated as described. H. E. BLAYDEN.

**Treatment of bitumen, bituminous preparations, tar, etc. for electrical and other uses.** D. ANDERSON & SON, LTD., and R. O. CHILD (B.P. 339,470, 25.11.29).—The material to be treated is mixed in a normal or heated condition with preferably an approximately equal quantity of a suitable solvent and treated with a suitable quantity of colloidal silicic acid. After settlement, the purified solution is decanted through a filter-bed of silicious material or treated with a further quantity of silicic acid and centrifuged before distilling to reduce the solution to a desired viscosity.

H. S. GARLICK.

**Separation of tars, tar oils, petroleum, and the like into fractions of technical value.** J. KÁRPÁTI (B.P. 336,945, 18.6.29. Cf. B.P. 283,569; B., 1929, 424).—The raw material is brought into solution in an aqueous organic medium, *e.g.*, aqueous alcohol (30–70%), at a raised temperature, preferably above the b.p. of the medium, and under pressure, and the solvent capacity of the medium is reduced in stages, *e.g.*, by progressively cooling the mixture or by varying the concentration of the solvent, whereby the dissolved substances are caused to separate in fractions of increasing solubility. Dissolution of the raw material may be so effected as to leave the pitch or asphalt undissolved. By this treatment tars may be fractionated into pitch, oils of high paraffin wax content, lubricating oils, hydrocarbon oils free from wax and cresols, unsaturated hydrocarbons, and phenols. The fractions possess a high degree of purity, and require little further refining.

A. B. MANNING.

**Obtaining products from hydrocarbons by heat treatment.** W. K. LEWIS and N. E. LOOMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,297, 24.6.30. Appl., 30.3.23).—A suitable heat-radiating material, *e.g.*, refractory chequerwork, is heated by furnace gases and serves to radiate heat to a pipe coil which receives the feed oil. Connected to the pipe coil is another coil placed in another compartment, and not receiving radiant heat, but heated by the hot products of combustion which finally escape to the stack, the temperature of this zone being regulated by the return of cool combustion products thereto. H. S. GARLICK.

**Conversion of hydrocarbons into hydrocarbons of lower mol. wt.** J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 339,274, 30.5.29).—The initial

hydrocarbon material is subjected to cracking at a high temperature and under reduced, atmospheric, or increased pressure, in the presence of a catalyst. One or more pieces consisting wholly of silicon or of silicon deposited upon a carrier are suitable as catalysts.

H. S. GARLICK.

**Cracking of hydrocarbon oils.** W. W. TRIGGS. From PANHANDLE REFINING Co. (B.P. 339,291, 29.6.29).—The plant comprises a cracking still and a preheating still combined in one structural unit containing two separately fired chambers divided by a central refractory partition with an opening above, together with a stack having a flue opening into the preheating still chamber whereby the heated gases and products of combustion from the cracking still pass over the top of the separating partition, and, by down-draught, are drawn through the preheating still chamber before passing to the stack. Hydrocarbon oils are preheated and introduced into a vaporising zone. The vapours are passed through a separate, superheated, cracking zone at 540–650°, and into a series of heat-exchanging and fractionating zones where they pass in indirect countercurrent with fresh make-up oil. A portion of the condensate from one of the fractionating zones is passed back to a preceding zone and used as solvent means to remove heavier condensate therefrom, which, together with the condensed higher-boiling uncracked portions, is caused to absorb heat from the partly cracked vapours drawn from the cracking zone, and is returned to the flashing zone. The light condensate is removed, stripped of lighter products absorbed during condensation, and finally the desired low-boiling products are collected while the make-up oil, preheated to the point of vaporisation, is introduced into the system as vapour. (Cf. B.P. 337,380; B., 1931, 54.)

H. S. GARLICK.

**Coking [heavy liquid hydrocarbons].** H. G. JENNISON and C. R. FABEN (U.S.P. 1,749,138, 4.3.30. Appl., 28.7.28).—Hot gases produced by the combustion of carbonaceous fuel are passed, under induced draft, through the tar, pitch, or petroleum residue, etc., which is contained in a metal chamber lined with refractory material, and the liquid hydrocarbons are thereby distilled to coke. The distillate is condensed and collected. When coking is practically complete air may be introduced into the heating gases in order to oxidise any residual hydrocarbons in the coke.

A. B. MANNING.

**Cracking of [fuel] oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,766,331, 24.6.30. Appl., 6.12.20. Renewed 25.5.29).—A stream of charging oil is passed under pressure through a heating zone at a temperature sufficient to vaporise a substantial part thereof, and the heated hydrocarbons are introduced under a pressure in excess of that in the heating zone into an enlarged expansion chamber. The vapours are dephlegmated and the reflux condensate is separately heated to cracking temperature and passed under pressure to the expansion chamber, from which any unvaporised residual oil is withdrawn. H. S. GARLICK.

**Treatment and purification of hydrocarbon oils.** L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,751,862, 25.3.30. Appl., 15.11.23. Renewed

2.7.28).—Distillates from the pressure distillation of hydrocarbon oils are purified by a continuous process in which they are treated successively with plumbite, sulphuric acid, and caustic soda, each treatment being followed by water-washing. In each treatment the oil and reagent are introduced into a mixing chamber and passed thence to a settling chamber. The water-washing is carried out by causing the oil to flow through a confined zone countercurrent to a spray of water. Throughout the whole treatment the material is maintained under a pressure above atmospheric.

A. B. MANNING.

**Apparatus for cleansing [lubricating and other] oils.** PRESTON, STALLARD & Co., LTD., and J. MANSBURY (B.P. 337,981—2, 8.11.29).—(A) A hot-water circulating pipe is centrally disposed in a heat-insulated tank having a funnel-shaped lower portion connecting with an outlet pipe. An electric heater extends upwardly into the water-circulating pipe and is controlled by an adjustable electric delay-action time switch actuated by clockwork. Water is circulated through oil in the tank by thermosiphon means and brings about coagulation of the impurities which, on cooling of the oil, form a layer at the oil-water interface. (B) The upper portion only of the tank (*i.e.*, that portion within which the oil collects) is surrounded by heat-insulating lagging, whilst the lower funnel-shaped portion, in which the water collects, is exposed to the atmosphere in order to accelerate cooling after the heater has been cut out.

H. S. GARLICK.

**Refining of liquid hydrocarbons.** SILICA GEL CORP. (B.P. 311,285, 7.5.29. U.S., 8.5.28).—A liquid hydrocarbon has added to it sufficient of an acid (*e.g.*, sulphuric acid) to react with only a portion of the gum-forming constituents contained therein, and is then brought into contact with a solid absorbent (*e.g.*, silica gel) at normal or raised temperatures (preferably at 121–149°). Alternatively, the hydrocarbon is brought into contact with a solid absorbent to which has been added a sufficient quantity of acid to react with only a portion of the gum-forming constituents. A sufficient superatmospheric pressure is maintained to keep the hydrocarbon in the liquid phase. The absorbent may be impregnated with one or more metallic oxides to facilitate removal of sulphur, and in place of sulphuric acids methyl sulphate or anhydrous aluminium chloride may be used.

H. S. GARLICK.

**Refining of liquid hydrocarbons.** J. JOHNSON, JUN., Assr. to J. R. TOWNSEND (U.S.P. 1,766,338—9, 24.6.30. Appl., [A] 28.3.23, [B] 11.8.25. Renewed [A] 29.3.30, [B] 28.3.29).—(A) Crude hydrocarbon oil is mixed with 2–10% of acidified absorbent earth and distilled up to a coking temperature. The vapours are collected and condensed into the normal commercial products, which may be re-treated separately if desired. (B) By distilling crude liquid hydrocarbons, *e.g.*, crude petroleum or shale oil, with a finely-powdered, acidified earth (95 pts. of absorbent clay or aluminium silicate and 3 pts. of sulphuric acid), the products carried off at the various customary temperatures will be practically pure gasoline, kerosene distillate, and lubricating oil stocks without any further treatment except a possible redistillation in the same manner.

H. S. GARLICK.

**Refining of petroleum and like hydrocarbons.** W. W. TRIGGS. FROM PAN AMER. PETROLEUM CO. (B.P. 338,482—4, 7.5.29).—Distillates are refined by treatment with sulphur dioxide and sulphuric acid. In (A) lamp oil is cooled by heat exchange with cold treated oil and then by heat exchange with liquid sulphur dioxide to  $-8^{\circ}$ . The oil is then passed upwards through two towers where it meets a countercurrent of liquid sulphur dioxide. It is next treated in a similar tower with fuming sulphuric acid ( $>15\%$   $\text{SO}_3$ ), and then passed through heat exchangers and the sulphur dioxide removed by distillation under reduced pressure. The process is continuous. In (B) heavier distillates such as transformer oil are treated first with sulphur dioxide at below  $0^{\circ}$  and then with fuming sulphuric acid ( $15\%$  oleum) in the presence of dissolved sulphur dioxide, at temperatures below  $15^{\circ}$ . The first extraction is carried out in a packed tower, the oil being passed upwards and refining agent run downwards over the packing material. In (C) distillates containing polymerisable hydrocarbons and sulphur compounds are mixed, after cooling to  $-8^{\circ}$  in a heat exchanger with liquid sulphur dioxide, and the mixture is passed successively through two treating tanks where it is brought into contact with sulphuric acid ( $d$  1.71—1.84) or, in the case of gasolines, cracked under 1000 lb. pressure with fuming sulphuric acid. After this treatment the oil is passed through the heat exchanger and the sulphur dioxide removed by heating under vacuum; the gas is also recovered from the acid tar. T. A. SMITH.

**Stabilisation of crude petroleum.** F. B. BIMEI, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,767,341, 24.6.30. Appl., 19.8.26).—Crude petroleum from the wells passes under reduced pressure through a preheater, where it is raised to a temperature not above  $150^{\circ}$ , into a rectifier from which liquid and gas containing vapour are separately removed. The gas is compressed to recover normally liquid constituents and the condensate is returned to the cooled oil flowing from the rectifier.

H. S. GARLICK.

**Refining the hydrocarbons of mineral oils.** W. W. GROVES. FROM ALLGEM. GES. F. CHEM. IND. (B.P. 338,579, 22.7.29).—The whole of the distillate up to  $400^{\circ}$ , except the benzine portion, obtained from mineral oil is treated with liquid sulphur dioxide instead of treating fractions as is the usual practice. This method enables the lubricating portion to be refined more easily than when a special lubricating oil cut is treated with the refining agent. The benzine portion is removed owing to the difficulty in separating it by distillation from liquid sulphur dioxide. The distillate from Persian crude oil is treated with  $150\%$  of the liquid sulphur dioxide. The products are  $25\%$  of extract ( $d$  0.933) and  $75\%$  of refined oil ( $d$  0.895). T. A. SMITH.

**Fractional extraction of petroleum hydrocarbons with alcohol.** T. A. WERKENTHIN, Assr. to SOLAR REFINING CO. (U.S.P. 1,766,768, 24.6.30. Appl., 30.12.27).—Topped crude oil, wax-bearing distillate or residue, heavy residual fuel oil, spent lubricating oil, or heavy residual oil from cracking may be pumped through a heat exchanger into a mixing coil where it is mixed with alcohol that has been similarly preheated

to a temperature below its b.p. The dissolved oil extract and undissolved oil is run into a settling tank where most of the undissolved oil settles out, the oil extract overflowing into a centrifugal separator where the last traces of undissolved oil are removed. The extract is then forced into a still where the alcohol is removed, the vapours passing through a dehydrator or by-pass line into the heat exchangers and condenser, the condensate being subsequently collected in a storage tank and re-circulated through the system. The undissolved oil collected in the bottom of the settling tank and separated by the centrifuge, from which "cracking stock" has been removed, undergoes a similar process of extraction in a second unit where the light lubricating components are extracted. Third and fourth units serve to extract in a similar manner the medium and heavy oil fractions, whilst the final undissolved material remains as an asphaltic residuum. Where the original oils were wax-bearing, the treatment produces a series of lubricating oils containing waxes notable for their good crystallising properties and ready removal from the oils.

H. S. GARLICK.

**Manufacture of oxidation products of high-molecular hydrocarbons, waxes, etc.** J. Y. JOHNSON. FROM I. G. FARBERIND, A.-G. (B.P. 337,130, 12.9.29).—In the utilisation of the heat of reaction by heat exchange which consists in injecting a spray or mist into the cooling jacket, the amount of spray has hitherto been adjusted so as to secure its complete evaporation. It is advantageous to have in contact with the reaction vessel in the cooling jacket a quantity of cooling liquid in addition to the spray or mist, so that sudden evolution of heat may be dealt with. C. HOLLINS.

**Deoxidiser for insulating oils.** C. J. RODMAN and A. H. MAUDE, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,736,464, 19.11.29. Appl., 20.7.23).—A device for preventing oxidation of insulating oil comprises a container with a lateral inlet near the base and a pipe at the top leading to the upper part of the oil tank. In the container are two concentric gauze cylinders, the intermediate space being filled with a mixture of sodium thiosulphate and sodium bicarbonate. Air entering at the bottom of the container passes through this mixture and is deprived of its oxygen, so that a mixture of carbon dioxide and nitrogen is introduced into the space above the oil. W. J. WRIGHT.

**Motor spirits.** L. W. E. TOWNSEND and A. RICHMOND (B.P. 338,581, 23.7.29).—High-boiling shale oil ( $230$ — $360^{\circ}$ ) is cracked at high temperature, the tar portion of the condensate being dehydrated in a stream of carbon dioxide at  $120^{\circ}$ . The pressure is then reduced to 2 mm. and the distillate collected separately. The addition of  $1$ — $5\%$  of this oil to petrol allows a considerable addition of paraffin oil to be made without impairing the anti-knock value of the spirit. T. A. SMITH.

**Internal-combustion engine fuel.** I. BOURIE (U.S.P. 1,752,724, 1.4.30. Appl., 4.3.26).—Denatured alcohol in relatively small proportions (e.g., 1 oz. per 10—15 gals.) is mixed with gasoline or crude oil.

H. ROYAL-DAWSON.

**Processing of coal and product thereof. Powdered coke product.** W. RUNGE, Assr. to INTERNAT. COAL

CARBONISATION Co. (U.S.P. 1,783,982—3, 9.12.30. Appl., 12.6.25).—See B.P. 253,498—9; B., 1927, 34, 99.

**Dehydration of moist [solid] fuels.** H. A. von STADEN and A. HOEHN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,757, 2.12.30. Appl., 23.10.28. Ger., 14.12.27).—See B.P. 317,771; B., 1929, 885.

**Fuel briquette.** J. S. FORD and F. MURRAY (U.S.P. 1,784,665, 9.12.30. Appl., 6.3.29. N. Zealand, 30.11.28).—See B.P. 329,437; B., 1930, 803.

**Carbonising apparatus.** O. H. HERTEL, Assr. to OLD BEN COAL CORP. (U.S.P. 1,783,190, 2.12.30. Appl., 7.5.25).—See B.P. 249,710; B., 1926, 429.

**Manufacture of active carbon.** J. VAN LOON (U.S.P. 1,782,493, 25.11.30. Appl., 15.2.27. Holl., 5.3.26).—See B.P. 292,213; B., 1928, 631.

**Manufacture of activated carbon.** R. DEFRISS and R. WÄLDER (U.S.P. 1,783,110, 25.11.30. Appl., 27.2.28. Austr., 3.3.27).—See B.P. 303,669; B., 1929, 195.

**Elimination of sulphur compounds from gases.** G. WIETZEL, J. JANNEK, and F. FRIED, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,782,590, 25.11.30. Appl., 29.3.28. Ger., 8.4.27).—See B.P. 319,396; B., 1929, 969.

**Cracking of hydrocarbons.** G. C. HARGROVE and W. B. MONTGOMERY, Assrs. to GASOLINE PRODUCTS Co. (U.S.P. 1,784,065, 9.12.30. Appl., 20.12.27).—See B.P. 290,996; B., 1929, 120.

**[Machines for] manufacture of briquettes.** W. V., and W. J. BOOTH (B.P. 339,868, 6.2.30).

**Pulverised fuel burners for locomotive and other furnaces.** R. ROOSEN (B.P. 339,901, 12.3.30. Ger., 22.3.29).

**Continuous distillation** (U.S.P. 1,756,032). **Adsorbent materials** (U.S.P. 1,752,721). **Distributors for gases carrying solids** (B.P. 338,327). **Treatment of exhaust gases** (B.P. 338,933).—See I. **Acetylene and hydrogen** (B.P. 337,088). **Absorption of olefines** (B.P. 336,633 and 336,603—4).—See III. **Solvent extraction of wood** (U.S.P. 1,762,785).—See V. **Waterproof cement** (U.S.P. 1,755,638). **Waterproof board** (B.P. 319,213). **Paving material** (U.S.P. 1,767,830). **Surfacing roads etc.** (B.P. 338,206). **Materials for road construction etc.** (B.P. 337,521).—See IX. **Sucker rod [for oil pumps]** (U.S.P. 1,764,068).—See X. **Rubber dispersions** (B.P. 315,839).—See XIV.

### III.—ORGANIC INTERMEDIATES.

See A., Jan., 60, **Condensation of hydrocarbons by electrical discharge** (LIND and GLOCKLER). 61, **Preparations of anhydrous alcohol** (ADICKES). 78, **Behaviour of benzenoid hydrocarbons in the Tesla discharge** (AUSTIN and BLACK). 81, **Purification of phenanthrene** (COHEN and CORMIER). **Acenaphthene series** (MORGAN and HARRISON). 84,  **$\beta$ -Naphthol-3-sulphonic acid** (HOLT and MASON). 87, **Di- and tri-arylamine derivatives** (JOSZT and LESNIAŃSKI).

#### PATENTS.

**Manufacture of acetylene and hydrogen in the electric arc.** J. Y. JOHNSON. From I. G. FARBENIND.

A.-G. (B.P. 337,088, 7.8.29).—Dilute hydrocarbon gases, e.g., waste gas from coal hydrogenation, are passed through a series of arcs (acetylene being removed after each) of increasing lengths, e.g., in the ratio 2.8 : 4 : 5. Suitable apparatus is described. C. HOLLINS.

**Manufacture of ethylene [from acetylene].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,999, 24.5.29).—In the hydrogenation of acetylene to ethylene a catalyst of compact tellurium at 450° or sheet nickel at 180° minimises the production of ethane even with excess of hydrogen. C. HOLLINS.

**Absorption of (A, B) ethylene by means of sulphuric acid, (C) olefines having three or more carbon atoms in the molecule by means of sulphuric acid, phosphoric acid, or concentrated sulphonic acids, and (A, C) manufacture of the corresponding esters, ethers, alcohols, or other compounds.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 336,633, 336,603—4, 4.6.29).—As ethylene absorption catalysts there are employed metals of the platinum group (A) in finely-divided condition, preferably on a carrier; (B) in soluble form, with or without addition of compounds of copper, iron, cobalt, or nickel, preferably either or both series of metal compounds being converted into soluble complex compounds by passing carbon monoxide or nitric oxide through the acid mixture. (C) Method (B) is extended to higher olefines, and soluble complex compounds may also be prepared by using ethylene in place of carbon monoxide or nitric acid. The absorbent for the higher olefines may be sulphuric acid, phosphoric acid, or benzenesulphonic acid. C. HOLLINS.

**Polymerisation of diolefines.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,019, 25.5.29).—In the polymerisation of diolefines with sodium etc., soluble plastic products are obtained by continuously removing the soluble products as formed by means of a solvent (light petroleum, ether, benzene, cyclohexane); e.g., solvent is distilled into and through the polymerisation vessel as in a Soxhlet apparatus. C. HOLLINS.

**Manufacture of aliphatic [acetic] anhydrides.** H. DREYFUS (B.P. 336,668 and 336,960, [A] 19.7.29, [B] 22.7.29).—(A) Asbestos is used as a carrier for catalysts in the conversion of acetic acid into anhydride by heat. (B) For the conversion of acetic and other aliphatic acids into anhydrides at 350—700°, oxides or acids of arsenic or antimony, or salts of these, are used as catalysts. C. HOLLINS.

**Catalytic manufacture of synthetic acetic acid.** SOC. FRANÇ. DE CATALYSE GÉNÉRALISÉE (B.P. 338,329, 27.7.29. Fr., 26.1.29).—Carbon monoxide is hydrogenated in the presence, as catalysts, of one or more of the phosphates, silicates, or borates of nickel, cobalt, chromium, iron, and copper, with or without any other metals, oxides, carbonates, or other salts. The reaction is effected at 500—550°/450—600 atm.

#### II. ROYAL-DAWSON.

**Manufacture of oxygenated organic compounds [from carbon oxides and hydrogen].** H. DREYFUS (B.P. 337,014, 24.7.29).—The use of catalysts comprising ferrites, ferrates, cobaltites, or cobaltates of alkaline-



earth metals at 250—350°/50—200 atm. leads to production of ethyl alcohol. The gas mixture may be replaced wholly or in part by methyl or higher alcohols produced by hydrogenation of carbon oxides.

C. HOLLINS.

**Manufacture of butyl alcohol [from ethyl alcohol].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 336,811, 17.2.30).—Ethyl alcohol is heated with magnesium and copper oxides (3 : 2 or 4 : 1) in an autoclave at 270°/130—140 atm. or 325°/200—300 atm., so that the alcohol is present only as vapour. A 30% conversion into butyl alcohol takes place in 2 hrs.

C. HOLLINS.

**Manufacture of tetrahydrofurfuryl alcohol.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 337,296, 25.1.30).—Furfuraldehyde is heated with hydrogen at 50—150°/6.5—120 atm. in presence of reduced nickel; 5—15% of water may be present.

C. HOLLINS.

**Manufacture of aliphatic acids and their esters [from carbon monoxide and alcohols].** BRIT. CELANESE, LTD., and W. BADER (B.P. 337,053, 27.7.29).—Carbon monoxide is passed into the "liquid" (methyl) alcohol containing an inorganic acid catalyst (phosphoric acids, preferably partly esterified with the alcohol) at 300—400°/100—300 atm. The use of aqueous alcohol (40—80%) or addition of steam is advantageous. Thus methyl alcohol is mixed with phosphoric acid at 60—90° and injected into a heated reaction vessel through which carbon monoxide and steam are passed under pressure. The products pass through a separator at 150—200° where phosphoric acid is returned to the mixer, to a condenser, and after removal of acetic acid and methyl acetate the carbon monoxide is recirculated.

C. HOLLINS.

**Manufacture of dialkyl sulphates [methyl sulphate].** ETABL. LAMBIOTTE FRÈRES (B.P. 336,681, 22.7.29. Fr., 25.6.29).—Dimethyl ether (or other dialkyl ether) is absorbed in 60% oleum at 30°, and the product is distilled. Preferably equimolecular proportions are employed, and the sulphuric acid left after distillation is used for the preparation of more ether from alcohol.

C. HOLLINS.

**Manufacture of pure glycerin.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,608, 12.7.29).—A "mist" comprising air or other carrier and a finely-divided liquid having a b.p. not above that of water is led at 15—30 mm. pressure into crude concentrated glycerin at its b.p.; the distillate is pure enough for nitration. Thus 82% crude glycerin from spent soap lye is heated at 160—180°/30 mm. and wet steam at 60° is led in; or a mist of carbon tetrachloride or benzene in air or carbon dioxide at 5° is passed into crude glycerin at 180°/20 mm., the condensate being collected at 110°.

C. HOLLINS.

**Manufacture of lecithin preparations.** ORANIENBURGER CHEM. FABR. A.-G. (B.P. 317,730, 19.8.29. Ger., 20.8.28).—Preparations for leather dressing etc. are made by dissolving lecithin and an emulsifying agent in a water-miscible solvent, with or without addition of oil; e.g., evaporated milk, glycerin, soya-bean lecithin,

and water; lecithin, glycerol monochlorohydrin, neatsfoot oil, sulphonated neatsfoot oil, and Marseilles soap; lecithin, glycerin, and egg-yolk (with flour and alum for tanning glacé leather).

C. HOLLINS.

**Manufacture of polyhalogenated fatty acids.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 336,623, 15.7.29).—A fatty acid above  $C_8$  is treated with chlorine or bromine or a mixture of chlorate and hydrochloric acid in such amount as to give a trihalogenated acid; in the case of unsaturated acids at least one halogen must be introduced by substitution. Ricinoleic acid gives products containing 3, 6, 7, or 16 atoms of chlorine; oleic acid takes up 3 or 4, stearic acid 6, and dihydroxystearic acid 5 atoms of chlorine. The products are colourless oils or sticky plastic solids and are useful as dyeing assistants.

C. HOLLINS.

**Manufacture of (A, B) hydroaromatic hydrocarbons and (A) derivatives thereof.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,583 and 336,616, [A] 10.7.29, [B] 17.4.29).—(A) Aromatic or partly hydrogenated aromatic hydrocarbons, or (B) phenols, are converted into hydroaromatic hydrocarbons by hydrogenation at pressures above 100 atm. and at temperatures (A) below 350°, (B) between 250° and 400°, in presence of catalysts immune from sulphur poisoning, viz., oxides or sulphides of metals of groups II—VII mixed with heavy metals of group I or VIII either as metals, oxides, or sulphides. (A) Derivatives of the aromatic hydrocarbons, such as phenols, amines, etc., are reduced also, but temperatures above 260° must be avoided if the substituent groups are to be retained. Examples are: (A) benzene over platinum and tungsten sulphides at 320°/200 atm. for cyclohexane; liquid naphthalene over nickel and molybdenum oxides and calcium carbonate on active charcoal at 300°/150 atm. for tetrahydronaphthalene; anthracene in an autoclave with cobalt and molybdenum sulphides on Florida earth at 220°/100 atm. for octa-, deca-, and dodecahydroanthracene; (B) phenol with cobalt and molybdenum sulphides on active carbon at 270—300°/150 atm. for cyclohexane; crude cresol with nickel and molybdenum sulphides on Florida earth at 300—350°/150 atm.;  $\alpha$ -naphthol pitch with cobalt-aluminium-zinc oxides at 320°/150 atm. for hydrogenated naphthalenes;  $\beta$ -naphthol with nickel and tungsten sulphides on active silica at 250—270°/150 atm. for tetra- and decahydro-naphthalenes.

C. HOLLINS.

**Manufacture of 1-chloromethylnaphthalene.** I. G. FARBERIND A.-G. (B.P. 337,289, 11.1.30. Ger., 12.1.29).—Hydrogen chloride is led into a mixture of naphthalene, aqueous formaldehyde, and hydrochloric acid at 60—65° for 10 hrs. Distillation of the separated oil affords 1-chloromethylnaphthalene, m.p. 31—32°, b.p. 153°/12 mm. (yield 83%, conversion 61%).

C. HOLLINS.

**Manufacture of condensation products of the naphthalene series [1:4- and 1:5-naphthalenedicarboxylic bishydroxyarylamides].** W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 336,800, 18.10.29).—Naphthalene-1:4- or -1:5-dicarboxylic dichloride is condensed with an aminophenol having at least one of the positions 2, 4, and 6 free. Products from the 1:5-acid chloride with *m*-aminophenol (m.p.



295—298°), *p*-aminophenol (m.p. 325—330°), 2-amino-*p*-cresol (m.p. 190—193°), and 1:7-aminonaphthol, and from the 1:4-acid chloride with *m*-aminophenol (m.p. 270—273°) and 1:4- and 1:7-aminonaphthols, are described. The products are suitable for coupling on the fibre for ice colours. C. HOLLINS.

**Production of dye intermediates [of the dianthraquinonyl series].** W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 336,982, 16.4.29).—An anthraquinone carrying a halogen or diazo group in position 1 and chlorine, bromine, or a sulphonc group in position 2 is treated with copper to give 1:1'-dianthraquinonyls. The 2:2'-dichloro- (m.p. above 300°), 2:2'-dibromo-, and 2:2'-disulpho-derivatives are described. C. HOLLINS.

**Manufacture of acetic acid.** H. DREYFUS (U.S.P. 1,784,583, 9.12.30. Appl., 22.10.26. U.K., 7.11.25).—See B.P. 268,845; B., 1927, 521.

**Ethyl alcohol gel.** H. OHLE and J. OTHMAR-NEUSCHELLER (U.S.P. 1,783,133, 25.11.30. Appl., 15.11.27. Ger., 30.11.26).—See B.P. 281,662; B., 1929, 315.

**Manufacture of urea.** H. C. HETHERINGTON and H. J. KRASE, Assrs. to A. B. LAMB (U.S.P. 1,782,723, 25.11.30. Appl., 2.3.26).—See B.P. 335,913; B., 1931, 12.

**Manufacture of aromatic hydroxyaldehydes.** G. REDDELEIN, H. LANGE, and H. PFANNENSTIEL, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,783,584, 2.12.30. Appl., 27.6.28. Ger., 30.7.27).—See B.P. 284,889; B., 1929, 276.

**Catalytic hydrogenation of aromatic amino-compounds.** F. KLINGEMANN, W. LOMMEL, E. KORTEN, and T. GOOST, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,782,729, 25.11.30. Appl., 2.10.26. Ger., 10.10.25).—See B.P. 300,285; B., 1929, 350.

**Production of (A) thymol, (B) phenols.** H. JORDAN, W. SCHOELLER, and R. CLERC, Assrs. to CHEM. FABR. AUF AKT. (VORM. E. SCHERING) (U.S.P. 1,784,598—9, 9.12.30. Appl., 15.8.27. Ger., [A] 22.2.27, [B] 16.11.26).—See B.P. 285,833 and 280,924; B., 1929, 699, 237.

**Production of alkylated phenols.** H. JORDAN, Assr. to CHEM. FABR. AUF AKT. (VORM. E. SCHERING) (U.S.P. 1,782,966, 25.11.30. Appl., 12.7.26. Ger., 4.7.25).—See B.P. 254,753; B., 1928, 740.

**Production of products of hydrogenation of alkylated phenols.** H. JORDAN, Assr. to CHEM. FABR. AUF AKT. (VORM. E. SCHERING) (U.S.P. 1,782,621, 25.11.30. Appl., 12.7.26. Ger., 4.7.25).—See B.P. 254,753; B., 1928, 740.

**Recovery of cresol from aqueous solutions of same.** H. SUIDA (U.S.P. 1,784,711, 9.12.30. Appl., 6.7.26. Austr., 13.7.25).—See B.P. 255,047; B., 1927, 316.

**Manufacture of aminonaphthol ethers.** A. ZITSCHER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,784,408, 9.12.30. Appl., 16.12.27. Ger., 3.1.27).—See B.P. 304,441; B., 1929, 276.

**Splitting off sulpho-groups from anthraquinone-sulphonic acid derivatives.** R. E. SCHMIDT, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,782,747, 25.11.30.

Appl., 13.4.26. Ger., 17.4.25).—See B.P. 250,968; B., 1927, 743.

**Oxidation products of high-molecular hydrocarbons etc.** (B.P. 337,130).—See II. Vat dye intermediates (B.P. 336,983). Naphthazarin derivatives (B.P. 316,950).—See IV. Wetting etc. agents (U.S.P. 1,750,198).—See VI. Fungicide (U.S.P. 1,760,000).—See XVI. Acetylmethylcarbinol and diacetyl (B.P. 337,025).—See XVIII. *tert*-Aminoalkyl aminoalkoxybenzoates (B.P. 317,296).—See XX.

#### IV.—DYESTUFFS.

See A., Jan., 37, Congo-acid sols (TANAKA). 58, Colorimetry without comparative standards (HOCK and MÜLLER). 75, Purely aliphatic *streptopentamethine* dyes (KÖNIG and REGNER). 82, Colours of substituted benzeneazophenols, including *o*-azophenols (HODGSON and ROSENBERG). 84,  $\beta$ -Naphthol-3-sulphonic acid (HOLT and MASON). Colour reactions of certain diamino- and nitroamino-diphenylmono- and -di-sulphides (HODGSON and ROSENBERG). 87, Di- and tri-arylamine derivatives (JOSZT and LESNIAŃSKI). 89, Chlorophenol-red (HARDEN). 96, Preparation of tetraethyldiaminodiphenylfurylmethyl chloride (MAHOOD and ALDRICH). 99, Diazotisation in the pyrazole series (REILLY and MAC-SWEENEY). 100, Bis-5-phenyl-2-pyrroleindigo (MADELUNG and UBERMANN). 103, Preparation of thiocyanine dyes (FISHER and HAMER).

#### PATENTS.

**Manufacture of [vat] dyes and [vat] dye intermediates [of the anthraquinone series].** W. SMITH, L. J. HOOLEY, and SCOTTISH DYES, LTD. (B.P. 336,983, 16.4.29).—The chlorine etc. in 2:2'-dichloro-, -dibromo-, or -disulpho-1:1'-dianthraquinonyl is replaced by hydroxyl, thiol (and disulphido-), imino-, amino-, substituted amino-, or aryloxy-groups by heating with metal hydroxides, sulphides, mercaptans, ammonia, amines, or phenols, especially in presence of copper salts. Ammonia, copper sulphate, and pyridine at 180° give flavanthrone. Products from aniline, sodium sulphide, calcium hydroxide, methylamine, aniline, 1-aminoanthraquinone, and phenol are also described. C. HOLLINS.

**Manufacture of derivatives of naphthazarin.** I. G. FARBENIND. A.-G. (B.P. 316,950, 6.8.29. Ger., 4.8.28).—Formaldehyde (or other aliphatic aldehyde) is added to the blue solution ("naphthazarin intermediate") obtained by reduction of 1:8- or 1:5-dinitronaphthalene in sulphuric acid. A black product is precipitated, which is useful as pigment for oil paints or lacquers, as a spirit dye, and in the wallpaper and stained paper industry. C. HOLLINS.

**Manufacture of chlorination products of isodibenzanthrone and their application.** A. G. BLOXAN. From Soc. CHEM. IND. IN BASLE (B.P. 336,775, 3.10.29).—Vat dyes which are insensitive to water are obtained by chlorinating isodibenzanthrone in presence of a halogen carrier (antimony pentachloride, iodine, ferric chloride) so as to introduce more than 2 chlorine atoms; e.g., with antimony pentachloride (3 pts.) in nitro-

benzene at 190–200°, or with sulphuryl chloride (or chlorine) and iodine in nitrobenzene at 100–105°, or with ferric chloride (6 pts.) in trichlorobenzene at 140–150°.

C. HOLLINS.

**Purification of vat dyes of the anthanthrone series.** I. G. FARBENIND. A.-G. (B.P. 316,149, 23.7.29. Ger., 23.7.28).—Halogenated anthanthrones are purified by grinding with 80–95% sulphuric acid or by diluting their solution in sulphuric acid to that concentration. In each case an insoluble (oxonium?) sulphate, readily hydrolysed by water, is formed and impurities remain in solution.

C. HOLLINS.

**Manufacture of azo dyes [pigments and ice colours].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 336,938, 10.7.29).—Non-sulphonated and non-carboxylated diazo, tetrazo, or diazoazo compounds are coupled in substance or on the fibre with 2:3-hydroxynaphthoic 4-alkoxy-2- or -3-methylanilides. Increased fastness to light due to the presence of the methyl group is claimed. Examples are: 6-methoxy-*m*-toluidide with diazotised 4-chloro-*o*-toluidine (red), *m*-xylidine (red), *m*-chloroaniline (scarlet), 2:5-dichloroaniline (brown-red), *m*-nitroaniline (orange-brown), *p*-nitroaniline → 2:5-dimethoxyaniline (blue-black), *o*-phenetidine →  $\alpha$ -naphthylamine (red-black); 5-methoxy-*o*-toluidide with diazotised 5-chloro-*o*-toluidine (blue-red), 2:4-dichloroaniline (dark red), 1-aminoanthraquinone (brown-red), dianisidine (coppered, dark blue). Other coupling components are the 5-ethoxy- and 5-butoxy-*o*-toluidides.

C. HOLLINS.

**Manufacture of direct-dyeing disazo dyes.** J. R. GEIGY A.-G. (B.P. 337,224, 15.11.29. Ger., 15.11.28).—3:3'-Diamino-4:4'-dihydroxydiphenylsulphone or its nuclear methyl or halogen derivatives are tetrazotised and coupled with 2 mols. of an aryl-J-acid. The dyes give, in presence of copper salts, violet shades on cotton.

C. HOLLINS.

**Manufacture of [acid] azo dyes [for wool].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,580, 14.6.29).—Milling dyes for wool are obtained by coupling a diazotised aniline, carrying in position 2 either hydrogen, halogen, alkyl, or alkoxyl, and in position 4 a hydroaromatic residue, with a sulphonated coupling component. Examples are: 4-amino-2':3':4':5'-tetrahydrodiphenyl → F-acid (red), G-acid (blue-red), toluene-*p*-sulphonyl-K-acid (claret), or H-acid ← *p*-nitroaniline (green-blue); 4-amino-3-methoxy-1':2':3':4':5':6'-hexahydrodiphenyl → N.W.-acid (red), G-acid (blue-red), H-acid (red-violet), 4-amino-1':2':3':4':5':6'-hexahydrodiphenyl → 1-*p*-sulphophenyl-3-methyl-5-pyrazolone (yellow), etc. Other first components are: 4-amino-3-methylhexahydrodiphenyl, 4-amino-3- or -4'-methyl-2':3':4':5'-tetrahydrodiphenyl, 4-amino-3-chloro- or -methoxy-2':3':4':5'-tetrahydrodiphenyl.

C. HOLLINS.

**Manufacture of substantive-dyeing o-carboxyazo dyes containing copper.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,971, 22.7.29).—Benzidine-3:3'-dicarboxylic acid is tetrazotised and coupled with 2 mols. of aminonaphtholdisulphonic acids (containing no *N*-substituent) and the resulting dye is coppered. The copper (other than a cuprammonium) salt may be

added during coupling. Examples of coupling components are: 2S-acid (green-blue), H-acid (green-blue), K-acid (blue), 2:8:3:6-aminonaphtholdisulphonic acid (grey; developed with *m*-phenylenediamine, grey, or  $\beta$ -naphthol, blue-grey), and 2S- and H-acids (blue).

C. HOLLINS.

**Manufacture of o-hydroxyazo dyes containing chromium.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,268, 26.7.29).—The dyes of B.P. 2956 of 1912 (B., 1912, 1072), or their deacylated products also obtainable by coupling a diazotised *o*-aminophenol-sulphonic acid with 1:7-aminonaphthol, are prechromed. [Stat. ref.]

C. HOLLINS.

**Manufacture of azo dyes [containing metal].** SOC. CHEM. IND. IN BASLE (B.P. 315,400, 21.6.29. Switz., 13.7.28).—An *o*-hydroxy- or *o*-carboxy-diazo compound is coupled with J-acid,  $\gamma$ -acid, or 2:5-aminonaphthol-1:7-disulphonic acid, with or without a middle component; the product is then treated with a metal compound and an oxidising agent in either order. Examples are: 4-chloro-*o*-aminophenol-6-sulphonic acid → J-acid, coppered, and oxidised with air, ferricyanide, persulphate, etc. (grey on cotton or viscose silk); 1:2:4-aminonaphtholsulphonic acid → J-acid, chromed, and oxidised with hypochlorite (green on wool or silk); *o*-aminophenol-4:6-disulphonic acid → J-acid, coppered, and oxidised with dichromate (grey on wool or silk); 4-nitro-*o*-aminophenol-6-sulphonic acid → J-acid, oxidised with hypochlorite, and coppered (greenish-grey); 4-chloro-*o*-aminophenol-6-sulphonic acid → J-acid → J-acid, coppered, and oxidised (grey).

C. HOLLINS.

**Manufacture of [direct] azo dyes containing copper.** SOC. CHEM. IND. IN BASLE (B.P. 315,664 and Addn. B.P. 315,665, 15.7.29. Switz., 14.7.28).—(A) A J-acid derivative of the type  $[C_{10}H_5(OH)(SO_3H) \cdot NH \cdot X]_2$ , where X is carbonyl, thiocarbonyl, or a heterocyclic residue (triazine, quinazoline, pyrimidine, etc.), is coupled alkaline with 2 mols. of a diazotised anthranilic acid or ester, or with 1 mol. of this and 1 mol. of a diazotised *o*-aminophenol, and the resulting dye is coppered in substance or on the fibre. (B) A J-acid derivative  $C_{10}H_5(OH)(SO_3H) \cdot NH \cdot X \cdot NH \cdot Ar \cdot N : N \cdot Ar'$ , where Ar is an aromatic residue and Ar' is a salicylic acid residue, is coupled in alkaline solution with a diazotised anthranilic acid or ester, and the dye is coppered as before. Examples are: (A) anthranilic acid or methyl anthranilate (2 mols.) → carbonyl-J-acid (coppered, red-brown on cotton); anthranilic acid → carbonyl-J-acid ← *o*-aminophenol-4-sulphonic acid (coppered, bordeaux-red); anthranilic acid (2 mols.) → product from cyanuric chloride, J-acid (2 mols.), and aniline (1 mol.) (coppered, red-brown); (B) 5-sulphoanthranilic acid → product of phosgenation of a mixture of J-acid and 4-amino-4'-hydroxyazobenzene-3'-carboxylic acid (coppered, yellow-brown).

C. HOLLINS.

**[Direct] disazo dyes [for cotton].** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 336,646, 12.7.29. Cf. U.S.P. 1,724,660; B., 1929, 974).—Direct orange to brown dyes are obtained by diazotising a non-sulphonated, non-carboxylated arylamine and coupling

through an aminobenzamidobenzoyl-J-acid as middle component with a sulphonyl- or carboxyaryl-pyrazolone or a sulphonated or carboxylated indole. Examples are: aniline, *m*-xylydine, or  $\alpha$ -naphthylamine  $\rightarrow$  4-*p*-aminobenzamidobenzoyl-J-acid  $\rightarrow$  1-*p*-sulphophenyl-3-methyl-5-pyrazolone or 2-methylindole-6-sulphonic acid.

C. HOLLINS.

**Manufacture of a green vat dye from 1:12-perylenequinone.** W. PENECKE, Assr. to F. BENSA (U.S.P. 1,783,137, 25.11.30. Appl., 5.1.28. Austr., 19.11.27).—See B.P. 300,922; B., 1930, 95.

**[Manufacture of an] isatin-carbazole compound vat dye.** M. P. SCHMIDT and O. LIMPACH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,783,588, 2.12.30. Appl., 8.7.29. Ger., 21.7.28).—See B.P. 316,099; B., 1930, 1060.

**Trimethylthionaphthen vat dyes.** E. HOFFA and H. HEYNA, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,782,724, 25.11.30. Appl., 30.3.28. Ger., 11.4.27).—See B.P. 288,554; B., 1929, 808.

**[Manufacture of] anthraquinonethioxanthone vat dyes.** A. LÜTTRINGHAUS and F. KAČER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,783,659, 2.12.30. Appl., 6.3.28. Ger., 10.3.27).—See B.P. 290,408; B., 1928, 667.

**Azo dyes.** E. FELLNER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,784,343, 9.12.30. Appl., 25.2.28. Ger., 17.8.22).—See G.P. 411,467; B., 1925, 624.

**Manufacture of [azo] dyes containing metals.** H. GUBLER, G. DE MONTMOLLIN, and J. SPIELER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,783,083—4, 25.11.30. Appl., 9.7.29. Switz., 14.7.28).—See B.P. 315,664—5; preceding.

**Condensation products of the naphthalene series.** (B.P. 336,800).—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Moisture relations of cotton.** VII. Hysteresis. A. R. URQUHART and N. ECKERSALL (J. Text. Inst., 1930, 21, T 499—510. Cf. Urquhart and Williams, B., 1926, 186; also A., 1929, 1140).—Absorption and desorption curves are merely boundaries of an equilibrium area, any point on which may be capable of representing the moisture regain of a sample of cotton under suitable conditions of humidity and pre-history. This is of importance in textile testing, since fixing the temperature and relative humidity at constant values may not be sufficient to ensure comparable conditions, and additional precautions may have to be taken if large differences of pre-history are possible. The desorption curve above 80% R.H. is itself indefinite, in that it varies with the amount of water previously in the cotton at the saturation point, but a definite curve can be obtained by removing water from cotton containing at least 40% of water. This aspect of the phenomenon must be considered when comparisons at high humidities are made. Cotton taken direct from the boll, or while still wet after kier-boiling or mercerising, is found to have an exceptionally high adsorptive capacity, and this is almost certainly accompanied by a high degree of swelling of cotton. Knowledge of this might prove of

advantage in subsequent processing. For example, kier-boiled and mercerised cloth dyed with direct sulphur, and vat dyes without previous drying gives darker shades and "brighter" hues than the same material air-dried or partly dried. B. P. RIDGE.

**Porosity; a primary property of textiles.** I. **Milling of wool fabrics.** II. **Thickness of cloths.** J. SCHOFIELD (J. Soc. Dyers and Col., 1930, 46, 368—372, 372—375).—I. Increase in thickness in regard to the milling of woollen fabrics has been investigated. In the acid-milling of a coarse serge fabric (East Indian wool-pulled worsted mixture), the linear shrinkages in warp and weft, increase in thickness, and decrease of porosity varied exponentially with the duration of milling. The processes of milling and felting are compared in relation to the effect on wool fabrics.

II. A method for measuring the thickness of a fabric by sandwiching it between two glass or metal plates is given, and suitable apparatus is described. From thickness measurements of a number of types of wool fabrics, and assuming that wool has  $d$  1.31, it is concluded that no fabric has one half of its volume occupied by the actual wool fibres, even with tightly spun and closely set worsteds; in heavily milled woollens of the beaver type the air space is about two thirds. The effects of various processes such as raising, milling, crabbing, and scouring on a woollen cloth, with particular regard to its change in weight and thickness, are given. A. J. HALL.

**Porosity; a primary property of textiles.** III. **Porosity of yarns.** IV. **Instruments and experimental methods.** J. SCHOFIELD (J. Soc. Dyers and Col., 1930, 46, 401—407, 407—409; cf. preceding abstract).—III. Formulae relating yarn diameters and (a) tension, (b) counts, are given, and yarn porosity is defined.

IV. Yarn diameter is measured by means of a "filament micrometer," which is described and illustrated by diagrams. B. P. RIDGE.

**Detection of fungus mycelium in mildewed cotton fabrics.** M. W. JENNISON (Science, 1930, 72, 346—347).—The presence of fungus mycelium in raw cotton, undyed yarns and fabrics can be quickly detected by differential staining with Pianese IIIb stain. The material is washed in water or alcohol, stained for 15—45 min., decolorised in acid alcohol, and dried. Cotton fibres stain green and the fungus mycelium a deep pink. L. S. THEOBALD.

**[Determination of] variations in lignin content of spruce wood.** P. KLASON (Svensk Pappers-Tidn., 1929, 32, 494—496; Chem. Zentr., 1930, i, 3814).—Methods for the determination of lignin in wood are discussed, and a procedure is specified.

A. A. ELDRIDGE.

**Paper-coating minerals and adhesives.** M. B. SHAW, G. W. BICKING, and M. J. O'LEARY (Bur. Stand. J. Res., 1930, 5, 1189—1203; cf. B., 1930, 858).—Four different clays and one compound of diatomaceous earth, casein, glue, and modified starches have been examined with regard to use in papermaking for high-grade printing. The clay coatings bound well to the fibre sheet and gave good printing-quality paper.

The coating with the diatomaceous earth compound did not adhere well to the base papers. The modified starches had not quite as strong adhesive quality as the casein or glue, but all coatings containing 18 pts. of starch per 100 pts. of clay were well bound to the body papers. Graded as to their clay suspending property, the adhesives were in the following descending order: starch, casein, glue. When the coated papers were printed by the half-tone process equally good results were obtained with the three different adhesives, although the starch-bound coatings may possibly have absorbed somewhat more ink. W. E. DOWNEY.

**Test for sulphite-cellulose extract.** GRASSER.—See XV.

See also A., Jan., 26, **Determination of texture of ramie fibres** (ECKLING and KRATKY). 74, **Cellulose xanthate** (LIESER).

#### PATENTS.

**Digesters for production of cellulose.** [Cascade] digestion of vegetable materials to obtain cellulose therefrom. E. C. BENTHALL and E. SPENCER (B.P. [A] 322,741 and [B] 322,754, 10.9.28).—(A) The digesters consist of cylindrical vessels containing a perforated plate near the bottom and one or more cone-shaped projections in the plate terminating below the upper level of the material being treated. The extraction liquid is circulated through the mass by means of vomit pipes which shower the liquid over the top of the charge. (B) In working with a series of digesters on the cascade principle the vomit pipes are closed before opening the lower valve of one digester to pass the liquid to the next; in this way the steam pressure in the upper part of the digester blows the liquid through the extracted mass, leaving it in a semi-dry state. A. R. POWELL.

**Treatment of cellulosic materials to render them more reactive towards esterification.** BRIT. CELANESE, LTD. (B.P. 338,745, 3.12.29. U.S., 11.12.28).—The material is treated first with 0.5–4% of nitric acid preferably in admixture with 75–330% of a lower aliphatic acid, e.g., glacial acetic acid, at 35–60° for about 1 hr. and then for a further hour at the ordinary temperature with 10–300% of an organic acid anhydride, e.g., acetic anhydride, in the absence of esterification catalysts other than the nitric acid used in the first stage of the pretreatment. The resulting material esterifies rapidly, and the products are of good colour and give solutions of high clarity. D. J. NORMAN.

**Manufacture (c) of derivatives of cellulose and (A, B) of artificial threads and other products from cellulose compounds.** L. LILIENFELD (B.P. 335,906, 335,993–4, 25.3.29).—(c) Alkali-cellulose is treated with a hydroxyalkylating agent (ethylene chlorohydrin,  $\alpha$ -chlorohydrin) and then xanthated. (A) Cellulose xanthate is treated with a hydroxyalkylating agent, and the product is formed into threads etc. and coagulated. (B) The product of (c) is formed into threads etc. and coagulated. C. HOLLINS.

**Production of lumen [acetate] silk.** O. VON KOHORN (O. KOHORN & Co.), and H. SCHUPP (B.P. 314,543, 27.6.29. Ger., 30.6.28).—Hollow cellulose ester filaments are produced at the normal dry-spinning temperature by adding

to the spinning solution a strongly acting cellulose ester coagulant of such a nature and in such quantity that immediately after extrusion and stretching there is formed at the surface of the filament a firm skin which becomes inflated by evaporation of the volatile solvent enclosed within the filament. Water and toluene, though ineffective separately, have the desired action when both are present. D. J. NORMAN.

**Manufacture of artificial materials from cellulose esters or ethers.** BRIT. CELANESE, LTD., R. P. ROBERTS, and L. W. GREGORY (B.P. 338,936, 29.7.29).—Filaments etc. obtained by coagulation of cellulose ethers or esters in aqueous media are improved both as regards tensile strength and transparency by impregnating them with high-boiling solvents, swelling agents, or plasticisers (diacetone alcohol, ethyl tartrate) and subsequently passing them round heated rollers while under tension. F. R. ENNOS.

**Production and treatment of textile and other materials made of or containing cellulose.** H. DREYFUS (B.P. 338,989, 31.5.29).—A cellulosic solution, e.g., viscose, is extruded into a coagulating bath consisting of an aqueous solution containing 20% or more of sulphonic acids (C<sub>6</sub> acids or upwards, e.g., benzenesulphonic acid) or of organo-mineral acids derived from mineral oxy-acids other than sulphuric acid by substitution of at least one, but not all, of the hydroxy-groups by organic radicals (e.g., methylphosphinic acid); or the filaments etc. after coagulation in the usual manner may be subsequently treated with the above solutions under tension, and finally washed. (Cf. B.P. 335,894; B., 1931, 59). F. R. ENNOS.

**Manufacture of artificial threads, hairs, bands, etc. from solutions of silk fibroin.** I. G. FARBENIND. A.-G. (B.P. 339,089, 14.11.29. Ger., 14.11.28).—Natural silk waste is dissolved in orthophosphoric acid, and the viscous solution so obtained is pressed through capillaries or nozzles to form threads, which are coagulated by means of an aqueous solution of an alkali salt of a weak acid in the presence of one or more alkali salts of a strong acid, and are afterwards stretched. F. R. ENNOS.

**Evaporative spinning of artificial silk.** NELSON'S SILK, LTD., and J. NELSON (B.P. 338,881, 25.7.29).—In the apparatus claimed the spinning head has an annular space closed at the bottom by a detachable sheet-metal jet having a ring of perforations through which the liquid is forced. Heated air is drawn through the spinning vessel by means of two pipes, one communicating with the space inside and the other with that outside the jet, whereby more efficient drying of the filaments is ensured. F. R. ENNOS.

**Elimination of gases from viscose silk cakes.** BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of NAAML. VENN. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 319,766, 5.9.29. Holl., 28.9.28).—The cakes are either superimposed with interposed plates, each having a central opening, while the uppermost cakes are covered over, or they are left in the spinning bowls, which are closed by tightly fitting covers. Air heated to at least 50° is then aspirated for a short time through the cakes. F. R. ENNOS.

**Production of fibrous material [from wood etc.].** D'A. M. CLARK. From RESTATS, INC. (B.P. 339,528, 11.1.30).—Waste wood or other ligneous material, preferably in a wet state and optionally after a mild digestion under pressure with water or caustic soda, is mixed with an abrasive agent such as sand or broken stone and ground to pulp under heavy pressure in a type of edge-runner. Wood of high resinous content may require two digestions, each followed by milling, but the cooking operations should be of such a character that the greater part of the gum and resin is retained by the fibre. The pulp, after separation of the abrasive agent, may be used for heat-insulating boards, or may be moulded to form panelling.

D. J. NORMAN.

**Solvent extraction of woods.** A. D. LITTLE, Assr. to A. D. LITTLE, INC. (U.S.P. 1,762,785, 10.6.30. Appl., 8.10.27).—Resins and terpenes are removed from coniferous woods, *e.g.*, longleaf yellow pine, by extraction with hydrocarbons of low average mol. wt. (28–78) which are gaseous at the ordinary temperature and pressure, *e.g.*, which have an absolute vapour pressure at 75° of 1000–20,000 mm. Hg. The butane fraction (propane 9%, butane 89%, pentanes 2%) from petroleum or natural gas is a cheap and effective solvent, and may be used at, *e.g.*, 75° under a pressure of 120 lb./in.<sup>2</sup> The wood is cut into chips of a size suitable for subsequent pulp-making and may contain 15–20% of moisture. Suitable plant is described in which provision is made for extracting the wood on the countercurrent principle, recovering the solvent, and distilling the extractives under reduced pressure. Average yields per ton for stump wood from long-leaf yellow pine are rosin 410 lb., turpentine 6.3 gals., pine oil 3.3 gals.

D. J. NORMAN.

#### Manufacture of [esterified] products from wood.

A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 336,969, 22.7.29).—Wood is acylated without appreciably affecting the combination between the cellulose and the other constituents of the wood by boiling the dried material for, *e.g.*, 15 hrs. under reflux with an organic acid anhydride, *e.g.*, 350% of acetic anhydride, in the absence of an inorganic catalyst. The wood may be powdered or treated in large sheets, in the latter case a superficial effect being obtained. The resulting wood ester after washing with acetone is insoluble in all solvents, but can be moulded at temperatures above 100°, giving products of high tensile strength and of improved resistance to water and light. Esterified wood gives only a faint colour reaction with phloroglucinol and hydrochloric acid.

D. J. NORMAN.

**Manufacture of pulp.** F. G. RAWLING, Assr. to MEAD PULP & PAPER CO. (U.S.P. 1,766,944, 24.6.30. Appl., 21.1.29).—The discoloration of chemical pulp (during drying is prevented by spraying dilute solutions of an acid-reacting salt, *e.g.*, an alkali bisulphate, bisulphite, dihydrogen phosphate, or alum on to the wet web of pulp before it reaches the suction box, in sufficient quantity to bring the  $p_H$  of the water to 5–7. Sodium bisulphate (nitre cake) solution (0.143 g. per litre) is particularly suitable.

D. J. NORMAN.

**Coating of material [for electrical conductors].** E. C. HALL, Assr. to WESTERN ELECTRIC CO., INC.

(U.S.P. 1,764,625, 17.6.30. Appl., 4.5.28).—A stranded core, *e.g.*, an electrical conductor, is passed through an aqueous suspension of fibrous pulp in contact with a revolving cylinder mould so that a narrow ribbon of pulp is formed with the core at the centre. This ribbon is then wrapped round the core to give a felted fibrous sheath. A surface film of, *e.g.*, cellulose acetate, nitro-cellulose, or varnish is then applied, penetration of the filming solution into the fibrous sheath being prevented by first impregnating the fibres with a liquid, *e.g.*, water, in which the coating agent is insoluble. Suitable apparatus is described.

D. J. NORMAN.

**Water-resistant paper.** Treating paper pulp to obtain paper of improved water-resistance. [Waterproof] paper-making. Greaseproof paper or paper which retains its strength in contact with grease. G. J. MANSON (U.S.P. 1,762,928–1,762,931, 10.6.30. Appl., [A, B, D] 14.3.27, [C] 24.10.27).—A pre-set aqueous dispersion of paraffin wax, or a mixture of this with other waxes or waterproofing agents, is used either alone or in conjunction with ordinary rosin size or a size made from oxidised rosin for imparting resistance to water and grease to paper and pulp products. A suitable emulsion, which gives good sizing effects for a wax content of 1–1½% in the finished paper and does not form spots, is prepared by agitating 33 pts. of wax with 40 pts. of water containing 4 pts. of sodium silicate (50% of dry solids) and 1 pt. of glue at 77° and then adding 2 pts. of alum dissolved in 20 pts. of water, the  $p_H$  of the final emulsion being 7–7.3. The wax particles, being coated with a film of silicic acid, show a retention of 75–90% on the machine, and do not foul the wires or felts. Paper sized with wax in this way has a good “rattle” and retains its ink-resistance and colour when exposed to ultra-violet rays.

D. J. NORMAN.

**Treatment of [pile] fabrics.** G. H. ELLIS, H. C. OLFIN, and E. E. WALKER, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,783,608, 2.12.30. Appl., 15.3.28. U.K., 12.9.27).—See B.P. 302,208; B., 1929, 202.

**Production of partially hydrolysed cellulose acetates.** S. I. VLES (U.S.P. 1,782,796, 25.11.30. Appl., 25.10.27. Holl., 20.7.27).—See B.P. 292,398; B., 1928, 637.

**Manufacture of cellulose [mixed] esters of both nitric acid and an organic acid.** F. BECKER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,771, 2.12.30. Appl., 7.1.28. Ger., 15.1.27).—See B.P. 283,595; B., 1929, 750.

**Preparation of textile fibres for spinning.** G. C. LAURENCY (B.P. 339,848, 25.1.30. Belg., 31.1.29).

[Securing two jets in series in] apparatus for manufacture of artificial filaments, threads, etc. BRIT. CELANESE, LTD., A. H. TIDMUS, F. A. REESON, and R. H. J. RILEY (B.P. 339,670, 5.9.29 and 5.6.30).

**Papermaking machines.** T. D. NUTTALL, and BENTLEY & JACKSON, LTD. (B.P. 340,087, 6.11.29).

**Separating fibre from air** (B.P. 339,514).—See I. Naphthazarin derivatives (B.P. 316,950).—See IV. Bleaching of bast fibres etc. (B.P. 339,550).

Colouring paper (B.P. 314,068).—See VI. Artificial wood (B.P. 316,224).—See IX. Dielectric material (U.S.P. 1,769,874).—See XI. Pyroxylin solutions (U.S.P. 1,768,253). Talking-machine records (B.P. 337,437).—See XIII. Purification of cellulose (B.P. 338,941).—See XXII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Theory of the dyeing of cellulose acetate silk.** E. CLAYTON (J. Soc. Dyers and Col., 1930, 46, 367—368).—Picramic acid dyes cellulose acetate silk to a reddish-brown shade which may be oxidised to black by means of sodium dichromate or to brownish-black by diazotisation and coupling with *m*-tolylenediamine. From the behaviour of picramic acid, picric acid, and 2-aminoanthraquinone towards cellulose acetate silk it is concluded that the dyeing process is one of adsorption (adsorption may play a small part) followed almost instantaneously by dissolution of the particles of the dye in the silk fibre. A true solution (in alcohol) of 2-aminoanthraquinone gives a shade on the silk which is 4—5 times as deep as that obtained with a suspension of equivalent concentration in the usual Turkey-red oil solution, but the dye may be removed by repeated extraction with alcohol or benzene in which the silk is not soluble. Dyed cellulose acetate is not an adsorption complex, since if a hot solution of cellulose acetate in acetone is coloured (dyed) with 2-aminoanthraquinone and then poured into excess of water the dye does not separate from the precipitated cellulose, whereas if poured into benzene (a solvent which precipitates cellulose acetate) a considerable proportion of the dye passes into the benzene. Any advances in the efficiency of dyeing methods applicable to cellulose acetate silk will probably depend on the ability to produce dyes which besides being soluble in cellulose acetate are either soluble in water or easily dispersed in an aqueous medium to give exceedingly fine suspensions.

A. J. HALL.

**Moisture relations in cotton.** URQUHART and ECKERSALL.—See V.

See also A., Jan., 58, **Colorimetry without comparative standards** (HOCK and MÜLLER).

## PATENTS.

**Bleaching, dyeing, etc.** H. WADE. FROM DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 323,397, 3.7.28).—The material to be treated is subjected in a vessel with a false bottom to the action of a stream of liquor from a centrally disposed spraying device, a second stream from a spray at the top of the vessel, and other streams emanating at various angles from sprays disposed around the sides of the vessel.

A. R. POWELL.

**Bleaching of bast fibres, especially flax, hemp, etc.** I. G. FARBERIND. A.-G. (B.P. 339,550, 15.2.30. Ger., 16.3.29).—The material is treated with the following reagents in the order named: (1) hydrogen peroxide solution containing 0.5—1 g. of active oxygen per litre and about 6% of sodium carbonate on the weight of fibre for about 12 hrs.; (2) hypochlorous acid of  $p_H < 5$  containing 1—8 g. of active chlorine per litre at the

ordinary temperature; (3) warm sodium carbonate solution (2—8 g. per litre) for about 1 hr.; (4) neutral or faintly alkaline hypochlorite solution (0.5—4 g. of active chlorine per litre); (5) hydrogen peroxide solution, containing 0.5—1 g. of active oxygen and 2—3 g. of sodium silicate per litre, for  $\frac{1}{2}$ —2 hrs. at 40—80°. For very resistant material, bath (4) may be faintly acid, in which case a further treatment with dilute sodium carbonate solution is necessary. The hydrogen peroxide from bath (5) may be used again in the first stage of the process.

D. J. NORMAN.

**Manufacture of coloured reserves in textile printing.** A. CARPMAEL. FROM I. G. FARBERIND. A.-G. (B.P. 336,968, 22.7.29).—White material is printed with reserve salt to which has been added a Rapid Fast colour (nitrosoamine and hydroxynaphthoic or acetoacetic arylamide), dried, and steamed in a rapid ager; it is then dyed in a roller vat and worked up as usual. The Rapid Fast colour appears upon a vat dye ground.

C. HOLLINS.

**Cleaning, wetting, and emulsifying [agents].** W. SCHRAUTH, Assr. to I. G. FARBERIND, A.-G. (U.S.P. 1,750,198, 11.3.30. Appl., 25.5.26. Ger., 30.5.23).—Naphthalene or tetrahydronaphthalene is sulphonated and condensed with an aliphatic or alicyclic alcohol above C<sub>4</sub>, e.g., amyl alcohol, cyclohexanol, methylcyclohexanol, hexyl alcohol.

C. HOLLINS.

**Brightening the colour of jute fibres.** J. BERLINERBLAU (B.P. 337,986, 12.11.29. Ger., 22.11.28).—The fibres, moistened with a 2—3% solution of hydrochloric acid, are treated with gaseous sulphur dioxide at normal pressure, the gas penetrating uniformly over the whole surface of the material.

H. ROYAL-DAWSON.

**Solution for fireproofing fibrous materials.** A. A. THORNTON. FROM SOC. ANON. INVENZIONI GUADAGNIN (S.A.I.G.) (B.P. 336,863, 10.12.29).—A solution containing ammonium chloride or carbonate, sodium chloride, boric acid, ammonia, and water is claimed.

H. ROYAL-DAWSON.

**Chemical treatment and washing of textile yarns in hank form.** BRYSLKA, LTD., and F. W. SCHUBERT (B.P. 323,360, 22.12.28).—The hanks are suspended on a series of laterally projecting arms on a roller chain which passes over a succession of baths containing the chemical and washing liquors. As the hanks come over each bath the latter is raised until the hanks are immersed, then lowered again after treatment to allow the material to drain.

A. R. POWELL.

**Dyeing [of cellulosic fibres].** A. BODMER, Assr. to HEBERLEIN & Co., A.-G. (Re-issue 17,894, 2.12.30, of U.S.P. 1,694,526, 11.12.28).—See B., 1929, 93.

**Coloration of materials made of or containing cellulose derivatives.** H. DREYFUS (U.S.P. 1,783,607, 2.12.30. Appl., 27.12.27. U.K., 23.11.26).—See B.P. 285,969; B., 1928, 331.

**Printing of fabrics.** C. DREYFUS (U.S.P. 1,783,606, 2.12.30. Appl., 30.6.27).—See B.P. 293,022; B., 1929, 811.

**Printing of wool with ester salts of leuco-compounds of vat dyes.** W. TAUSSIG, Assr. to DURAND,

& HUGUENIN (U.S.P. 1,784,768; 9.12.30. Appl., 12.10.28. Ger., 13.10.27).—See B.P. 298,648; B., 1929, 1012.

[Circulating system for] dyeing and like machines. C. E. OLDROYD (B.P. 339,909, 20.3.30).

Lecithin preparations (B.P. 317,730). Polyhalogenated fatty acids (B.P. 336,623).—See III. Chlorinated isodibenzanthrones (B.P. 336,775).—See IV. Waterproof fibrous materials (B.P. 315,839). Coloured rubber articles (B.P. 338,303).—See XIV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Production of mixtures of ammonium sulphate and nitrate. W. GLUUD, W. KLEMP, and F. BRODKORB (Ber. Ges. Kohlentech., 1930, 3, 170—196; Chem. Zentr., 1930, i, 3818—3819).—Optimal technical conditions for the production of mixtures of ammonium sulphate and nitrate from gaseous ammonia, 45—50 wt.-% nitric acid, and sulphuric acid ( $d$  1.70) have been determined. Calcium sulphate has been successfully substituted for sulphuric acid. A. A. ELDRIDGE.

Equilibrium between calcium phosphate, ammonia, and ammonium carbonate. N. D. PELIKH (J. Appl. Chem., Russia, 1930, 3, 351—360).—Ammonium hydrogen carbonate, particularly in the presence of carbon dioxide, reacts less readily than ammonium carbonate with calcium phosphate; in presence of ammonia partial precipitation of triammonium phosphate takes place, the yield not being proportional to the concentration of the solution.

### CHEMICAL ABSTRACTS.

Mechanical analysis of finely-divided natural phosphates. L. T. ALEXANDER and K. D. JACOB (U.S. Dept. Agric., Tech. Bull. No. 212, 1930, 24 pp.).—The coarse material is removed by sedimentation, a dispersion agent added, and the suspension set aside for a length of time calculated from the modified Stokes' formula,  $V = 301.84r^2/\eta$ , in which  $\eta$  is the viscosity of water at the temperature of the experiment (20—30°), so that particles 5  $\mu$  in diam. will have fallen 10 cm. By pipetting out from a depth of 10 cm. a portion of the suspension, the weight of particles < 5  $\mu$  may be determined. The percentage of particles > 50  $\mu$  is estimated by sieving, and that of particles from 50  $\mu$  to 5  $\mu$  by difference. It is possible that the method can be applied to the analysis of limestone and marl.

D. K. MOORE.

Preparation of basic cupric acetate from the neutral salt. Z. A. IOFA, S. M. KOBRIN, and L. L. KLYACHKO (J. Appl. Chem., Russia, 1930, 3, 366—374).—The salt  $2\text{Cu}(\text{OAc})_2 \cdot \text{Cu}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$  crystallises in blue prisms, whilst  $\text{Cu}(\text{OAc})_2 \cdot \text{Cu}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$  (obtained below 45°) forms sky-blue hairs, and  $\text{Cu}(\text{OAc})_2 \cdot 2\text{Cu}(\text{OH})_2$  forms square tablets. The salt  $\text{Cu}(\text{OAc})_2 \cdot \text{Cu}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$  is prepared from the normal acetate, calcium oxide, and copper sulphate. CHEMICAL ABSTRACTS.

Micrography of red oxides of iron. ANON. Cadmium yellow. FINK and GROSVENOR.—See XIII.

See also A., Jan., 22, Phosphorescent calcium sulphide (GOYLE and SINGH). 38, Titania gel (KLOSKY). 39, Optimum H : N ratio for ammonia

formation (GILLESPIE and BEATTIE). 41, Phase equilibria in the system  $\text{Cr}_2\text{O}_3$ — $\text{SiO}_2$  (BUNTING). System  $\text{Na}_2\text{SO}_4$ — $\text{NaF}$ — $\text{NaCl}$ — $\text{H}_2\text{O}$  (FOOTE and SCHAIRER). System  $\text{NaNO}_3$ — $\text{Na}_2\text{SO}_4$ — $\text{MgCl}_2$ — $\text{H}_2\text{O}$  (LEIMBACH). 49, Extraction of rubidium and caesium from carnallite (JANDER and BUSCH). 53, Preparation of pure rhenium compounds (KRAUSS and STEINFELD). 55, Separation of alkali halides (COUSIN and DUFOUR). Bromo-iodometry (VAN DER MEULEN).

### PATENTS.

[Centrifugal] atomisers for liquids [sulphuric acid etc.]. J. LÜTJENS (B.P. 324,565, 11.3.29).—The spindle of the atomising tray is jacketed in a tube the upper end of which forms with a supply cup and an inverted bell a seal in the acid flowing down the tube; the lower end dips into the acid in the atomising tray to form a second seal. The cup is divided into sectors by means of radial partitions which do not reach to the circumference of the bell which rotates with the spindle of the tray. A. R. POWELL.

Apparatus for storing or treating nitric acid. H. FRISCHER (B.P. 339,032, 26.9.29).—The portion of the apparatus which remains covered by the nitric acid may be made of aluminium alloys containing magnesium or silicon; the uncovered portion, which is exposed to fumes, is constructed of iron-silicon or chromium-nickel alloys. W. J. WRIGHT.

Elimination of silica in the treatment of natural silicates with acids to render soluble some of their constituents. F. JOURDAN (B.P. 339,028, 24.9.29. Ital., 22.10.28).—To reduce the amount of silica in solution, the temperature of reaction is raised to about 117° and energetic stirring is employed. The silica may be further reduced by adding to the acid about 40% of a salt identical with one of those produced in the reaction, and by acidifying the clear liquid just sufficiently to prevent salts in solution from separating. W. J. WRIGHT.

Manufacture of bleaching powder. E. KREBS (B.P. 338,940, 30.7.29).—To regulate the temperature in vertical absorbers and prevent the reaction from being restricted to only a few of the shelves, air is admitted at one or two points in the bottom of the absorber, whereby entangled chlorine is conveyed to the upper shelves. The chlorine may be introduced at various parts of the absorber, and may be drawn off at certain points, passed through a cooler, and re-admitted. If necessary, the absorber itself may be cooled by irrigation. W. J. WRIGHT.

Manufacture of anhydrous aluminium chloride. W. R. AUSTIN and J. N. SMITH, Assrs. to F. G. CLARK (U.S.P. 1,752,936, 1.4.30. Appl., 7.7.23).—A mixture of aluminous material and carbon is introduced into the arc of an electric furnace, a stream of chlorine being admitted simultaneously. Intensive local heating at 3600° is thus obtained, and the atmosphere of carbon monoxide and aluminium chloride vapour, surrounding the reacting agents, insulates the reaction zone from the walls of the furnace, so that the temperature of these may be as low as 100°. W. J. WRIGHT.



**Recovery of alkali xanthates.** W. HIRSCHKIND, ASSR. to GREAT WESTERN ELECTRO-CHEM. CO. (U.S.P. 1,753,787, 8.4.30. Appl., 28.11.24).—The mother-liquor obtained in the manufacture of alkali xanthates is treated with an alkali hydroxide to precipitate a further quantity of xanthate, a total yield of 80% being thus obtainable. The residual mother-liquor may be used for the froth-flotation of ores. W. J. WRIGHT.

**Preparation of cyanates.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 339,220, 19.3.30. Ger., 10.4.29).—Alkaline-earth compounds, or basic alkali compounds except the hydroxides, are caused to react at about 140° with urea. An alkaline-earth cyanate may be prepared first, and then this converted into the corresponding alkali cyanate by treating it in aqueous solution with an alkali compound. W. J. WRIGHT.

**Treatment of aluminous materials.** B. R. F. KJELLGREN (U.S.P. 1,752,641, 1.4.30. Appl., 23.8.24).—The material is heated with sulphuric acid at 150–200°, leached with water to remove soluble sulphates, and treated with an ammonium salt to produce ammonium alum, this being heated to recover ammonium sulphate by sublimation and to yield aluminium sulphate. W. J. WRIGHT.

**Treatment of aluminous materials for production of aluminium sulphate and alumina therefrom.** B. R. F. KJELLGREN (U.S.P. 1,752,599, 1.4.30. Appl., 17.8.25. Swed., 27.6.25. Renewed 22.7.29).—An improvement of the process described in U.S.P. 1,752,641 (preceding abstract) consists in heating the material with anhydrous ammonium sulphate in an atmosphere of sulphur dioxide and nitrogen formed by dissociation of ammonium sulphate, the ammonium alum being subsequently decomposed in a similar atmosphere. W. J. WRIGHT.

**Manufacture of chromium trioxide.** C. ARNOLD. From HARSHAW CHEM. CO. (B.P. 338,938, 30.7.29).—Oleum and an alkali compound, such as anhydrous sodium dichromate, are continuously introduced into an insulated and electrically-heated reaction vessel, provided with stirrers. The vessel is preheated to about 195°, but the reaction is maintained at 195–210° by the exothermic heat and controlled by the feed. The molten products pass from the bottom of the vessel through an exit pipe at the top into an insulated centrifuge or gravity separator, from which the lighter portions are led through a pipe to any suitable collecting vessel, and the heavier portions to a rotating drum or band conveyor, the solidified product being removed in flakes. W. J. WRIGHT.

**Manufacture of chromates.** O. F. TARR, ASSR. to MUTUAL CHEM. CO. (U.S.P. 1,752,863, 1.4.30. Appl., 3.4.29).—Chrome ore is roasted in an oxidising atmosphere with only about half its weight of burnt lime and with sufficient alkali carbonate to combine with only part of the chromium, the leached and dried product being then roasted with a further amount of alkali carbonate to combine with the remaining chromium. W. J. WRIGHT.

**Manufacture of phosphorus oxychloride and thionyl chloride.** G. J. SCHUDEL, ASSR. to ELKO

CHEM. CO. (U.S.P. 1,753,754, 8.4.30. Appl., 18.11.26).—A mixture of liquid sulphur dioxide and liquid chlorine is caused to react with phosphorus contained in a liquid carrier, which may consist of phosphorus oxychloride or a mixture of this with thionyl chloride from a previous operation, the products being separated by fractional distillation. W. J. WRIGHT.

**Purifying rare gases.** R. E. MIESSE, ASSR. to NEW PROCESS METALS CORP. (U.S.P. 1,753,298, 8.4.30. Appl., 3.1.27).—The gases are introduced into a glass bulb, in which are fixed two electrodes, one or both of these consisting of an alloy of cerium and lanthanum with traces of other rare-earth metals. On passing an electric discharge through the gases, particles of the alloy either combine with the impurities or take them up and deposit them on the walls of the container. W. J. WRIGHT.

**Preparation of catalytic gels.** E. B. MILLER and G. C. CONNOLLY, ASSRS. to SILICA GEL CORP. (U.S.P. 1,782,857, 25.11.30. Appl., 19.11.26).—See B.P. 280,934; B., 1929, 283.

**Production of colloidal silicic acid.** A. P. OKATOFF (U.S.P. 1,783,304, 2.12.29. Appl., 12.11.28).—See B.P. 328,241; B., 1930, 613.

**Apparatus for production of ammonia-air mixtures.** H. PAULING (U.S.P. 1,784,074, 9.12.30. Appl., 29.12.27. Ger., 15.10.26).—See B.P. 292,830; B., 1928, 641.

**Production of hydroxides of alkali metals.** E. HEINZE, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,784,066, 9.12.30. Appl., 26.3.26. Ger., 1.4.25).—See B.P. 290,719; B., 1928, 522.

**Manufacture of metallic nitrates.** F. A. HENGLEIN, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,647, 2.12.30. Appl., 28.11.27. Ger., 8.12.26).—See B.P. 306,998; B., 1929, 393.

**Recovery of sulphur from alkali polysulphides.** H. LANGHEINRICH, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,725, 2.12.30. Appl., 5.8.29. Ger., 23.8.28).—See B.P. 323,908; B., 1930, 325.

**Manufacture of iron carbonyl.** A. MITTASCH and C. MÜLLER, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,744, 2.12.30. Appl., 25.3.26. Ger., 30.3.25).—See B.P. 244,895; B., 1926, 191.

**Separation of cobalt and iron from solutions rich in iron as obtained by lixiviating roasted pyrites.** N. E. LENANDER, ASSR. to ORKLA GRUBE-AKTIEBOLAG (U.S.P. 1,783,046, 25.11.30. Appl., 27.6.29. Ger., 26.7.26).—See B.P. 329,543; B., 1930, 712.

**Cooling agents** (B.P. 339,194).—See I. Deoxidiser (U.S.P. 1,736,464).—See II. Hydrogen and acetylene (B.P. 337,088).—See III. Fireproofing solution (B.P. 336,863).—See VI. Thoriated tungsten (U.S.P. 1,764,644).—See X. Gas-filled tubes (U.S.P. 1,769,025).—See XI. Paint remover (B.P. 337,944).—See XIII. Fertilisers (B.P. 336,651, 337,402, and 337,415). Compound fertilisers and sodium bicarbonate (B.P. 338,007).—See XVI.

## VIII.—GLASS; CERAMICS.

## Influence of radiation on the coloration of glasses.

P. GILARD and A. LECRENIER (*Chim. et Ind.*, 1930, 24, 1035—1051).—As a result of exposing glasses of various compositions to the action of radium radiation the authors concluded that soda-RO-silica glasses assumed a brownish tint when RO was an alkaline-earth oxide, the corresponding potash glasses being somewhat more yellow; barium oxide gave greener tints than calcium or magnesium oxide; soda-lime-borosilicates acquired brownish-grey tints, whilst the corresponding potash glasses were bluish-grey. The violet colouring of irradiated pure fused silica was not the same as that of pure crystal quartz, nor was the intense coloration of glass with only 0.018% MnO wholly caused by a chemical effect of the rays on the manganese oxide. Rather was the cause physical, namely a rearrangement of the atomic groupings with corresponding effect on the absorption.

M. PARKIN.

## Use of fuel oil. LUBBOCK.—See II.

See also A., Jan., 18, Double-refracting "Corex" glass (RAYLEIGH). 36, Measurements with kaolin suspensions (REIFENBERG).

## PATENTS.

Coloured heat-resisting glass. W. C. TAYLOR, Assr. to CORNING GLASS WORKS (U.S.P. 1,754,065 and 1,754,182, 8.4.30. Appl., [A] 12.6.25, [B] 10.6.26).—Lithia  $1\frac{1}{2}$ —3%, an equal or greater amount of potash, with alumina 1—3%, and boric oxide less than twice the alkali, constitute with 79% or more of silica a low-expansion borosilicate glass which is capable of being coloured by the usual colouring oxides in the same way as an ordinary glass. A preferred base glass is  $\text{SiO}_2$  82.6,  $\text{B}_2\text{O}_3$  8.8,  $\text{K}_2\text{O}$  3.4,  $\text{Li}_2\text{O}$  3,  $\text{Al}_2\text{O}_3$  2.2%, which gives a green with 1.4% CuO and 0.012% CoO, yellow with 0.25% NiO, 0.2%  $\text{MnO}_2$ , and 0.6% of "uranium oxide" [? sodium uranate], and a blue with good absorption in other parts of the spectrum with 1% CuO and 0.1% CoO. In (B) the green glass is specially claimed.

M. PARKIN.

## Manufacture of marbled or multicoloured glass.

C. LOCREILLE (B.P. 339,583, 8.4.30. Belg., 9.4.29).—Glasses of different colours melted separately at 1000—1200° are poured into a mass of ground-colour glass of approximately the same composition and melted at a somewhat higher temperature (1000—1400°).

L. A. COLES.

## Non-discolouring safety glass. DU PONT VISCOLOID

Co. (B.P. 310,475, 26.4.29. U.S., 26.4.28).—Non-discolouring plasticising agents comprising, e.g., amyl acetate, ethylene glycol diacetate or ethyl ether, triacetin, alkyl phthalates, and not more than 12% of camphor, are used in the manufacture of the plastic layer having pyroxylin or a carbohydrate ester or ether as base.

L. A. COLES.

## Light filters for increasing and producing colour contrasts.

DEUTS. GASGLÜHLICHT-AUER-GES. M.B.H., Assecs. of F. WEIDERT (B.P. 338,334, 29.10.29. Ger., 15.12.28).—Glasses suitable for use as spectacle lenses or in illuminating systems or for accentuating colour contrasts contain 0.3 g.  $\text{Nd}_2\text{O}_3$  per  $\text{cm}^2$  of irradiated

surface. Ultra-violet light may be absorbed by adding ceric oxide, and the contrast can be further intensified in certain cases by adding other oxides, such as those of cerium or praseodymium.

M. PARKIN.

Compositions for coating [the interior surface] of vitreous articles. A. M. HAGEMAN and A. F. LINDSTROM, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,752,792, 1.4.30. Appl., 27.1.25).—Two preferred compositions are: (a) aluminium silicate (or kaolin) 100 g., sodium hydroxide 15 g., zinc phosphate substantially 50 g., with 1 litre of sodium silicate solution,  $d$  1.025; (b) aluminium silicate 50 g., zinc phosphate 50 g., with 1 litre of water. The ingredients are ground in a ball mill to a fine slip and applied to such articles as electric lamp bulbs to produce a light-diffusing layer. Suitable pigments may replace part of the "filler" (aluminium silicate) to give coloured effects. The coating is set by the momentary application of a relatively high temperature, after which it is baked at 300—400° for 3—4 min.

M. PARKIN.

## Insulating a glass sheet from a receiving surface.

Y. BRANCART (B.P. 338,477, 5.4.30. Belg., 20.4.29).—The receiving surface is insulated from a glass sheet produced by casting or rolling by the gaseous products of combustion from pulverised solid (e.g., charcoal, retort carbon, or poplar wood sawdust), liquid (e.g., petrol) or plastic fuel containing no gases which would injure the surface either applied to the surface or burned below it, the surface in the latter case being perforated. Alternatively, a solid rod coated with the fuel mixture is passed between the surface and the glass.

M. PARKIN.

Manufacture of refractory articles [for contact with molten glass]. HARTFORD-EMPIRE Co., Assecs. of P. G. WILLETTTS (B.P. 316,129, 7.6.29. U.S., 23.7.28).—Mixtures are used containing 35—88%  $\text{Al}_2\text{O}_3$ , 11—63%  $\text{SiO}_2$ , not more than 1.5% of oxides of iron, magnesium, and alkali metals, and sufficient fluxes (alkaline-earth oxides) to cause the sole formation of mullite or corundum crystals in a glassy matrix when the mixture is heated below the fusion point of the materials. Suitable mixtures comprise, e.g., Georgia Klondike white kaolin, Arkansas clay No. 23, and potters' flint.

L. A. COLES.

Manufacture of refractory materials from natural magnesium silicates. METALLGES. A.-G. (B.P. 337,605, 7.11.29. Ger., 9.11.28).—Ferruginous magnesium silicates (olivine, peridotite, serpentine, asbestos, etc.) are heated in an oxidising atmosphere with magnesia or substances yielding it in quantities at least sufficient to convert the iron into magnesium ferrite, the free silica into magnesium orthosilicate, and compounds of aluminium, chromium, etc., when present, into magnesium aluminate, chromate, etc. The initial mixtures may be worked to a paste with, e.g., sulphite-cellulose waste liquor and moulded under high pressure before firing.

L. A. COLES.

Glass-melting tanks. HARTFORD-EMPIRE Co., Assecs. of W. T. BARKER (B.P. 340,110, 30.11.29. U.S., 20.5.29).

Manufacture of glass sheets. CHANCE BROS. & Co., LTD., and A. L. FORSTER (B.P. 339,764, 20.11.29).

Continuously drawing tubes and rods of glass and the like. S. G. S. DICKER. From NAAML. VENN. MAATS. TOT EXPLOITATIE VAN UITVINDINGEN (B.P. 340,076, 21.10.29).

Moulding of articles in pure fused silica. QUARTZ & SILICE (B.P. 340,167, 13.2.30. Fr., 18.4.29).

[Press for] manufacture of objects of porcelain, earthenware, clay, and other ceramic masses. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRIZITÄTS GES. (B.P. 318,866, 10.9.29. Ger., 10.9.28).

Synthetic resins (B.P. 315,807—8, 338,002, and 338,024).—See XIII.

## IX.—BUILDING MATERIALS.

Cement chemistry in theory and practice. H. KÜHL (Cement, 1930, 3, 1297—1304, 1456—1463, 1607—1616).—The historical development of research on cement is surveyed, noting the work of Cobb (J.S.C.I., 1910, 29, 69) as the origin of the "dynamic" method of investigation. It has been found possible to correlate laboratory trials with the full-scale production by mixing the constituents in a small porcelain mill, burning the mixture in the form of small balls or rods on platinum in an electric furnace, and finally grinding the clinker in the porcelain mill. Quantities of 50—100 g. may be used and provide sufficient material for the small test-bars for the measurement of mechanical strength. It seems probable that at 800° only monocalcium aluminate and monocalcium silicate are present; conversion of the latter into dicalcium silicate begins at 900° and proceeds at a more rapid rate as the temperature rises, until at 1150° both mono-salts are converted into the dicalcium compounds. Dicalcium ferrite, forming at 1250°, is the essential preliminary to the formation of tricalcium silicate, and at 1500° dicalcium ferrite, tricalcium silicate, and dicalcium aluminate are the chief constituents. The spontaneous falling to powder of clinker burned at about 1250° is attributed to the increase in volume caused by the conversion of the  $\alpha$ - or  $\beta$ - into the  $\gamma$ -modification of dicalcium silicate. From a mineralogical study alite and celite may be regarded as the principal constituents of a normal Portland cement.

C. A. KING.

Overheating of aggregates [is] found detrimental to concrete. W. H. BACHELDER (Eng. News-Rec., 1930, 105, 973).—The highest strength of concrete was attained when the batch was laid at 21°. Approx. 20% of the strength was lost if the temperature was as high as 55°.

C. A. KING.

Asphaltic bitumen. JACKSON. Creosote oil. POPOV. Use of fuel oil. LUBBOCK.—See II.

## PATENTS.

[Electric kiln etc. for] manufacture of cement and lime. H. J. BENHAM (B.P. 316,715, 19.5.28).—The charge passes through a slightly inclined rotating tube furnace where it is preheated by the gases issuing from the shaft furnace into which the material is discharged from the rotary furnace. The shaft is provided with a series of baffles inclined downwards at 45° and

fixed alternately to opposite walls, and tapers towards its lower end into a constricted space in which the electrodes are situated. Below this chamber is a conveyor on to which the sinter falls for transfer to the crushers.

A. R. POWELL.

Manufacture of cement and other pulverulent or granular products. R. POURBAIX (B.P. 338,463, 19.2.30).—As an alternative to the use of paste mills the cement mixture is mixed with water in a diluter from which it runs in a number of streams on to an inclined screen, the coarser particles being washed off the screen into a collector and returned to the diluter. A series of screens may be used by perforating the bottom of the collecting tank of an upper screen and allowing the cement slip to fall in the form of jets on to a lower inclined sieve.

C. A. KING.

Cements, concretes, and mortars. J. W. BATTERSBY (B.P. 338,242, 27.8.29).—Prior to mixing with water, Portland cement or lime for mortar is mixed with 1—5% of a soluble hydrated alkali silicate, which may be ground together with cement clinker. For special use in sealing porous places the silicate may equal the cement in quantity.

C. A. KING.

Waterproof cements. E. L. GEORGE. From SOC. ANON. INVENZIONI GUADAGNIN (S.A.I.G.) (B.P. 336,860, 9.12.29).—The components consist of washed sand, resin, gypsum, alum, flaxseed oil, and water.

H. ROYAL-DAWSON.

Manufacture of waterproof cement. J. M. EVANS, ASSR. to G. W. WALLACE (U.S.P. 1,755,638, 22.4.30. Appl., 20.6.28).—Portland cement is mixed with, e.g., 5—25% of clay which has been used for treating petroleum or its fractions.

L. A. COLES.

Production of a cement-containing cold glaze for concrete, brickwork, etc. N.V. NEDERL. FABR. VAN BETON-EMAILLE "FORTOLIET" (B.P. 313,577, 14.6.29. Holl., 14.6.28).—A paste (1 pt.) containing a soluble soap and calcium chloride in about equal proportions is diluted with 10—15 pts. by wt. of water, and the whole mixed with double its weight of cement.

H. ROYAL-DAWSON.

Treatment of gypsum and alabaster to give it the appearance and hardness of marble. F. J. G. GARNER (B.P. 338,465, 28.2.30).—Native block gypsum etc., after being shaped, is heated to 150—200° and immersed in a solution containing magnesium sulphate 1 oz., calcium chloride (fused) 1 oz., concentrated sodium silicate solution 1 lb., and water 1 gal. The stone is then polished.

C. A. KING.

Manufacture of compositions containing wood fibre [artificial wood]. O. KAYSER (B.P. 316,224, 23.7.29. Ger., 25.7.28).—An aqueous suspension of wood pulp, e.g., coarse mechanical pulp of 5—12% consistency, is fed continuously on to a revolving cylinder mould in sufficient quantity to give a homogeneous board (thickness up to 25 mm.). Strong suction, e.g., 0.5 atm., is applied to the reverse side of the mould. The product shows no tendency to flake and can therefore be veneered.

D. J. NORMAN.

Preparation of waterproof board. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 319,213, 16.9.29).

U.S., 17.9.28).—The outer absorbent coverings of plaster board are treated on one or both sides, just prior to the introduction of the plaster core, with a thin layer of an aqueous dispersion of a waterproofing agent of such a type that complete coalescence of the dispersed waterproofing agent does not take until substantially the whole of the aqueous phase has been removed. This permits the plaster core to dry completely before the formation of a continuous film of the waterproofing agent. An aqueous dispersion of asphalt (m.p. 38–93°) with colloidal clay as the dispersing agent is suitable.

D. J. NORMAN.

**Treatment [insect- and fire-proofing] of wood.** F. S. VIVAS, Assr. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,768,175, 24.6.30. Appl., 8.10.27).—The wood is successively treated *in vacuo* with sodium carbonate solution (2½ lb./gal.), dried *in vacuo* to remove about 60% of the water, and treated under pressure with copper sulphate solution (½ lb./gal.) to precipitate  $\text{Cu}(\text{OH})_2 \cdot \text{CuCO}_3$  in the pores.

L. A. COLES.

**Impregnated wood and process of treating wood.** J. R. COOLIDGE, 3RD, Assr. to MONTAN, INC. (U.S.P. 1,766,606, 24.6.30. Appl., 3.6.27).—The wood is impregnated at, e.g., 82–93° and under 200 lb./in.<sup>2</sup> with a 20–40% solution containing about equal proportions of ammonium chloride (or sulphate), borax, ammonium phosphate, and, if desired, sodium chloride; the pores of the treated wood, after a partial drying, may then be sealed with soap solution. (Cf. U.S.P. 1,734,714–5; B., 1930, 146.)

L. A. COLES.

**Products for double-impregnation of wood.** SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 339,247, 2.9.29. Fr., 8.4.29. Addn. to B.P. 310,805; B., 1930, 1031).—The first impregnating solution in the prior process is replaced by a solution containing an alkali (sodium) sulphide as solvent for the arsenic sulphide (which may be formed *in situ*, as before), and, if desired, pyrolignite obtained by the action of an acetate on hydrophenolic compounds; the second impregnating solution is that described in B.P. 310,804 (cf. B., 1930, 1031).

L. A. COLES.

**Preparation of paving material.** G. H. ALVEY and E. A. YOUNG, Assr. to UVALDE ROCK ASPHALT CO. (U.S.P. 1,767,830, 24.6.30. Appl., 2.4.28).—The material comprises crushed (e.g., Uvalde) rock asphalt, a heavy-oil flux rich in constituents evaporating below 163°, and dilute acid.

L. A. COLES.

**Forming and using asphaltic or bitumastic materials for surfacing roads or waterproofing purposes.** F. A. HILL (B.P. 338,206, 16.8.29).—Asphaltic residues (35–55%) are mixed intimately with a fuel oil or petroleum product (65–45%) from which the lighter fractions including kerosenes have been distilled. The mixing is made preferably at above 100°.

C. A. KING.

**Manufacture of materials suitable for road construction, coating purposes, etc. from asphalt, tar, and the like.** J. Y. JOHNSON. From I. G. FARR-ENIND, A.-G. (B.P. 337,521, 4.9.29).—The tenacity, elasticity, and stability to cold of the products are improved by the addition of 0.1–20% (usually 0.5–5%) of synthetic polymerisation products of diolefines

(preferably of butadienes), prepared, e.g., as described in B.P. 312,201 (B., 1929, 612); these may be vulcanised or added in aqueous suspension or emulsion.

L. A. COLES.

**Accelerating the hardening of hydraulic binding means.** W. KIRCHNER, Assr. to CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER A.-G. (U.S.P. 1,782,471, 25.11.30. Appl., 21.3.27. Ger., 11.1.27).—See B.P. 275,897; B., 1927, 816.

**[Copperclad-felt] flashing and valley material for roofs etc.** INTERNAT. COPPERCLAD CO., Assees. of W. Mcl. SHAKESPEARE (B.P. 340,168, 17.2.30. U.S., 1.3.29).

**Dryers for wallboard** (B.P. 314,013, 314,982, and 337,432).—See I. **Fibrous material** (B.P. 339,528). **Solvent extraction of wood** (U.S.P. 1,762,785). **Products from wood** (B.P. 336,969).—See V.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Production of high-test cast iron.** R. MOLDENKE (Trans. Amer. Electrochem. Soc., 1930, 58, 385–401).—High-test cast iron (formerly misnamed “semi-steel”) is defined as any variety of cast iron of unusual strength and elasticity. The tensile strength must exceed about 30,000 lb./sq. in. of the transverse strength 2500 lb./in.<sup>2</sup>, with 0.25 in. deflection of the Standard Arbitration Test Bar. The constitution of such cast irons is discussed and the conditions necessary for their production are outlined. The advantages and disadvantages of the cupola, reverberatory furnace, open-hearth furnace, and electric furnace in the production of cast iron of this quality are examined at length. It is concluded that although the bulk of high-test cast iron will undoubtedly be made in the cupola, the higher grades, particularly of alloy cast iron, will be produced in the electric furnace. Future development will probably lie in the production of cast irons of special composition which are subsequently heat-treated. Thus a high-strength cast iron containing about 1.75% Si, after treatment for 5 hrs. at about 815°, had a tensile strength of over 100,000 lb./in.<sup>2</sup> and a high elasticity, and the fracture appearance was the same throughout sections of any thickness.

H. J. T. ELLINGHAM.

**Proposed method for accurately evaluating results of corrosion tests of ferrous metals.** K. PITTSCHNER (Trans. Amer. Electrochem. Soc., 1930, 58, 173–185).—The essential feature of the method consists in treating the corroded specimen of iron with excess of solid iodine in the presence of water. Metallic iron dissolves whereas the corrosion products are unattacked and can be filtered off. After driving off the excess of iodine, the iron is precipitated with ammonia solution, the ferric hydroxide dissolved in dilute hydrochloric acid and reduced with a slight excess of stannous chloride. Potentiometric titration with dichromate gives two end-points, the first corresponding to the excess of stannous chloride present and the second to the stannous and ferrous chlorides. Details of the titration are given. The application of the method to the determination of rates of corrosion of ferrous metals and the pro-

protective value of various coatings on iron and steel is discussed. H. J. T. ELLINGHAM.

**Destructive action of molten zinc, at and above galvanising temperatures, on metals and alloys.** VIII. **Action on copper, copper alloys, and high-silicon iron ("duriron" and "ferralt").** W. G. IMHOFF (Amer. Met. Market, 1930, 37, No. 140, 10, No. 141, 10; cf. B., 1930, 1114).—Copper, "everdur" (Cu 94.76, Si 4.13, Mn 0.74, P 0.06, Fe 0.04%), and the high-silicon iron are readily attacked.

## CHEMICAL ABSTRACTS.

**Detection of tellurium in bismuth.** H. TÖPELMANN (Z. anal. Chem., 1930, 82, 284—295).—The metal (10 g.) is dissolved in nitric acid and the solution evaporated with sulphuric acid to expel nitric acid. The residue is dissolved in 50 c.c. of hydrochloric acid and the solution treated with 1 mg. of arsenic as sodium arsenite and then with 5 c.c. of 10% stannous chloride solution. Next day the brown precipitate of arsenic and tellurium is collected, washed with dilute stannous chloride solution and then with distilled water, and dissolved in a few drops of nitric acid. The solution is evaporated to dryness and the residue transferred by means of a few drops of hydrochloric acid to a depression in a carbon electrode. A spectrograph is then taken with a condensed spark and the plate is examined for the presence of the line 2385.8 Å., which can be detected if the bismuth contains only 0.00005% Te. A. R. POWELL.

**Cemented tungsten carbide. Action of the cementing material.** L. L. WYMAN and F. C. KELLEY (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 354, 21 pp.).—Cobalt added as a binder becomes a solvent for the tungsten carbide grains. CHEMICAL ABSTRACTS.

**Effect of certain alloying elements on structure and hardness of aluminium bronze.** S. F. HERMANN and F. T. SISCO (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 365, 23 pp.).—Addition of cobalt, manganese, or silicon markedly increases the hardness of aluminium bronze, whilst the effect of nickel or iron is slight and erratic. The coefficient of equivalence of nickel with respect to the aluminium content is small; for iron it is zero, for manganese and silicon appreciable, and for cobalt negative. CHEMICAL ABSTRACTS.

**Aluminium-silicon-magnesium casting alloys.** R. S. ARCHER and L. W. KEMPF (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 352, 34 pp.).—Aluminium-silicon-magnesium alloys have good foundry characteristics, including ease of casting and relative insensitivity to high pouring temperature. Resistance to corrosion is greater than that of an aluminium-silicon-copper (4%) alloy, especially when the iron content is low and no precipitation heat-treatment is used. The density and thermal expansion are lower than in the aluminium-copper alloys, whilst the electrical and thermal conductivities are high. CHEMICAL ABSTRACTS.

**Plating on [steel] radiator shells.** O. P. WATTS (Trans. Amer. Electrochem. Soc., 1930, 58, 187—205).—Photomicrographs of cross-sections of composite plating (copper-nickel-chromium) on steel radiator shells are reproduced, and various types of faults are pointed out and related to the pretreatment of the steel surface and

the conditions of plating. Tests for pinholes in nickel and chromium plate and cracks in the latter were also carried out, and the results compared with those of immersion tests in lake-water and sea-water. Data are given showing the extent of local variations in the thickness of nickel and copper deposits under various conditions of plating. H. J. T. ELLINGHAM.

**Electrodeposition of iron-nickel alloys from cyanide solutions.** L. E. STOUT and J. CAROL (Trans. Amer. Electrochem. Soc., 1930, 58, 207—219).—An investigation has been made of the composition of iron-nickel alloys ( $x_A\%$  Ni) obtained by the electrolysis at various temperatures and current densities of solutions prepared by dissolving nickel cyanide, potassium ferrocyanide, and tartrate in water so as to give a total metal concentration (iron + nickel) of 14 g./litre, but with various proportions of iron and nickel ( $x_s\%$  Ni). The presence of tartrate prevents the formation of an insulating deposit on the anode and improves conditions at the cathode. When  $x_s < 60\%$ ,  $x_A$  remains practically constant (about 10% at 25°) except at very high current densities, when it increases somewhat, but when  $x_s$  exceeds about 60%,  $x_A$  increases rapidly especially at higher temperatures and current densities. For a given value of  $x_s$ ,  $x_A$  increases with current density, and at high current densities it also increases with temperature except when  $x_s$  is low, when  $x_A$  remains nearly constant. Values of the current efficiency of metal disposition are also recorded. Excess of free cyanide increases  $x_A$ . Thus with an addition of 75 g. of potassium cyanide per litre  $x_A$  reached 100% at 5 amp./dm.<sup>2</sup> when  $x_s$  was only 5%. The deposits were all light grey in colour, but tended to become darker and more lustrous with increasing iron content. Deposits obtained at 25° with low current densities had the best appearance. H. J. T. ELLINGHAM.

**Deposition of nickel-cobalt alloys.** C. G. FINK and K. H. LAH (Trans. Amer. Electrochem. Soc., 1930, 58, 241—249).—Determinations have been made of the composition of cobalt-nickel alloys electrodeposited at 1 amp./dm.<sup>2</sup> from solutions at 50° containing cobalt and nickel sulphates in various proportions, sodium chloride, and boric acid. The percentage of cobalt in the deposit increases rapidly with increasing proportion of this metal in the bath, and is also increased by raising the temperature, the current density, the  $p_H$  value of the bath, or its total metal concentration. Increasing the current density above 2 amp./dm.<sup>2</sup> or the  $p_H$  above 7 resulted in unsatisfactory deposits. Alloys of a whiteness most nearly approaching that of silver contained 25—45% Co, and were obtained by electrolysis at 1—2 amp./dm.<sup>2</sup> of a 1—2N-nickel sulphate, 0.1—0.4N-cobalt sulphate, 0.15N-sodium chloride, 0.3—0.5N-boric acid solution at 50°. Deposits containing over 25% Co are as hard as pure cobalt deposits, according to measurements by a specially devised scratch-hardness tester. By intermittent immersion tests in 2% sodium chloride solution it is found that the corrodibility of the alloys is a minimum when the metals are in equal proportions. H. J. T. ELLINGHAM.

**Deposition of nickel at a low  $p_H$ .** W. M. PHILLIPS (Trans. Amer. Electrochem. Soc., 1930, 58, 221—228).—

Nickel-plating from baths of  $p_H$  5.4—6.3, as recommended by the Bureau of Standards, has to be carried out at low current densities, and the production of deposits notably thicker than the usual  $0.8 \mu$  therefore takes a considerable time. It is now shown that with a bath of much lower  $p_H$  ( $< 3.0$ ) considerably higher current densities can be used without causing peeling or cracking at the edges. This increased range of permissible current density is particularly advantageous in the plating of irregularly shaped articles. Further advantages of the low  $p_H$  bath are that turbidity of the solution is avoided and corrosion of the anodes so greatly improved that the composition of the bath is maintained without periodic addition of nickel salts. On the other hand, there is at first a greater tendency for a low  $p_H$  bath to cause pitting, but it is believed that this disadvantage disappears when the bath has been in use for some time. Also the current efficiency of nickel deposition from such baths is lower than that from high  $p_H$  baths, falling to about 70% at  $p_H$  1.0. This decrease hardly affects current costs, but may result in undue building up of nickel in the bath. By replacing some of the nickel anodes by lead plates, however, this effect is easily corrected and there is the additional advantage that the lead anodes reduce the tendency to pitting. The  $p_H$  of such baths tends to increase gradually and is more difficult to control, but this is not serious because the permissible  $p_H$  range is very wide as compared with that of high  $p_H$  baths. For bright nickel-plating, the low  $p_H$  bath gives best results at low temperatures. Such baths are not suitable for plating on zinc-base die castings. Graphs are given to indicate the permissible range of current density for solutions of various  $p_H$  values at various temperatures. H. J. T. ELLINGHAM.

**Rapid determination of nickel in plating baths.** A. WOGGINZ (Chem.-Ztg., 1930, 54, 967).—The solution (25 c.c. containing 0.2—0.5 g. Ni) is diluted with an equal volume of water and treated with ammonium chloride and ammonia until a pale blue colour is produced. After transferring to a 250-c.c. graduated flask, the surface of the solution is covered with a thin layer of benzene, 25 c.c. of a 10% solution of sodium sulphide are added, and the aqueous solution is diluted to the mark and filtered through a dry paper. In 100 c.c. of the filtrate the excess sulphide is determined iodometrically in the usual way. A. R. POWELL.

**Practical difficulties associated with the electro-deposition of chromium.** J. W. CUTHBERTSON (Electroplaters' and Depositors' Tech. Soc. Advance copy. Oct., 1930).—The work is usually plated first with nickel, and in order that this underlayer may be quite sound and remain so during the subsequent deposition of chromium exceptionally thorough preliminary cleaning and scouring is necessary. In the deposition of chromium from a bath at  $40^\circ \pm 3^\circ$  in which the ratio, molarity  $\text{CrO}_3$  : normality  $\text{SO}_4$ , is about 50 or somewhat higher, the best results are obtained at 65—85 amp./ft.<sup>2</sup> Composition of the bath, current density, and temperature must be carefully controlled. Iron anodes are unsatisfactory in that their use tends to increase the concentration of trivalent chromium in the bath, thus decreasing the throwing power and narrow-

ing the range of conditions within which good deposits can be obtained. Lead is the only reliable anode material. The disposition of the anodes in the bath is important owing to the poor throwing power, but moving the work during plating causes no appreciable improvement. No attempt should be made to plate large and small work at the same time in one vat. Small articles should be wired separately, not put into a basket. Practical experiences in the plating of various types of article are given. Copper articles are best plated without a nickel undercoating, but the deposit is softer than usual and not so blue in colour.

H. J. T. ELLINGHAM.

**Heat-treatment of chromium deposits to increase their resistance to corrosion.** R. J. WIRSHING (Trans. Amer. Electrochem. Soc., 1930, 58, 251—254).—Heat-treatment at temperatures above  $90^\circ$  greatly increases the resistance to corrosion of chromium-plating directly deposited on copper, brass, or stainless steel. The resistance to corrosion of a chromium deposit on copper, as determined by the calcium chloride spray test, was increased sevenfold by treatment for  $\frac{1}{2}$  hr. at  $230^\circ$ .

H. J. T. ELLINGHAM.

**Electrodeposition of lead-thallium alloys.** C. G. FINK and C. K. CONARD, JUN. (Trans. Amer. Electrochem. Soc., 1930, 58, 279—284).—A fine-grained, adherent deposit of a lead-thallium alloy containing about 30% Tl can be obtained by electrolysis of a stirred perchlorate solution, containing 30 g. Tl and 5 g. Pb per litre with 20—30 g. of free perchloric acid, at 5 amp./ft.<sup>2</sup> at  $25^\circ$ . This alloy contains about the lowest percentage of thallium which will confer very marked acid-resisting properties. Alloys of other compositions can be obtained by varying the proportions of the metals in the electrolyte, but the actual concentration of thallium in the bath must not be much higher because the anode polarisation then becomes excessive and the cathode deposit poor. Raising the current density also causes unsatisfactory deposits.

H. J. T. ELLINGHAM.

**Calcium chloride testing of electroplated deposits.** H. C. MOURGY (Trans. Amer. Electrochem. Soc., 1930, 58, 265—270).—Since chromium-plated motor-car parts are often exposed to corrosion by calcium chloride used for laying dust or melting ice on roads, the introduction of a calcium chloride spray test is recommended as a guide in the improvement of chromium deposition for such service. There is no direct relation between the results of this test and those of the sodium chloride spray test, and it is not put forward as a new accelerated test of durability.

H. J. T. ELLINGHAM.

**Fuel oil.** LUBBOCK.—See II. **High-test grey iron.** MORKEN. **Throwing efficiency of electrolysis baths.** PAN.—See XI.

See also A., Jan., 32, **Iron-silicon alloys** (HAUGHTON and BECKER). 33, **Copper-tin alloys** (IMAI and OBINATA). 41, **System cobalt-chromium** (WEVER and HASCHIMOTO). **Carburising and graphitising reactions between iron-carbon alloys** (BECKER). 47, **Cathodic separation forms of tin** (FOERSTER and DECKERT). 53, **Oxides of iron and their reduction** (RODE). 57, **Electrolytic separation of antimony and copper** (HÖLEMANN).

## PATENTS.

**Heating furnaces suitable for metallurgical purposes.** (SIR) R. A. HADFIELD and R. J. SARJANT (B.P. 338,893, 26.6.29).—The furnace is furnished (preferably by forced draught) with recuperatively heated supplies of primary air below the firebars and secondary air at some point above the fire. Control valves connected by link work and a dash pot are provided so that when the secondary supply is decreased the primary is increased, and the control may be regulated by hand or effected automatically when the fire door is opened. B. M. VENABLES.

**Open-hearth furnace.** B. P. WHEELER (U.S.P. 1,769,362, 1.7.30. Appl., 17.6.21).—An open-hearth furnace is provided with an auxiliary air duct connecting with the gas port to form a combined air and gas port. The passage of air is assisted by means of a blower. C. A. KING.

**Centrifugal amalgamator and separator.** P. A. NEUMANN (U.S.P. 1,767,893, 24.6.30. Appl., 6.9.27. Can., 18.9.26).—The rotating drum has practically cylindrical walls surmounted by a trap in the shape of a pocket closed by a wide-meshed screen and having a cross-section like a C. The bottom of the basket is provided with directional vanes surmounted by a frustum of a cone the top of which is closed by a flat plate on to which the ore stream is directed by means of a vertical pipe. Mercury in a thin layer is maintained against the sides of the drum by centrifugal force and the ore stream is directed against this mercury by the cone and vanes. A. R. POWELL.

**Production of metals [cast iron].** J. E. FLETCHER (B.P. 338,535, 19.8.29).—Iron ore is smelted in a large rotary furnace, and from time to time portions of the charge are transferred to a smaller rotary furnace in which it is further heated until the metal and slag are sufficiently fluid for tapping. A. R. POWELL.

**Cyanide pot [for steel hardening].** V. P. RUMELY, Assr. to HUDSON MOTOR CAR Co. (U.S.P. 1,769,313, 1.7.30. Appl., 4.12.28).—The steel pots are immersed before use in a solution containing 12 lb. of iron filings, 10 gals. of phosphoric acid, and 125 gals. of hot water. When so treated the vessels have a surface coating which contains iron phosphate and the working life is increased by 50%. C. A. KING.

**Annealing of crystalline substances [e.g., steel].** E. G. HERBERT (B.P. 338,511, 16.8.29).—The substance after hardening by cold-work or by quenching is further hardened by placing it in rotating magnetic field, which sets up internal alternating stresses. A. R. POWELL.

**Heat treatment for hardened steel.** VEREIN. STAHLWERKE A.-G. (B.P. 314,056, 27.5.29. Ger., 22.6.28).—Hardened steel is cooled below 0° to suppress elongation caused by the conversion of austenite into martensite, then reheated at 100–150° for 1–8 hrs. to suppress the contraction which takes place in the first stage of the martensite transformation. A. R. POWELL.

**Heat treatment of [high-speed tool] steel.** W. B. SULLIVAN, Assr. to CHROBALTIC TOOL Co. (U.S.P. 1,766,314, 24.6.30. Appl., 2.6.26).—The steel is soaked

at a temperature between the Ac<sub>3</sub> and Ar<sub>3</sub> points for sufficient time to allow the transformation to go to completion, quenched, and tempered to produce the required hardness. A. R. POWELL.

**Heat treatment of alloy [non-rusting] irons and steels.** P. R. KUEHNICH, and DARWINS, LTD. (B.P. 338,912, 28.8.29).—Chromium steels containing more than 1% C, less than 1% Ni, and 1% Si are annealed at 1150–1250° in thin sheets and quenched in a solution of calcium chloride, preferably cooled below 0°; the hot metal may also be stamped into shape and cooled in the die or press. A. R. POWELL.

**Stable-surface alloy steel.** R. P. DE VRIES (U.S.P. 1,763,421, 10.6.30. Appl., 20.1.26).—Steel resistant to scaling up to 950° contains 1–10% Cr, 0.5–6% Si, 0.2–6% Al, 0.5–5% Cu, and 0.05–1% C. The preferred composition is 4% Cr, 2% Si, 2% Al, 1.5% Cu, and 0.36% C. A. R. POWELL.

**Production of low-carbon [ferro]-alloys [containing silicon].** F. A. FAHRENWALD (U.S.P. 1,757,298, 6.5.30. Appl., 29.5.26).—A mixture of steel together with the high-carbon ferro-alloy is melted in the presence of silica, and the temperature is increased so that the affinity of oxygen becomes greater for carbon than for silicon. C. A. KING.

**Changing the properties [increasing the permeability] of silicon steel.** J. C. KARCHER, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,752,490, 1.4.30. Appl., 4.5.28).—The steel, in the form of sheets, is annealed at 1000–1250° for 1–12 hrs., cooled to 718° at the rate of 10°/min., then rapidly cooled by placing it between two cold copper plates. (Cf. U.S.P. 1,677,139; B., 1928, 676.) A. R. POWELL.

**[Inhibitor for] corrosion prevention of iron and steel [in pickling baths].** M. M. HARRISON (U.S.P. 1,766,902, 24.6.30. Appl., 14.7.28).—A derivative of ethylenediamine, especially the *s*-diphenyl compound, is claimed. A. R. POWELL.

**Steel sucker rod and the like and its production.** W. J. CROOK, Assr. to EMSCO DERRICK & EQUIPMENT Co. (U.S.P. 1,764,068, 17.6.30. Appl., 8.1.27).—Steel containing 0.26–0.5% C and 0.5–1% Mn is annealed at 700–930° until it has an austenitic structure, then quenched in oil to produce a martensitic structure. The resulting metal has a high tensile strength, ductility, and resistance to fatigue; it is suitable for making sucker rods for oil-well pumps. A. R. POWELL.

**Production of metal [copper] films [on iron and steel].** W. E. WATKINS, Assr. to COPPER PLATE SHEET & TUBE Co. (U.S.P. 1,766,417, 24.6.30. Appl., 6.1.27).—The metal to be coated is painted with a mixture of a heavy oil and finely-divided copper or copper oxide and carbon, and then heated slowly to 1000° to burn off the oil and cause the copper to sinter and adhere in a continuous film to the iron. A. R. POWELL.

**Flotation process [for sulphide ores].** G. LUTZ, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,765,308, 17.6.30. Appl., 30.1.28).—The use of an alkyl naphthalene-sulphonic acid, in which the alkyl group contains at least two carbon atoms, is claimed as a frothing agent. A. R. POWELL.



**Roasting of [sulphide ore or flotation concentrate] fines.** H. J. CORDY and W. J. BURGOYNE, ASSRS. to GEN. CHEM. CO. (U.S.P. 1,758,188, 13.5.30. Appl., 30.1.28).—The finely-divided ores etc. are suspended in an oxidising gas and introduced into a roasting chamber into which a supplementary supply of the gas is injected adjacent to the walls to prevent "scarring" and/or the formation of accretions on the chamber walls.

C. A. KING.

**Concentration of oxidised ores of lead, silver, and copper.** E. H. SNYDER and W. D. GREEN, ASSRS. to COMBINED METALS REDUCTION CO. (U.S.P. 1,762,364, 10.6.30. Appl., 25.1.28).—Each ton of the oxidised ore is ground with 200–400 lb. of sodium chloride and 7–12 lb. of sodium sulphide and agitated in a 2 : 1 pulp ratio in a flotation machine with or without the addition of oil or other frothing agent.

A. R. POWELL.

**Treatment of refractory auriferous ores.** C. L. VOTAW and C. A. ANDREWS (U.S.P. 1,766,767, 26.6.30. Appl., 25.4.28).—The ore is ground wet with sodium cyanide, hydroxide, and bicarbonate, and, after drying, the mixture is smelted with lead material in the usual way.

A. R. POWELL.

**Brazing rod.** A. R. LYTLE, ASSR. to U. C. & C. RES. LABS., INC. (U.S.P. 1,764,571, 17.6.30. Appl., 7.9.28).—An alloy of 68–73% Cu, 30.75–23.5% Zn, up to 1% Sn, and 1.25–2.5% P is claimed. The alloy melts at about 800° and requires little or no flux when used for brazing brass.

A. R. POWELL.

**Preparation of zinc dust.** H. A. GRINE, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,762,716, 10.6.30. Appl., 29.11.27).—Zinc is distilled in a horizontal cylindrical retort and the vapours are passed through a conical jet into a large condensing chamber filled with inert gas. The pressure in the retort forces the vapour in a powerful jet into the condenser, where it is rapidly cooled so as to produce extremely finely-divided zinc dust.

A. R. POWELL.

**[Zinc] alloy.** C. PACK and J. C. FOX, ASSRS. to DOEHLER DIE-CASTING CO. (U.S.P. 1,767,011, 24.6.30. Appl., 8.8.27).—A die-casting alloy containing more than 75% Zn, 1–15% Al, 1–10% Cu, and less than 5% Ni and 5% Li is claimed. The lithium prevents the disintegration of the castings on ageing or on exposure to moist atmospheres and refines the grain structure.

A. R. POWELL.

**Galvanising process.** C. PLETSCH (U.S.P. 1,755,559, 22.4.30. Appl., 15.5.24).—Articles are cleaned by pickling in acid, then treated with a solution containing zinc chloride, any free acid introduced being neutralised, and dried; finally molten zinc containing small quantities of aluminium and tin is applied by usual methods.

C. A. KING.

**Applying a metal coating to aluminium by galvanic means.** M. K. DE TRAIRUP (B.P. 339,339, 3.9.29).—The aluminium article is plated at 1.5 volts for a few min. in a bath containing 10 g. of sodium hydroxide, 10 g. of sodamide, 2 g. of stannous chloride, 0.5 g. of zinc chloride, 0.25 g. of mercuric nitrate, and 0.5 g. of potassium hydroxide per litre, whereby it becomes plated with a thin uniform adherent coating

of zinc-tin amalgam on to which gold, silver, or copper can be deposited from any of the usual baths.

A. R. POWELL.

**Heat treatment of wire and strip prior to galvanising or other form of surface treatment.** L. D. WHITEHEAD (B.P. 339,155, 6.1.30).—The wire or strip is passed through horizontal tubes (with slightly upturned ends) containing fused metal, salts, etc. for annealing or cleaning purposes. A furnace is described in which the tubes are embedded in cast iron turnings and supported on cast iron plates which are protected by thin tiles of refractory material forming the roof of a heating flue.

B. M. VENABLES.

**Surface protection of magnesium alloys.** R. E. LOWE, ASSR. to DOHERTY RES. CO. (U.S.P. 1,765,341, 17.6.30. Appl., 14.3.27).—The alloy is cleaned with 10% nitric acid, then immersed in a 5% solution of phosphoric acid until coated with a black film, washed, and stored in a 5% solution of ammonium phosphate for 2–5 days. After washing and drying, the article is heated to obtain good adherence of the film of magnesium ammonium phosphate.

A. R. POWELL.

**Manufacture of aluminium.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 338,668, 4.7.29).—A fused bath comprising 14% of sodium chloride, 6% of potassium chloride, and 80% of aluminium chloride is electrolysed at 100°, using a rotating cathode and a current density of 1 amp./dm.<sup>2</sup> Alternatively, the bath may consist of 82% of aluminium chloride and 18% of sodium chloride; in this case a current of 4.2 amp./dm.<sup>2</sup> may be used at 160°.

A. R. POWELL.

**Preserving aluminium from attack by alkalis.** G. ADOLPH (B.P. 336,854, 29.11.29).—Water glass or other alkali silicates and hydrogen peroxide or compounds which on decomposition afford hydrogen peroxide are added separately or together to alkaline solutions used in contact with aluminium.

H. ROYAL-DAWSON.

**Aluminium alloy.** O. KAMPS (B. P. 339,469, 25.11.29).—The alloy consists of aluminium with 0.3–1.5% Ni, 0.05–0.9% Th, and up to 6.8% of a 10% cerium-copper alloy. The molten alloy is very fluid and can be used for making thin-walled sand castings which, after heat treatment at 535–480°, quenching in a barium chloride bath, and ageing at 50–300°, preferably 180°, have a tensile strength of over 40 kg./mm.<sup>2</sup> with an elongation of 15%.

A. R. POWELL.

**[Aluminium bronze] alloy.** J. V. O. PALM, ASSR. to CLEVELAND GRAPHITE BRONZE CO. (U.S.P. 1,764,034, 17.6.30. Appl., 1.10.26).—The alloy consists of 85–90% Cu, about 4% Al, 5–8% Zn, and 1–2% Fe.

H. ROYAL-DAWSON.

**Treatment of tin-containing material.** MITSUBISHI KOGYO KABUSHIKI KAISHA (B.P. 316,177, 27.5.29. Jap., 24.7.28).—Low-grade tin ore is mixed with pyrites and the mixture heated at 1000–1100° in a rotary kiln fired with producer gas, whereby the tin sublimes as sulphide.

A. R. POWELL.

**Metallurgy of tin [electrolytic reduction of cassiterite].** W. O. SNELLING (U.S.P. 1,766,463, 24.6.30. Appl., 5.3.28).—Finely-ground tin ore is subjected to an

electrolytic reduction treatment, leached with a solvent for metallic tin, *e.g.*, hydrochloric acid, and again ground and leached. The second grinding operation removes films of insoluble impurities from the cassiterite particles so that these particles are freed for further reduction.

A. R. POWELL.

**Preparing metal for painting.** J. H. GRAVELL, Assr. to AMER. CHEMICAL PAINT Co. (U.S.P. 1,765,331, 17.6.30. Appl., 23.5.29).—The surface of the metal is rubbed with a solution of ammonium dihydrogen phosphate containing ethyl and butyl alcohols, which removes both rust and oil.

A. R. POWELL.

**Agent for protecting metals against the effects of frost.** G. ERDBRÜGGER (B.P. 336,739, 9.9.29).—The composition consists of an intimate mixture of ground carnallite and liquid bitumen or crude mineral oil.

H. ROYAL-DAWSON.

**Oxidation inhibitor [for metals].** R. S. DEAN, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,764,609, 17.6.30. Appl., 12.5.24).—In order to prevent oxidation and scaling of a metal surface during heat treatment it is first sprayed or painted with a 5% colloidal suspension of boron trioxide in carbon tetrachloride peptised with 1% of 95% alcohol. The thin film of fused boron trioxide which forms during the heat treatment is adherent and impermeable to gases.

A. R. POWELL.

**Thoriated tungsten filament.** J. H. RAMAGE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,764,644, 17.6.30. Appl., 27.6.26).—A solution of ammonium tungstate and fluoride or oxalate is boiled and poured into an excess of concentrated hydrochloric acid containing thorium nitrate equivalent to the fluoride or oxalate in the tungstate solution. The resulting mixture of tungstic acid and thorium oxalate or fluoride is washed, dried, and reduced in hydrogen, and the metal is pressed into bars which are sintered, swaged, and drawn into wire.

A. R. POWELL.

**Applying the basic colour of wood to metal which is then to be imprinted with the grain of the wood.** MASA GES. M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 319,283, 18.9.29. Ger., 19.9.28).—The metal surface is coppered or browned electrolytically before being printed or sprayed with the basic colour of the wood and grained.

H. ROYAL-DAWSON.

**Apparatus for agglomerating and roasting minerals.** A. DE SAMSONOV, Assr. to SOC. POUR L'ENRICHISSEMENT ET L'AGGLOMERATION DES MINERAIS SOC. ANON. (U.S.P. 1,784,658, 9.12.30. Appl., 23.12.27. Belg., 4.1.27).—See B.P. 283,133; B., 1929, 58.

**[Treatment of iron for manufacture of] metallic apparatus for carrying out chemical and other processes.** F. LAPPE, E. KOCH, M. PIER, and H. SAUER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,726, 2.12.30. Appl., 23.3.27. Ger., 25.3.26).—See B.P. 293,077; B., 1928, 644.

**Purification of pig iron.** K. HOFMANN, Assr. to F. KRUPP A.-G. (U.S.P. 1,782,923, 25.11.30. Appl., 9.5.29. Ger., 25.5.28).—See B.P. 312,361; B., 1930, 1114.

**Magnetic materials [iron-nickel alloys].** G. W. ELMEN, Assr. to WESTERN ELECTRIC Co., LTD. (U.S.P.

1,768,237, 24.6.30. Appl., 5.8.25).—See B.P. 263,207; B., 1927, 144.

**[Copper-nickel-zinc] alloy.** C. PHILIPPOSIAN (U.S.P. 1,783,139, 25.11.30. Appl., 14.8.29. Switz., 3.11.28).—See B.P. 325,131; B., 1930, 332.

**Recovery of tin contained in the residues of tin-plate manufacture.** J. LALEY, Assr. to PETITS FILS DE F. WENDEL & CIE. (U.S.P. 1,784,490, 9.12.30. Appl., 3.4.28. Fr., 23.1.28).—See B.P. 304,639; B., 1929, 288.

**Improving the resistance to corrosion of magnesium and its alloys.** A. BECK and G. SIEBEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,770, 2.12.30. Appl., 30.1.29. Ger., 2.2.28).—See B.P. 305,197; B., 1929, 857.

**Highly active [metallic] catalyst.** K. MARX, H. BEHNCKE, and K. BRODERSEN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,662, 2.12.30. Appl., 29.11.27. Ger., 14.12.26).—See B.P. 282,410; B., 1928, 452.

**Cleaning of metal surfaces.** C. F. DINLEY (B.P. 340,047, 28.9.29).—See U.S.P. 1,729,765—7; B., 1930, 18.

**Electrolytic apparatus for refining aluminium and for like processes.** P. L. HULIN, Assr. to Soc. ANON. COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES, & CAMARGUE (U.S.P. 1,782,616, 25.11.30. Appl., 19.6.28. Ger., 4.7.27).—See B.P. 293,353; B., 1929, 290.

**Electrolytic production of light metals [particularly magnesium].** C. ARNOLD. From DOW CHEM. Co. (B.P. 339,833, 11.1.30).—See U.S.P. 1,755,380; B., 1931, 29.

**Apparatus for electrolytically depositing metal on metal tubes.** F. G. MARTIN and W. RAMSEY, Assrs. to J. STONE & Co., LTD. (U.S.P. 1,783,885, 2.12.30. Appl., 29.9.27. U.K., 22.4.27).—See B.P. 299,100; B., 1929, 739.

**Electrodeposition of metals.** J. HOLLINS, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,782,614, 25.11.30. Appl., 22.1.29. U.K., 25.2.28).—See B.P. 314,558; B., 1929, 687.

**Casting of metals [aluminium and its alloys].** BIRMINGHAM ALUMINIUM CASTING (1903) Co., LTD., and C. VAUGHAN (B.P. 339,721, 22.5.30).

**Mercury boilers (B.P. 319,220 and 339,237).—See I. Fractionation of ores etc. (B.P. 318,966).—See II. Apparatus for nitric acid (B.P. 339,032). Alkali xanthates (U.S.P. 1,753,787).—See VII. Metal-melting furnace (U.S.P. 1,752,887). Contact element (U.S.P. 1,769,229). Electrodeposition of metals (U.S.P. 1,768,358). Making articles by electrodeposition (B.P. 339,428). Loaded copper conductors (B.P. 338,169).—See XI. Metallic paints (B.P. 337,682). Rust-preventive paints (B.P. 314,499).—See XIII.**

## XI.—ELECTROTECHNICS.

**Detroit rocking electric furnace as applied to the production of high-test grey iron.** C. H. MORKEN (Trans. Amer. Electrochem. Soc., 1930, 58, 229—240).—The Detroit indirect arc furnace is described and the advantages of the rocking action in the production of

high-test grey iron are discussed. The furnace may be used for straight cold-melting of various charges, including iron borings without briquetting, for duplexing cupola iron, and for working up cold materials such as ferro-alloys or steel scrap with molten cupola iron. Some features of these different processes are discussed in relation to the composition and properties of the product and the cost of operation. The success of this type of furnace in this field is attributed to its simplicity of operation and control and to its ability to utilise scrap material of low market value.

H. J. T. ELLINGHAM.

**Effect of various factors on the output of Leclanché cells.** V. A. KOSTJEEV (Trans. Amer. Electrochem. Soc., 1930, 58, 155—172).—An investigation has been made of the effect of such factors as the nature, concentration, and quantity of electrolyte, and the temperature on the output of Leclanché cells made up under identical conditions using a depolarising mix consisting of 4 pts. of Caucasian pyrolusite ( $91.1\% \text{ MnO}_2$ ) and 1 pt. of graphite enclosed in a muslin bag. The quantity of electricity furnished by a cell during discharge through an approximately constant resistance until its terminal voltage had fallen to 0.75 volt was measured in each case, and the percentage utilisation of the manganese dioxide in the cell calculated on the basis of the reaction  $2\text{MnO}_2 + \text{H}_2\text{O} = \text{Mn}_2\text{O}_3 + 2\text{OH}' + 2\oplus$ . A "factor of quality" was obtained by multiplying this percentage utilisation by the number of amp.-min. of electricity obtained per 10 g. of depolarising mix. After remaining idle for an hour, the cell was again discharged to the same terminal voltage, and a second factor of quality calculated. Using cells with an ammonium chloride electrolyte, it was found that increasing the concentration of the solution increases the first factor of quality, the variation of the second factor being irregular, whereas increasing the volume of the electrolyte with a constant amount of depolariser raises both factors. The first factor is also increased by raising the temperature or decreasing the thickness of the depolarising mass around the carbon rod. Annealing the zinc anode had no effect. The above proportions of pyrolusite and graphite were shown to be the most advantageous. The first factor of quality is notably increased by using ammonium bromide as electrolyte and still more with the iodide. Of a number of other inorganic salts tested, only magnesium chloride gave a factor approaching that for ammonium chloride. With electrolytes containing ammonium and magnesium chlorides the factor decreased notably with increasing proportion of the latter, but the zinc was more regularly corroded and less salts were deposited. Of a large number of salts of organic acids of various types which were tried as electrolytes only ammonium salts were of any value, and of these only the tartrate gave a factor of quality exceeding that of ammonium chloride. With ammonium tartrate the utilisation of the pyrolusite is greatly improved, and the factor of quality is about three times that of a similar cell with ammonium chloride. Moreover, the zinc is more regularly corroded and no insoluble salts are formed. The probable reactions occurring in this cell are discussed.

H. J. T. ELLINGHAM.

## Engineering development of photovoltaic cells.

I. Some operating characteristics. C. G. FINK and D. K. ALPERN (Trans. Amer. Electrochem. Soc., 1930, 58, 133—153).—An investigation has been made of the operating characteristics of cells of the type  $\text{Cu, Cu}_2\text{O} | 1\% \text{ Pb}(\text{NO}_3)_2 \text{ solution} | \text{Pb}$ , in which the copper-copper oxide electrode (the cathode) is photosensitive and the lead electrode photochemically inert. Such cells have a sensitivity of about 150 microamp./lumen. The terminal voltage of the cell with a given external resistance rises rapidly at first with increasing intensity of illumination, but tends to approach a maximum value at higher intensities. From these values of the terminal voltage,  $E$ , and the corresponding data for the current delivered by the cell, values of the apparent internal resistance,  $r$ , are calculated, and it is found that  $E/r$  is a linear function of the intensity of illumination over the range studied. The sensitiveness of the cell is a maximum for light of a wave-length about 4600 Å.

H. J. T. ELLINGHAM.

**Computation of throwing efficiency [of electrolysis baths].** L. C. PAN (Trans. Amer. Electrochem. Soc., 1930, 58, 255—263).—The throwing power as defined by Haring and Blum (B., 1923, 556 A) is a function of the ratio of the distances of the two cathode surfaces from the anode. A modification of this definition, suggested by Heatley, gives a quantity (now distinguished as the "throwing efficiency") which shows how near the actual throwing power for a given distance ratio approaches the ideal throwing power for this ratio. Measurements made on a cyanide cadmium bath, a nickel bath, and an acid zinc bath show that although the throwing efficiency varies much less than the throwing power with change in the distance ratio, it is by no means independent of the latter.

H. J. T. ELLINGHAM.

**Asphaltic bitumen.** JACKSON.—See II. **High-test cast iron.** MOLDENKE. **Corrosion tests of ferrous metals.** PITSCHNER. **Plating on radiator shells.** WATTS. **Iron-nickel alloys.** STOUT and CAROL. **Nickel-cobalt alloys.** FINK and LAH. **Nickel.** PHILLIPS. **Determination of nickel in plating baths.** WÖGRINZ. **Chromium.** CUTHBERTSON; WIRSHING. **Lead-thallium alloys.** FINK and CONARD. **Testing of electroplated deposits.** MOUGEY.—See X. **Cadmium yellow.** FINK and GROSVENOR.—See XIII. **Electrofiltration.** PURI.—See XVI.

See also A., 1931, 9, **Photo-electric cells** (SCHOTTKY; LANGE; TEICHMANN; IVES). 28, **Ferromagnetic materials in weak alternating fields** (GOLDSCHMIDT). 35, **Colloid science and electrotechnics** (OSTWALD). 43, **Thermionic valve potentiometer** (MÜLLER). 44, **Gaseous combustion in electric discharges** (FINCH and PATRICK). 47, **Cathodic separation forms of tin** (FOERSTER and DECKERT). 57, **Electrolytic separation of antimony and copper** (HÖLEMANN). 78, **Behaviour of benzenoid hydrocarbons in the Tesla discharge** (AUSTIN and BLACK).

## PATENTS.

[Carbide resistors of] electric furnaces. CARBORUNDUM Co., Assees. of R. C. BENNER, G. J. EASTER,

and C. E. HAWKE (B.P. 338,131, 10.7.29. U.S., 7.12.28).—The life of carbide resistors is increased by operating them in a non-oxidising atmosphere, *e.g.*, by surrounding the electrodes with granular carbon.

J. S. G. THOMAS.

**Induction furnace.** P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,768,881, 1.7.30. Appl., 14.8.29).—A refractory crucible of the surrounded pool type has a number of intermediate axial and radial refractory walls co-operating with it to form similar and separate sections distributed peripherally within the crucible, whereby the effective length of the secondary circuit is increased. Means are provided in the walls for emptying the crucible.

J. S. G. THOMAS.

**[High-frequency metal-melting] induction furnace.** G. A. CHUTTER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,752,887, 1.4.30. Appl., 13.4.28).—Means responsive to variations in the power factor of the furnace are provided for varying the coupling between the primary winding of the furnace and the material to be melted, *e.g.*, by causing movement of the winding relative to the container.

J. S. G. THOMAS.

**Electrode and contact element [for making and breaking electric circuits].** A. J. MANDELL (U.S.P. 1,769,229, 1.7.30. Appl., 19.5.25).—A contact element made of a nickel or cobalt alloy containing 0.5–10% Si and, if desired, one or more of the elements aluminium, chromium, tungsten, zirconium, titanium, manganese, or vanadium is claimed. Thus a nickel alloy containing 0.5–10% Si and 0–5% Mn is suitable.

J. S. G. THOMAS.

**[Elimination of hum in] electrical transformers.** H. J. CAMERON, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,769,906, 1.7.30. Appl., 27.12.29).—The laminated core and coils of the transformer are impregnated with a 7:3 rosin-asphalt mixture.

J. S. G. THOMAS.

**(A) Detector element. (B–D) Current-rectifying devices.** W. O. SNELLING (U.S.P. 1,766,461–2, 1,766,464, and 1,766,467, 24.6.30. Appl., [A] 7.3.23, [B] 14.2.27, [C] 20.9.28, [D] 31.7.29).—For rectifying purposes the use of the following is claimed: (A) An oxide of an element (at. wt. > 198) of the odd series of the fourth long period of the periodic system, *e.g.*, litharge, at least partly reduced by sulphur. (B) A metal, *e.g.*, copper, heated at a red heat in an atmosphere containing sulphur vapour and an inert fluid diluent, *e.g.*, nitrogen. (C) A substantially dry lead salt, *e.g.*, lead carbonate, reduced by an element of the sulphur group, *e.g.*, sulphur, selenium, or tellurium. (D) A substantially dry lead salt, *e.g.*, basic lead carbonate, reduced by a binary sulphur compound, *e.g.*, hydrogen sulphide.

J. S. G. THOMAS.

**Production of rectifying units.** C. E. OGDEN, Assr. to KODOL RADIO CORP. (U.S.P. 1,769,852, 1.7.30. Appl., 27.6.27).—A copper plate heated at about 1040° in an atmosphere containing oxygen and chlorine is rapidly cooled to 760° and then quenched, whereby the oxide coating is partly reduced to metal.

J. S. G. THOMAS.

**Electrolytic rectifier.** F. EICHENBERGER, Assr. to GLOBAL CORP. (U.S.P. 1,765,573, 24.6.30. Appl., 28.5.23.

Switz., 10.6.22).—An inactive anode and a cathode composed essentially of silicon, *e.g.*, a silicon carbide core coated with silicon, are arranged in an aqueous electrolyte.

J. S. G. THOMAS.

**[Electrolyte for] electrolytic rectifier.** E. F. LUNDEEN, Assr. to WILLARD STORAGE BATTERY CO. (U.S.P. 1,769,228, 1.7.30. Appl., 14.4.27).—An electrolytic solution containing an organic acid, a phosphate, and a small proportion of a chromate is claimed. Thus a solution containing citric acid, ammonium phosphate, potassium phosphate, potassium chromate, and, if desired, sodium benzoate is suitable.

J. S. G. THOMAS.

**Electrolytic process and apparatus [for electro-deposition of metals].** H. C. HARRISON (U.S.P. 1,768,358, 24.6.30. Appl., 21.5.25).—Anode and cathode are completely separated in the cell by enclosing an elongated, flexible cathode of ribbon form in a tunnel-like compartment of porous, non-conducting material semi-permeable to the electrolyte and of low electrical resistance when saturated with electrolyte. The cross-section of the tunnel-like compartment conforms with, and is slightly larger than, that of the cathode. Electrolyte is forced through the cell in two distinct streams, the greater part being forced under high pressure and at high speed through the cathode compartment on both sides of the cathode, while the smaller part is forced under lower pressure through the anode compartment. Extremely high cathode current densities, *e.g.*, 2000 amp./sq. ft., may be employed.

J. S. G. THOMAS.

**Making articles by electrolytic deposition of galvanoplastic metal.** G. ROSENQVIST (B.P. 339,428, 24.10.29).—Apparatus comprising a lining of flexible material arranged between a mould and its supporting frame, and mould sections arranged in contact with the lining material and forming the walls of an electrolytic bath, is claimed.

J. S. G. THOMAS.

**Manufacture of loaded copper electrical conductors.** ELECTRICAL RES. PRODUCTS, INC., Asses. of V. E. LEGG (B.P. 338,169, 14.8.29. U.S., 24.11.28).—A copper conductor is heated at about 1000° in the presence of a reducing agent, *e.g.*, charcoal, then loaded with magnetic material, and the whole heat-treated in the absence of inert gases or other means of removing reducing gases (or materials producing such gases) which are present in the furnace or in the loaded conductor.

J. S. G. THOMAS.

**Condenser and material for dielectric or insulation purposes.** S. BOYER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,769,874, 1.7.30. Appl., 4.12.25).—Dielectric material, *e.g.*, paper, is impregnated with an aromatic compound, *e.g.*, nitrobenzene, having a sp. inductive capacity of 10 and over, and a resistivity of at least 10<sup>9</sup> ohm-cm.

J. S. G. THOMAS.

**Positive-ion emitter.** C. H. KUNSMAN, Assr. to A. B. LAMB (U.S.P. 1,767,218, 24.6.30. Appl., 29.9.25).—A mixture containing a positive-ion emitting substance and a promoter of the emission is claimed. Thus, *e.g.*, a mixture composed of partly or wholly reduced iron oxides, potassium oxide (about 1%), and aluminium oxide (about 1%) when heated emits potassium ions.

J. S. G. THOMAS.

**Electron-emitting cathodes for electric-discharge devices.** GRAMOPHONE CO., LTD., and G. B. BAKER (B.P. 339,451, 14.11.29).—A refractory core, *e.g.*, of tungsten, or oxide-coated filament is coated with a salt, *e.g.*, barium or caesium tartrate, which on heating in the absence of oxygen leaves a residue containing the oxide of an electron-emitting metal and free carbon.

J. S. G. THOMAS.

**[Localising alkali metal in] electric-discharge devices.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF E. E. CHARLTON (B.P. 319,686, 28.8.29. U.S., 26.9.28).—The anode or a carrier other than an electrode is coated with material, *e.g.*, a colloidal suspension of graphite in water, which absorbs alkali-metal vapour liberated by heating a mixture of caesium (or rubidium) chloride and calcium in the device.

J. S. G. THOMAS.

**Manufacture of [gas-filled] luminescence [discharge] tubes.** W. F. HENDRY, ASSR. to MANHATTAN ELECTRICAL SUPPLY CO., INC. (U.S.P. 1,769,025, 1.7.30. Appl., 13.9.27).—The gas filling, *e.g.*, neon, is purified by bubbling through sodium-potassium alloy at room temperature before entering the tube.

J. S. G. THOMAS.

**[Slotted] collecting electrodes for use in electrical precipitation of suspended particles from gaseous fluids.** LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 321,869, 27.12.28).—The electrodes are provided with a series of transverse strips suspended outside their centre of gravity and connected to a displacing mechanism which moves to and fro and thus causes the strips to strike against their supporting rod.

A. R. POWELL.

**Apparatus for electrical separation of suspended material from gases.** LODGE-COTTRELL, LTD. From INTERNAT. PRECIPITATION CO., INC. (B.P. 338,427, 14.1.30).—Curtains of rods are arranged in intersecting rows dividing the precipitator space into compartments, in each of which discharge electrodes are arranged. Means for passing gas through the compartments in a direction either parallel or transverse to the rods, and for bringing the gas into contact with the discharge and collecting electrodes, are provided. J. S. G. THOMAS.

**Electric purification of gas by means of rectified alternating current of high potential.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 338,944, 1.8.29. Ger., 17.4.29).—A resistance of suitable magnitude is arranged in parallel with the filter so that, between two charging impulses, the field intensity at the discharge wires falls below that required to produce the working discharge. A resistance, the value of which is independent of the applied voltage, *e.g.*, a liquid resistance, is also arranged in parallel with the filter.

J. S. G. THOMAS.

**Electrical precipitation [from gases].** H. A. WINTERMUTE and C. W. J. HEDBERG, ASSRS. to RESEARCH CORP. (U.S.P. 1,766,421—2, 24.6.30. Appl., [A] 19.4.26, [B] 28.11.27).—(A) A discharge electrode extends into the gas passageway formed by spaced laminar collecting electrodes, and baffles, *e.g.*, plates spaced from one end of the walls of the passage, are arranged to produce a substantially uniform transverse distribution of flow in the passage way. (B) Gas is treated first in a "dry"

section of the precipitation plant and then in a "wet" section comprising collecting electrodes flushed with liquid.

J. S. G. THOMAS.

**Apparatus for electrical precipitation [from gases].** W. SYKES, ASSR. to NEBLETT ENG. CO. (U.S.P. 1,767,265, 24.6.30. Appl., 29.11.26).—Flues each containing a pair of negatively-charged discharge electrodes are formed by partitions constituting the positive electrodes in a casing, and gas descends through some of these flues which are closed at their upper ends, and ascends through the remaining flues. The total cross-sectional area of those flues in which the gas ascends is greater than that of the remainder. J. S. G. THOMAS.

**Electrical precipitating apparatus [for gases].** A. ARRAS, ASSR. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,767,338, 24.6.30. Appl., 3.5.28. Ger., 27.5.27).—The hollow spaces or interior chambers of the collecting electrodes are maintained under a slightly reduced pressure sufficient to prevent the formation of gas cushions or regions of increased pressure in the openings of the collecting surfaces leading in to the hollow spaces.

J. S. G. THOMAS.

**Electric furnace.** A. IMBERY, ASSR. to GLOBAR CORP. (U.S.P. 1,784,670, 9.12.30. Appl., 8.10.28. U.K., 10.10.27).—See B.P. 303,222; B., 1929, 177.

**Production of magnetic cores.** K. EISENMANN and F. BERGMANN, ASSRS. to I. G. FARBEIND. A.-G. (U.S.P. 1,783,560—1, 2.12.30. Appl., 18.3.29. Ger., 30.3.28).—See B.P. 320,611 and 319,854; B., 1930, 21.

**[Introducing potassium, caesium, or rubidium into electric] discharge tubes.** J. H. DE BOER and P. CLAUSING, ASSRS. to N.V. PHILIPS' GLOEILAMPEN-FABR. (U.S.P. 1,767,437, 24.6.30. Appl., 18.10.26. Holl., 12.12.25).—See B.P. 264,258; B., 1927, 226.

**[Attaching and operating the] electrodes of electric furnaces and electrolytic vats.** P. GIROD, ASSEE. OF A. FRANCHINI (B.P. 338,371, 20.11.29. Fr., 27.8.29).

**Production of anodes for cells of electric dry batteries.** W. SCHMIDT (B.P. 339,881, 17.2.30. Ger., 21.3.29).

**Manufacture of [helical] filaments for electric lamps.** WESTINGHOUSE LAMP CO., ASSEES. OF S. WADSTEN and C. W. HUNT (B.P. 339,803, 20.12.29. U.S., 29.12.29).

**Electric discharge tubes with rare-gas fillings.** A. LEDERER (B.P. 314,864, 25.6.29. Austr., 3.7.28. Addn. to B.P. 306,831).

**Mixing systems (B.P. 338,123).** Dispersal of fog (B.P. 339,295).—See I. Measuring the calorific value of gases (U.S.P. 1,767,771). Bituminous preparations (B.P. 339,470). Deoxidiser for insulating oils (U.S.P. 1,736,464).—See II. Acetylene and hydrogen (B.P. 337,088).—See III. Coating of conductors (U.S.P. 1,764,625).—See V. Aluminium chloride (U.S.P. 1,752,936). Purifying rare gases (U.S.P. 1,753,298).—See VII. Coating of vitreous articles (U.S.P. 1,752,792).—See VIII. Cement kiln (B.P. 316,715).—See IX. Annealing steel (B.P. 338,511). Plating aluminium (B.P. 339,339). Aluminium (B.P. 338,668). Tin (U.S.P. 1,766,463). Tungsten filament (U.S.P. 1,764,644). Applying the grain of wood to

metal (B.P. 319,283).—See X. Fertilisers (B.P. 337,402).—See XVI. Treatment of fatty substances for ointments etc. (B.P. 316,264).—See XX.

## XII.—FATS; OILS; WAXES.

**Bleaching of linseed oil.** ANON. (Farben-Ztg., 1930, 36, 595—596).—A general account of the decolorising of linseed oil is given, reference being made to the various processes used, the importance of temperature and time of contact with the bleaching agent, and the tendency to re-darken. The last-named is attributed to excessive temperature and pressure at filtration, the presence of acid, and the use of poor-quality filter paper.

S. S. WOOLF.

**Detection of castor and arachis oils in "Abrasinol."** H. WOLFF and J. RABINOWICZ (Farben-Ztg., 1930, 36, 596—597. Cf. Stock, B., 1930, 1162).—The determination of solubility in alcohol and of acetyl value are recommended for determining castor oil in this connexion. Arachis oil can generally be determined by measurement of refractive index, but conclusive information is obtained by isolating the fatty acids and separating the arachis oil acids from elæostearic acid by fractional crystallisation or fractional precipitation of the magnesium salts.

S. S. WOOLF.

**Vitamins-A and -D in fish oils.** E. M. NELSON and J. R. MANNING (Ind. Eng. Chem., 1930, 22, 1361—1363).—Commercial grades of sardine (*i.e.*, pilchard), menhaden, Alaska herring, salmon, Maine herring, and tuna oils were tested by feeding experiments on rats. With respect to vitamin-A, salmon oil is as potent as low-grade and only one third as potent as high-grade cod-liver oil. Sardine, Alaska herring, and tuna oils are one tenth as potent as good medicinal cod-liver oil. With respect to vitamin-D, the relative potencies are: cod-liver oil (good medicinal) 100, tuna 125, sardine 100, menhaden 75, salmon 50, Alaska herring 30, Maine herring 15. At current prices, some of these commercial oils are cheaper sources of vitamins than is cod-liver oil.

W. J. BOYD.

**Soya-bean oil meal.** ROBISON.—See XVI.

See also A., Jan., 62, Unsaponifiable matter from oils of elasmobranch fish (DAVIES and others; also HEILBRON and WILKINSON, p. 80). 63, Synthetic mixed triglycerides (BHATTACHARYA and HILDITCH). 111, Pig-stomach fat (HEPBURN and TREXLER). 131, Oil in Para rubber seed (IWAMOTO).

## PATENTS.

**Cleansing and working of [butter or like] fats.** R. BENDLIN and O. URBASCH (B.P. 338,053, 2.1.30. Austr., 9.1.29).—The fat is divided into fine threads by extrusion from a vermicelli press and is further comminuted by jets of the cold liquid washing medium which removes impurities. The fat particles are separated by settling and centrifuging.

E. LEWKOWITSCH.

**Production of scouring compositions.** W. J. A. HUYZER (B.P. 337,355, 26.7.29. Cf. B.P. 335,005; B., 1930, 1097).—Mixtures of magnesite (about 50-mesh) and soap powder are claimed.

H. ROYAL-DAWSON.

**Apparatus for degreasing materials [by volatile solvents].** IMPERIAL CHEM. INDUSTRIES, LTD., and

F. E. ROGERS (B.P. 337,770, 1.8.29).—Mechanical inter-connecting devices, controlled by a common handle, are devised, so that the heating means (*e.g.*, in the plant described in B.P. 278,891; B., 1927, 946) can be brought into operation only when the cooling means are in action and the lid of the degreasing vessel is locked down.

E. LEWKOWITSCH.

**Apparatus for recovering fats from emulsions containing solid particles.** J. A. L. ROZIÈRES, ASSR. to SOC. FRANÇ. DE CENTRIFUGATION (U.S.P. 1,782,974, 25.11.30. Appl., 16.7.28. Fr., 11.10.27).—See B.P. 289,350; B., 1928, 456.

**Apparatus for separating fat or other material from gases.** G. W. F. CATLIN (U.S.P. 1,784,418, 9.12.30. Appl., 8.1.29. U.K., 3.2.28).—See B.P. 311,204; B., 1929, 565.

**Pure glycerin** (B.P. 336,608). **Polyhalogenated fatty acids** (B.P. 336,623).—See III. Treatment of fatty substances for ointments etc. (B.P. 316,264).—See XX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**"Chalking" [of paints].** III. R. KEMPF (Farben-Ztg., 1930, 36, 553—555; cf. B., 1930, 1119).—The question of whether chalking is to be regarded as a favourable or unfavourable manifestation of paint films is discussed, with full references to the literature. Excessive chalking early in the life of a film is undesirable, but slight and regular chalking at advanced film-age may be advantageous. Where retention of gloss and resistance to rubbing are essential chalking is a drawback, but this is not so in the case of white paints required to maintain their colour, nor in that of interior paints not subjected to rubbing. The relation between chalking and durability is not yet established.

S. S. WOOLF.

**Micrography of coloured pigments.** II. **Red oxides of iron.** ANON. (Farben-Ztg., 1930, 36, 597—599).—A summary of the methods adopted and results obtained by H. Wagner and H. Pfanner in their work on the microscopical, chemical, and physical properties of a range of 30 natural and artificial iron oxides. (Cf. B., 1930, 1119; 1931, 33.)

S. S. WOOLF.

**Electrolytic production of cadmium yellow.** C. G. FINK and W. M. GROSVENOR, JUN. (Trans. Amer. Electrochem. Soc., 1930, 58, 271—277).—An electrolytic method of preparing cadmium sulphide of suitable colour for use as a pigment has been investigated. The cathode compartment of a diaphragm cell contains a steel shaft fitted with iron discs which acts as cathode and rotates in dilute sulphuric acid containing finely-ground ferrous sulphide in suspension. Rods of electrolytic cadmium are arranged around the outside of the cathode compartment and dip into a dilute acid solution which forms the anolyte. On passing the current cadmium sulphide is precipitated just outside the diaphragm with a current efficiency approaching 100%, and a slime of metallic iron is deposited on the cathode.

H. J. T. ELLINGHAM.

**"Run" amber.** I. C. PLONAIT (Farben-Ztg., 1930, 36, 555—556).—The process of "running" amber is described, five chief grades differing in colour being obtained. The constants of these are tabulated: acid

values 12—24 (cf. run copal, 52—90), saponif. values 19—54, ash content below 0.1%. S. S. WOOLF.

**Asphaltic bitumen.** JACKSON.—See II.

See also A., Jan., 78, **Behaviour of benzenoid hydrocarbons in the Tesla discharge** (AUSTIN and BLACK). 81, **Acenaphthene resins** (MORGAN and HARRISON). 89, **Constituents of kawa root** (BORSCHKE and others). 94, **Resin acid of the isosylvic acid group** (ASCHAN). [Resin from] **autoxidation of piperitone in alkaline solution** (TREIBS).

#### PATENTS.

[Metallic] **paints.** BRIT. THOMSON-HOUSTON CO., LTD., Assces. of L. V. ADAMS (B.P. 337,682, 11.1.30. U.S., 11.1.29).—Suspensions of finely-divided metal, *e.g.*, aluminium or zinc, in volatile solvent solutions of resinous condensation products of polybasic acids, polyhydric alcohols, drying oils, fatty acids derived therefrom, and rosin are claimed. S. S. WOOLF.

[Rust-preventive] **paints.** CONSORT. F. ELEKTRO-CHEM. IND. G.M.B.H. (B.P. 314,499, 26.6.29. Ger., 28.6.28).—The use of polyvinyl acetates in alcohol or benzene solution as vehicles for such paints is claimed. S. S. WOOLF.

**Binding compounds for painting purposes.** A. ROGLER (B.P. 337,523, 5.9.29).—Drying or semi-drying oils are heated at 100—150° with 10—30 wt.-% of zinc oxide, titanium oxide, or other suitable metal oxide until a semi-solid mass is obtained. This is dissolved in solvents, and pigments, resins, etc. are incorporated if desired. Rapid hardening is claimed. S. S. WOOLF.

**Manufacture of non-caking pigmented coating compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,061, 6.5.29. U.S., 18.5.28).—A protective agent adapted to inhibit caking of pigment, *e.g.*, rubber in colloidal solution in petrol, is deposited on pigment particles before, during, or after grinding the pigment in the vehicle. This treatment is particularly suitable for use with cellulose ester compositions containing damar gum. S. S. WOOLF.

**Compositions and processes for removing coating material from coated surfaces.** H. WADE. From J. H. GRAVELL (B.P. 337,461, 2.8.29).—Painted surfaces are treated with a hot aqueous solution of an alkali hydroxide and a phenol, *e.g.*, cresylic acid (preferably present to the extent of 25% of the weight of alkali hydroxide), and subsequently washed with a high-pressure stream of water. Residual alkalinity is removed by treatment with dilute acid, *e.g.*, phosphoric acid. S. S. WOOLF.

**Paint and varnish remover.** N.C.W. PAINT & VARNISH REMOVER CO., LTD., and T. K. BONNAR, JUN. (B.P. 337,944, 19.10.29).—Quicklime is covered with soft soap and water, the mixture stirred, sodium carbonate, ammonium carbonate, and sodium thiosulphate are successively added, and the product is stirred and allowed to cool and thicken. H. ROYAL-DAWSON.

**Manufacture of [pencil] leads, crayons, and chalks.** A. and P. PISCHEL (B.P. 337,633, 27.11.29).—A sulphonated fatty oil, *e.g.*, Turkey-red oil, is added to the usual mass of clay (or loam) and graphite, before or during kneading and pressing. H. ROYAL-DAWSON.

**Making pyroxylin solutions of low viscosity.** H. HIBBERT and J. L. PARSONS, Assrs. to HAMMERMILL PAPER CO. (U.S.P. 1,768,253, 24.6.30. Appl., 8.6.26).—Before nitration, cellulose is subjected to oxidation by suspending it in a slightly alkaline or slightly acid solution of potassium permanganate or a peroxide or chromate at 22—26°, and then washed with sodium bisulphite solution and water. W. J. WRIGHT.

**Manufacture of resinous condensation products.** VARNOL CHEM. CORP. (B.P. 315,870, 11.7.29. U.S., 21.7.28).—A phenol-aldehyde or -ketone condensation product (made preferably in presence of solvent such as turpentine, drying or non-drying oils, natural or synthetic resins, ester gums) is dehydrated at 120° and heated with a glycerol abietate (or other resinous glyceride containing free alcoholic groups) until all volatile matter is driven off at about 300°. The phenolic groups are thus etherified and the products are neutral resins of high m.p. suitable for varnishes. C. HOLLINS.

**Manufacture of artificial masses.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,806, 6.8.29).—Polymerised vinyl alcohols are treated with aliphatic aldehydes, *e.g.*, formaldehyde or trioxymethylene, or with substances furnishing them, *e.g.*, acetals, in the presence of acid catalysts, such as mineral acids, aluminium chloride, sodium bisulphate; heat and/or pressure may be applied, if desired. A subsequent vulcanisation improves the product, especially if insaturated aldehydes, *e.g.*, acraldehyde, have been used. S. S. WOOLF.

**Production of urea-aldehyde resins.** W. W. TRIGGS. From TOLEDO SCALE MANUFACT. CO. (B.P. 337,357, 27.4.29).—Urea is heated with excess of formaldehyde in a slightly acid solution, and the residue of formaldehyde is combined in stages by the addition of urea in suitably diminishing quantities, the  $p_H$  value of the solution during these initial condensations being controlled by additions of a strong organic base which does not react with formaldehyde, *e.g.*, triethanolamine. The solution is then concentrated, polybasic acid-polyhydric alcohol condensation products are added, and the mixture is further concentrated, to give moulding powders etc. S. S. WOOLF.

**Incorporation of "alkyd" resins into drying oils.** BRIT. THOMPSON-HOUSTON CO., LTD., Asses. of R. H. KIENLE (B.P. 316,319, 27.7.29. U.S., 27.7.28).—Drying oil fatty acids are heated with a polyhydric alcohol, *e.g.*, glycerol, in chemical excess, until a homogeneous product is obtained, a drying oil is added, the solution is stirred until clear, and the product is resinified by addition of a polybasic acid, *e.g.*, phthalic anhydride. Alternatively, the drying oil is introduced into a mixture of the remaining constituents in a stage of incomplete resinification, and resinification is then completed. S. S. WOOLF.

**Synthetic resins and compositions containing them.** BRIT. CELANESE, LTD. (B.P. 315,807—8, 18.7.29. U.S., 18.7.28).—(A) Benzenesulphonamide or (B) xylene-sulphonamide is condensed with an aldehyde (formaldehyde), preferably in presence of catalyst, to give a resin, which is incorporated with cellulose derivatives (acetate) for lacquers, plastics, or adhesives for non-splintering glass. C. HOLLINS.



**Manufacture and use of synthetic resins.** BRIT. CELANESE, LTD. (B.P. 338,002 and Addn. B.P. 338,024, [A] 18.11.29, [B] 5.12.29. U.S., [A] 17.11.28, [B] 5.12.28).—Toluenesulphonamide-aldehyde (e.g., formaldehyde) condensation products, produced if desired by reacting in the presence of 5–10% of urea and removing excess of reactants, are condensed (A) with a tolunesulphonamide and the resin so obtained is heated at 160–200° for 4–8 hrs., to give a benzene-insoluble resin suitable for use in lacquers, films, adhesives, etc., or (B) with an aldehyde, e.g., formaldehyde, and subsequently heated, to yield benzene-soluble products. S. S. WOOLF.

**Preparation of talking-machine records.** A. O. THOMAE (B.P. 337,437, 1.7.29).—Gelatinised acetylcellulose with camphor or camphor substitutes is dried at 40–80° for 24 hrs. per 0.1 mm. of thickness, the material is rendered fluid by heat or by dissolution, e.g., in acetone, and sprayed or brushed on a matrix bearing a sound record, and the coated matrix is heated to 60–90° under a pressure of about 200 atm. S. S. WOOLF.

**Manufacture of sound records.** DURUM PRODUCTS CORP., Assees. of H. T. BEANS (B.P. 337,796, 5.7.29. U.S., 2.4.29).—Resorcinol, preferably ground, is dissolved in heated formaldehyde and stirred at 70–75° until suitable viscosity is attained, volatile solvent (alcohol), abrasive (rouge), and plasticiser (glycerin) are introduced, and the product is bonded to a resilient support, e.g., paper-stock sheet, and dried. Tone-grooves are then impressed on the surface by use of a die heated to 160°, polymerisation of the surface material occurring in about 1 min. [Stat. ref.] S. S. WOOLF.

**Resinous condensation product.** A. H. V. DURR, Assr. to COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ETABL. KUHLMANN (Re-issue 17,878, 25.11.30, of U.S.P. 1,739,447, 10.12.29).—See B., 1930, 625.

**Adhesive composition.** W. O. HERRMANN and W. HAEHNEL, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,784,008, 9.12.30. Appl., 9.5.27. Ger., 11.5.26).—See B.P. 271,090; B., 1928, 793.

**Polyhalogenated fatty acids** (B.P. 336,623). **Naphthazarin derivatives** (B.P. 316,950). **Azo pigments** (B.P. 336,938).—See IV. **Solvent extraction of wood** (U.S.P. 1,762,785).—See V. **Coating materials from asphalt etc.** (B.P. 337,521).—See IX. **Preparing metal for painting** (U.S.P. 1,765,331).—See X.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Use of asphalts and bitumens on [rubber] estates.** H. SUTCLIFFE (Rubber Res. Inst. Malaya, 1930, 2, 113–124).—Fluid mixtures of asphaltum with kerosene or various proprietary fungicides are suitable for wound treatment of rubber trees. The penetration of the mixtures is so low, even with young tissue, that it permits the use of ingredients which by themselves would be quite unserviceable on account of their excessive penetration. D. F. TWISS.

**Bacteriology of *Hevea* latex.** A. S. CORBET (Rubber Res. Inst. Malaya, 1930, 2, 139–155).—Several new species of micro-organism from *Hevea* latex are described. One of these, viz., *Micrococcus epimetheus*, affects both

acidification and flocculation of latex; it may be a frequent cause of bubble-formation in sheet rubber, but its action in this direction can be inhibited by preventing the  $p_H$  of the coagulating tank from exceeding 4.4. D. F. TWISS.

**Coagulation of *Hevea* latex.** R. G. FULLERTON (Rubber Res. Inst. Malaya, 1930, 2, 156–181).—The behaviour of latex of various concentrations, of pasteurised latex, of latex preserved with ammonia, sodium hydroxide, or formalin, and of the “cream” and “skim” obtained from latex by centrifugal action, with respect to the occurrence of two successive coagulation zones on progressive decrease of  $p_H$ , has been examined with the aid of the quinhydrone electrode. D. F. TWISS.

**The lipin of *Hevea* latex.** E. RHODES and R. O. BISHOP (Rubber Res. Inst. Malaya, 1930, 2, 125–135).—The serum from the alcoholic coagulation of latex, when concentrated and extracted with ether, yields a product resembling the plant-lipin complexes; its chemical and physico-chemical properties are described. D. F. TWISS.

**Effect on vulcanisation of the lipin of *Hevea* latex.** B. J. EATON, E. RHODES, and R. O. BISHOP (Rubber Res. Inst. Malaya, 1930, 2, 136–138; cf. preceding abstract).—The addition of *Hevea* lipin to alcohol-coagulated rubber, “slab” rubber, or pale crêpe rubber causes an increase in the rate of vulcanisation, which, however, is very slight in the last case. The effect of lecithin on vulcanisation is considerably greater than that of the lipin, but both cause a peculiar crumbling of the rubber during the earlier stages of the mixing operation. D. F. TWISS.

**Hot-air vulcanisation. Development of its technical practice.** P. BREDEMANN (Gummi-Ztg., 1930, 45, 375–376, 413–416, 458–462, 502–503).—A description is given of various types of plant and equipment for the vulcanisation of different articles, such as footwear and rubbered textile material, in heated air. D. F. TWISS.

**Mechanical properties of rubber in compression at low temperature.** W. D. DOUGLAS (India-rubber J., 1930, 80, 899–901).—Investigation of the effect of temperatures down to –50° on the stress-strain characteristics of rubber in compression shows that, with comparatively high initial rates of loading to equal maximum loads, rubber at low temperatures absorbs a greater amount of energy than at normal temperatures. D. F. TWISS.

**Standardisation of testing of rubber with ozone.** E. P. W. KEARSLEY (Kautschuk, 1930, 6, 244–249).—An apparatus and method are described for securing standard conditions in the exposure of rubber samples 3 in. × 0.5 in. × 0.1 in. for 30 min. to an atmosphere containing 1 pt. of ozone in 50,000 pts. The test-pieces were maintained at the intended extension for 3 days before exposure to the special atmosphere, and were subsequently examined for surface-cracking at a standard stretch of 100%. With increase in the degree of extension of the exposed sample the cracks were smaller, but their number rapidly increased with extension up to 10%, at which the surface was covered with cracks (“maximal crack formation”). If the period of stretch

before exposure was increased, the point of maximal crack formation moved towards a higher degree of stretch. The nature and number of the cracks were not seriously influenced by the concentration of the ozone for any one degree of extension, although it affected the depth of the cracks. Over-vulcanisation only slightly affected the formation of cracks; under-vulcanised rubber showed less tendency to cracking, probably on account of the greater plasticity. Alteration of the degree of humidity did not affect the results, but increase of temperature from 15° to 70° approximately halved the time, this result being attributable to the greater tendency of the stretched rubber to retract when heated. With increasing proportions of zinc oxide up to 30 vols. on a mixture of rubber (100 pts.), sulphur (3 pts.), zinc oxide (5 pts.), and mercaptobenzthiazole (0.5 pt.), the extension necessary for maximal crack formation was increased up to 30%. From comparative tests on the effect of zinc oxide, natural whiting, and carbon black on the same mixing, it is shown that decreasing particle size and consequent increased permanent set lead to increase in the degree of extension at which maximal cracking results.

D. F. TWISS.

#### Importance of the theory of the main-valency chain for knowledge of the structure of rubber.

E. LINDMAYER (Kautschuk, 1930, 6, 249–252).—The characteristic features of rubber are attributed to its molecular structure comprising a straight or needle-shaped main-valency chain with free terminal valencies. This view is supported by analogy with other natural products such as starch, and it explains many of the phenomena observed with natural rubber, such as its anisotropy and the effects of vulcanisation. The inferiority of synthetic rubbers is ascribed to polymerisation yielding *cis*-configurations in the molecule instead of the desired elongated chain of valencies. D. F. TWISS.

See also A., Jan., 38, **Structure viscosity of caoutchouc solutions** (DOGADKIN and PEWSNER). 93, **Action of nitric acid on caoutchouc** (GORGAS). **Isoprene and caoutchouc** (STAUDINGER and others). 131, **Oil and lipase-like enzyme in Para rubber seed** (IWAMOTO).

#### PATENTS.

##### Purifying and concentrating [rubber] latex.

W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,754,535, 15.4.30. Appl., 28.12.27).—Latex is first submitted to centrifugal force (cf. U.S.P. 1,630,411; B., 1927, 610) sufficient, however, to remove only coagulated particles exceeding 5  $\mu$  in diam. The separated coarse cream is washed with water and the washings are added to the refined latex. After introducing an agent, *e.g.*, carrageen, capable of accelerating creaming, the refined latex is again centrifuged, but with such intensity as to yield a concentrated latex and a rubber-free serum. The concentrated latex while still under centrifugal force is washed with a solution of a protective colloid such as haemoglobin so as to displace all the natural serum and to nullify the effect of any residual separation-accelerating agent. The proportion of carrageen for maximum acceleration is about 0.5%, and the amount permissible in latex capable of stable re-dispersion is about 0.05%. It is desirable initially

to clarify the carrageen solution by centrifuging, and convenient to introduce it when washing the "coarse cream" in the first stage.

D. F. TWISS.

**Dispersed vulcanised rubber.** W. C. GEER and H. L. TRUMBULL, Assrs. to B. F. GOODRICH Co. (U.S.P. 1,754,886, 15.4.30. Appl., 13.6.25. Renewed 15.9.28).—Vulcanised rubber plasticised with the aid of a rubber softener, such as 30% of kerosene, is intimately mixed on a tight mill with a colloidal emulsifying paste, *e.g.*, an aqueous paste of casein and sodium carbonate. The resulting pasty emulsion is diluted with water and then treated in a colloid mill until the particle size is reduced to approximately that of natural rubber latex.

D. F. TWISS.

**Preparation of dispersions containing rubber and manufacture of waterproof fibrous materials therewith.** N.V. BATAAFSCHE PETROLEUM MAATS., Assees. of L. KIRSCHBRAUN (B.P. 315,839, 17.7.29. U.S., 19.7.28).—A hot plastic mixture of rubber and bituminous material, such as asphaltum or coal-tar pitch, is mixed in a kneading or masticating machine at 70–93° with a relatively stiff paste of colloidal clay. The rubber mixture gradually becomes dispersed in the aqueous paste. Clay of the bentonite type will disperse in this way 5–20 times its weight of rubber-bitumen mixture. For the manufacture of waterproof fibrous materials, a diluted mixture of the dispersion with fibres is felted on a papermaking machine.

D. F. TWISS.

**Manufacture of coloured articles from dispersions of rubber and the like.** DUNLOP RUBBER CO., LTD., D. F. TWISS, E. A. MURPHY, and R. G. JAMES (B.P. 338,303, 11.10.29).—Articles formed from aqueous dispersions of rubber, *e.g.*, by dipping, are coloured by incorporating in the original dispersions, or applying to the surface while still wet, substances capable of reacting with another chemical agent, applied later, with formation of the desired pigment. *E.g.*, a wet, shaped deposit from latex containing  $\beta$ -naphthol may be immersed in a diazo solution; a wet freshly-formed deposit may be immersed wholly or partly in a solution of indigo-white and then exposed to air; shaped latex deposits coagulated with the aid of an aluminium salt or an acid, respectively, may be immersed in a hot solution of alizarin or in an alkaline sulphide solution of a sulphur dye.

D. F. TWISS.

**Manufacture of caoutchouc-like masses.** J. BAER (B.P. 314,524, 13.6.29. Switz., 29.6.28).—A solution of an alkali or alkaline-earth sulphide or polysulphide is caused to react with unsaturated halogen derivatives of unsaturated hydrocarbons, *e.g.*, isoprene.

H. ROYAL-DAWSON.

**Preservation of [rubber] latex.** K. SCHUSTER and H. HOPFF, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,783,057, 25.11.30. Appl., 6.4.28. Ger., 20.4.27).—See B.P. 289,022; B., 1928, 533.

**Manufacture of rubber goods from rubber dispersions.** P. KLEIN and A. SZEGVARI, Assrs. to AMER. ANODE, INC. (U.S.P. 1,783,435, 2.12.30. Appl., 13.4.27. Hung. and Ger., 14.4.26).—See B.P. 269,505; B., 1927, 853.

**Use of mono- and di-carbalkoxydiarylthioureas**

as accelerators in the vulcanisation of rubber. C. J. T. CRONSHAW and W. J. S. NAUNTON, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,782,842, 25.11.30. Appl., 27.10.26. U.K., 1.3.26).—See B.P. 264,682; B., 1927, 230.

**Adhesive [from rubber emulsion].** G. S. MATHEY, ASSR. to JOHNSON & JOHNSON (U.S.P. 1,784,740, 9.12.30. Appl., 3.6.27).—See B.P. 286,527; B., 1928, 378.

**Polymerised diolefines** (B.P. 337,019).—See III. **Materials for road construction etc.** (B.P. 337,521).—See IX. **Non-caking pigments** (B.P. 312,061).—See XIII.

## XV.—LEATHER; GLUE.

**Effect on gelatin and dehaired skin of the salts of the lyotropic series and the counteracting influence of tanning materials.** G. GRASSER, H. OHOKI, and V. HIRAKAWA (J. Fac. Agric. Hokkaido, 1930, 23, 165—176).—In low concentration none of the salts had a measurable effect on the m.p. or shrinkage temperature, but a liquefying concentration exists for each salt. The minimum liquefying concentration for anions followed approximately the order of the Hofmeister series, decreasing from phosphate to thiocyanate; a similar, but not identical, series was obtained by determining the minimum concentration of salt having an appreciable effect on the shrinkage temperature of skin. Lithium, calcium, barium, and strontium lowered the shrinkage temperature. The m.p. of gelatin tanned with formaldehyde was not lowered by salts; the m.p. of that tanned with chromium was lowered only by salts having a stripping action. Solutions of potassium thiocyanate, bromide, or iodide, or of lithium chloride liquefied vegetable-tanned gelatin; these salts and sodium nitrate liquefied gelatin tanned with benzoquinone, methylal, acetaldehyde, and paraldehyde. For gelatin tanned with bromine, the m.p. increased with increasing concentration of lactate, acetate, and fluoride; it was unaffected or decreased with increasing concentration of other salts. Similar, but specifically different, effects were exhibited by the same salts on iodine-tanned gelatin or iodine- or bromine-tanned skin.

### CHEMICAL ABSTRACTS.

(1) **Test for sulphite-cellulose extract.** (2) **Application of ultra-violet rays to tannery chemistry.** G. GRASSER (J. Fac. Agric. Hokkaido, 1930, 23, 151—156).—(1) The extract (10 c.c. diluted to 50 c.c.) is rendered alkaline with sodium hydroxide, boiled for 10 min., with stirring, in a dish, acidified with sulphuric acid (20%), again boiled, cooled, and filtered. The filtrate is extracted with ether (5 c.c.); the extract is treated with alcoholic phloroglucinol (1%, 1 c.c.) and concentrated hydrochloric acid (2 c.c.). In presence of sulphite-cellulose extract a red colour, due to the presence of vanillin, is developed. (2) Characteristic fluorescence is observed with variously tanned leathers. The condensation of phenolsulphonic acid or  $\beta$ -naphthol with formaldehyde is accelerated by ultra-violet light.

### CHEMICAL ABSTRACTS.

**Paper adhesives.** SHAW and others.—See V.

See also A., Jan., 96, **Tannins from kola nuts** (FREUDENBERG and OEHLE). 131, **Carboxylic tannins** (REICHEL). **Presence of tannins in flowers** (IONESCO).

## PATENTS.

**Manufacture of leather.** L. MELLERSH-JACKSON. From LEATHER MAKERS' PROCESS Co. (B.P. 339,138, 23.12.29).—The wet skins are struck out, placed on a drying board, and the marginal portions of the skin affixed to the latter by means of adhesive fabric strips. D. WOODROFFE.

**Treatment of fur.** C. D. PARKS (U.S.P. 1,766,452, 24.6.30. Appl., 8.5.25).—Fur-bearing skins are immersed in an almost boiling solution of a suitable acid, e.g., sulphuric, which is agitated and cooled by a current of air, and then the loosened fur fibres are separated from the decomposed skin. D. WOODROFFE.

**Lecithin preparations** (B.P. 317,730).—See III.

## XVI.—AGRICULTURE.

**Laws of soil colloidal behaviour. III. Isoelectric precipitates.** S. MATTSON (Soil Sci., 1930, 30, 459—495: cf. B., 1929, 904; 1930, 72).—The preparation of isoelectric precipitates of iron and aluminium "hydroxides" from the respective sulphates and chlorides is described. Hydroxides obtained from the sulphates are isoelectric at a lower  $p_H$  than those from the chlorides. The sulphate content of the former was higher than the equivalent chloride content of the latter. Addition of phosphate or silicate ions to the sesquioxide complex lowers the isoelectric  $p_H$  by displacing both the diffusible acid ions and the hydroxyl ions. The smaller the phosphate- or silicate-ion concentration of the solution used the greater is the relative proportion entering the complex. With solutions of high concentration the complex tends to approach, but does not attain, saturation (i.e.,  $P_2O_5/Al_2O_3 = 1$  and  $SiO_2/Al_2O_3 = 3$ ), provided cations forming insoluble phosphates and silicates are absent and colloidal silica is not precipitated. All ions in the system mutually displace one another according to their respective energies. At the isoelectric point a small number of diffusible anions are always in combination in the complex. The adsorptive surface layer of both crystalline and natural colloids changes constantly with external conditions. Hence a stoichiometric relationship between any two ions in the system is impossible. There is, however, a balance between the anions and cations in the system. The bearing of the above on observed variations in the nature of soil colloids of different climatic zones is discussed.

A. G. POLLARD.

**Electrofiltration: a method of removing exchangeable bases from soil colloids.** A. N. PURI (Soil Sci., 1930, 30, 413—419).—Two arrangements are described. Electrofiltration affords a satisfactory means of pretreating soils for mechanical analysis. Results so obtained agree with those for soils treated by the author's methods using sodium chloride + sodium hydroxide (B., 1929, 1026) and 0.5N-hydrochloric acid (B., 1928, 796).

A. G. POLLARD.

**Oxidation of pyrites and sulphur [in soils] as influenced by lime and magnesia—a 12-year lysimeter study.** W. H. MACINTIRE, W. M. SHAW, and J. B. YOUNG (Soil Sci., 1930, 30, 443—457. Cf. B., 1923, 1036 A, 1143 A; 1924, 27, 345).—Quicklime, magnesia, limestone, and dolomite increased the amount of sulphates leached from soil in lysimeter experiments,

but with dressings equivalent to 2000 lb. of lime per 2 million lb. of soil the outgo of sulphate was always less than that supplied in rainfall. Differences in the effects of lime and magnesia on the rate of leaching of sulphate from soils treated with sulphur, pyrites, and ferrous sulphate indicate that the micro-organisms causing the oxidation of organic soil-sulphur and elementary sulphur differ from those concerned in the oxidation of pyrites. The incomplete recovery of sulphate from soils treated with sulphur, pyrites, and ferrous sulphate in certain instances is ascribed to the formation of insoluble sulphates and to the reduction of sulphates. Both lime and magnesia decreased the outgo of potash from untreated soils and from those receiving sulphur additions. The outgo of nitrate was decreased by the sulphur treatments, but supplementary treatment with lime or magnesia more than counterbalanced this effect. Light dressings of magnesia increased the nitrate outgo more than equivalent lime dressings, but the reverse was the case with heavy applications. A. G. POLLARD.

**Influence of inorganic nitrogen compounds on reaction and replaceable bases of Norfolk sand.** R. M. BARNETTE and J. B. HESTER (Soil Sci., 1930, 30, 431—437).—The acidity of permanent lawn-grass plots on the sandy soil examined was increased by continuous treatment with ammonium sulphate and to a smaller extent by ammonium phosphate, but was decreased by sodium nitrate. A top dressing of 3 in. of clay reduced this effect with both ammonium salts. The increased hydrogen-ion concentration of the soil following ammonium sulphate treatment was proportional to the amount of sulphate applied and was correlated with the decrease in the total replaceable cation equivalents. Ammonium sulphate decreased the replaceable bivalent cation content to a greater extent than the univalent cation content, and a replacement of univalent cations by bivalent cations in the lower depths of the soil was indicated. A. G. POLLARD.

**Effect of organic matter on several physical properties of soils.** L. D. BAVER (J. Amer. Soc. Agron., 1930, 22, 703—708).—The presence of organic matter increases the absorptive capacity of the soil for cations from 30 to 60% and for water from 20 to 40%; it also raises both upper and lower plastic limits. Oxidation of the organic matter increases the percentage of clay. CHEMICAL ABSTRACTS.

**Sorptive properties of peats in the light of Freundlich's adsorption formula.** A. MUSIEROWICZ (Rocz. Nauk Roln. Les., 1929, 21, 129—152).—The sorption of the ions  $\text{Ca}^{2+}$ ,  $\text{K}^+$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  by Dublany and Mizun peats follows Freundlich's formula; differences are recorded. Fertilisation of the high-land peat with calcium increases the adsorption of potassium and ammonium. CHEMICAL ABSTRACTS.

**Nitrogen and organic matter in Hawaiian pineapple soils.** A. L. DEAN (Soil Sci., 1930, 30, 439—442).—The C:N ratio of the soils examined averaged 7:1 (cf. Jenny, B., 1929, 407). A. G. POLLARD.

**Preparation of soil samples for pipette analysis.** E. WINTERS, JUN., and M. B. HARLAND (J. Amer. Soc. Agron., 1930, 22, 771—780).—Air-dried soil (10 g.) is stirred during 1 hr. with 0.1N-hydrochloric acid (200 c.c.),

kept overnight, filtered, and washed with suction. It is then shaken for 48 hrs. with 0.11% sodium carbonate.

CHEMICAL ABSTRACTS.

**Soil sampler.** L. J. PESSIN (Science, 1930, 72, 459—460).—A sampler which enables a cylinder of soil of any desired length to be obtained from soils free from gravel or rock is described. L. S. THEOBALD.

**Mechanical analysis as an indicator of the impermeability in connexion with the dispersion of soils.** T. SAWARENSKY (Mat. Op-Stroj raooob. Mughan, 1929; Proc. Internat. Soc. Soil Sci., 1930, 5, 160—163).—Methods usually adopted for the preparation of a soil sample for mechanical analysis aim at obtaining maximum dispersion of the material, and for this reason do not give information as to the natural condition of the soil and its permeability to water. A method of aggregate rather than ultimate analysis for this purpose is desirable.

A. G. POLLARD.

**Microbiological soil analysis.** T. BAUMGÄRTEL (Landw. Jahrb., 1930, 71, 593—614).—Recent progress in the adaptation of microbiological methods to soil analysis is discussed, with special reference to Beijerinck's *Azotobacter* method. A. G. POLLARD.

**Nutritive value of Polish phosphorites as fertilisers.** H. SOWIŃSKA (Rocz. Nauk Roln. Les., 1929, 21, 1—18).—Comparative tests revealed no marked differences between five different phosphorites. Yields of oats (grain and straw) are recorded. CHEMICAL ABSTRACTS.

**Availability of phosphates [as fertilisers].** S. D. CONNER (Amer. Fertiliser, 1930, 72, No. 8, 16—18).—Average yields of barley for seven crops showed little difference in the relative availability of various insoluble phosphates when the phosphoric acid was applied alone and fixed by the soil, or fixed first by treatment with an excess of any one of several basic compounds. The availability of superphosphates is not adversely affected by admixture with calcium carbonate or with aluminium or iron silicate. CHEMICAL ABSTRACTS.

**Effect of artificial fertilisers on soil reaction.** S. T. JENSEN (Tidsskr. for Planteavl., 1929, 35, 380; Bied. Zentr., 1930, 59, 538—539).—A survey of changes in soil reaction and lime requirement as revealed by continuous fertiliser trials. A. G. POLLARD.

**Application of the colorimetric determination of phosphoric acid in Neubauer's seedling method.** K. PFEILSTICKER (Z. anal. Chem., 1930, 82, 276—284).—Neubauer's method for the determination of small quantities of phosphoric acid in plants or soils is modified as follows: the material is ignited to destroy organic matter and the residue digested with nitric acid to convert the phosphorus compounds into orthophosphoric acid; 1 c.c. of the solution is pipetted into 100 c.c. of water. The diluted solution is treated with 4 c.c. of molybdate solution (made by diluting a mixture of 200 c.c. of 10% ammonium molybdate solution and 200 c.c. of sulphuric acid to 1 litre) and 4 c.c. of stannous chloride solution (made by diluting a solution of 0.25 g. of tin foil in 5 c.c. of hydrochloric acid to 120 c.c.). After shaking for 15—20 min., the colour is compared in a Duboscq colorimeter with that of standards prepared in the same way.

A. R. POWELL.

**Influence of plants on the water and nutrient**

relationships of soils. V. DENISSIVSKI (Landw. Versuchs-Stat. Kiev, 1929, No. 28; Bied. Zentr., 1930, 59, 546—547).—Comparison is made of the effects of the growth of oats, peas, and potatoes on the nutrient contents of a strongly podsolised loam. The reduction in nitrate content of the soil after cropping was greatest with oats and least with potatoes. The withdrawal of water by the crop is one of the causes of retardation of nitrification in the soil. Cropping did not appreciably affect the water-soluble phosphate content of the soil. The water-soluble calcium content was greatest under peas and least under potatoes. The ammonia content of soil under peas and oats was smaller than in fallow soil, ammonification being retarded by the lowered moisture content of the cropped land. Towards the middle of the growth period the [water-soluble?] potash content of the cropped soils decreased. From spring to summer the water-soluble humus increased in both cropped and fallow soils. Variations in the nitrate and calcium contents of the subsoil during winter were directly related to the amounts of these nutrients present in the late spring. In fallow soil and potato plots the  $pH$  increased, partly as a result of increased nitrification. The decrease in moisture content of the cropped soils was in the order, potatoes < peas < oats.

A. G. POLLARD.

Relation of ash constituents of pasture plants to the oxidation-reduction potentials of nutrients. H. P. COOPER and J. K. WILSON (Soil Sci., 1930, 30, 421—430).—Changes in the mineral constituents of the herbage occurring during depletion of the soil are recorded. Nitrogen- and silica-free ash contents of grasses decrease with soil depletion. Elements with relatively high standard electrode potentials (e.g., K<sup>+</sup> and Ca<sup>2+</sup>) frequently predominate in plant ash. A strong negative correlation exists between the calcium and potassium contents of the ash of grasses. A correlation is suggested between the qualitative order of removal of metallic cations from soil colloids by electrodialysis and the order of the energy of removal, in equivalent volts, of the in-most normal valency electron of elements. Many organisms differentially absorb atomic nutrient cations in the same qualitative order as they are removed from soil colloids by electrodialysis. High nutrient demands by plants for optimum growth are associated with intolerance of shade and high feeding value, and *vice versa*. The quality of radiant energy necessary for the reduction of common nutrient ions varies considerably, phosphorus being one of the most difficult to reduce. Ionic phosphorus is more easily reduced than phosphorus in the molecular form.

A. G. POLLARD.

Crop rotation experiments. F. MÜNTER (Z. Pflanz. Düng., 1930, 9B, 529—552).—The yield of and nutrient intake by crops during numerous rotations is recorded. Sugar beet removed a greater proportion of nutrients from soils than any other crop examined, and was followed, in order, by potatoes and oats. Nitrogen deficiency following root crops was especially apparent in roots, oats, and barley, after oats in potatoes, and after wheat in roots. Phosphate deficiency was observed only in peas after roots. The best preparatory crops were, in decreasing order, legumes, potatoes, and sugar beet.

A. G. POLLARD.

Effect of manganese, copper, zinc, boron, and arsenic on growth of oats. J. S. McHARGUE and O. M. SHEDD (J. Amer. Soc. Agron., 1930, 22, 739—746).—Addition of small quantities of copper and zinc, as compared with manganese, increased the yield of straw and grain, whilst boron and arsenic decreased the yield of grain.

CHEMICAL ABSTRACTS.

Effect of fertilisers on yield and composition of wheat. H. F. MURPHY (J. Amer. Soc. Agron., 1930, 22, 765—770).—Phosphate (especially when combined with potash), but not nitrogen and/or potash, increases the yield of wheat on Kirkland sandy loam. The protein content of the wheat increases as the nitrogen is increased, but falls when phosphate is added.

CHEMICAL ABSTRACTS.

Effect of climate on nitrogen content of maize, barley, and red clover. E. J. DELWICHE and W. E. TOTTINGHAM (J. Amer. Soc. Agron., 1930, 22, 681—688).—Clover contains more protein when grown in South than in North Wisconsin; with hay and cereals no differences were observed.

CHEMICAL ABSTRACTS.

Influence of hydrogen-ion concentration on the development and growth of buckwheat. L. S. KACHIONI-VALTER (Bull. Sta. Plant Acclim. Detskoje Selo [Russia], 1927, No. 5, 73—96).—Buckwheat is very sensitive to hydrogen ions. In presence of chloride ion favourable development was obtained at  $pH$  5.0—6.5 and injury was observed at  $pH$  7—9. The influence of chloride depends on the  $pH$ ; in its absence the optimal range diminished.

CHEMICAL ABSTRACTS.

Rate of intake, accumulation, and transformation of nitrate nitrogen by small grains and Kentucky bluegrass. M. M. MCCOOL and R. L. COOK (J. Amer. Soc. Agron., 1930, 22, 757—764).—The intake by plants of nitrogen added to the soil can be detected in the expressed sap after 15 hrs. Changes of concentration of nitrate nitrogen in the soil are proportional to the changes in the sap. Ammonium sulphate, urea, and ammonium dihydrogen phosphate are slower than is sodium nitrate in affecting the nitrogen content of the sap.

CHEMICAL ABSTRACTS.

Phosphorus content of grassveld. M. HENRICI (Farming in S. Africa, 1930, 5, 29—30).—South African pasture grasses are low in phosphorus content; those in Bechuanaland contain more than those in the East Transvaal highveld. In the former the minimum value occurs during the summer drought, and in the latter in winter.

CHEMICAL ABSTRACTS.

Potash demand of grasses and leguminosæ. J. WEIGERT and E. HILTNER (Ernähr. Pflanze, 1930, 26, 172—175; Chem. Zentr., 1930, ii, 294).—Grasses react chiefly to nitrogen and leguminosæ to potash; leguminosæ exert a large demand on the soil potash.

A. A. ELDRIDGE.

Control of celery blights. J. D. WILSON and A. G. NEWHALL (Ohio Agric. Exp. Sta. Bull., 1930, No. 461, 30 pp.).—Celery blight was controlled by spraying with a 5:5:50 Bordeaux mixture or dusting with a 1:4 mixture of monohydrated copper sulphate and slaked lime. Copper dust mixtures containing copper in any other form were inferior. The admixture of inert materials (e.g., kaolin, fuller's earth), while improving

adhesiveness, did not add to the toxicity of the dusts. A. G. POLLARD.

**Dicalcium phosphate as a mineral supplement for dairy cows.** C. C. HAYDEN, C. F. MONROE, and C. H. CRAWFORD (Ohio Agric. Exp. Sta. Bull., 1930, No. 455, 26 pp.).—Significant differences in the daily or total milk production of cows due to the feeding of dicalcium phosphate were not observed where rations included legume hay with a grain mixture.

A. G. POLLARD.

**Soya beans and soya-bean oil meal for pigs.** W. L. ROBISON (Ohio Agric. Exp. Sta. Bull., 1930, No. 452, 42 pp.).—Pig-feeding trials show that a mineral salt supplement to soya-bean meal is necessary. Cooked beans gave better results than uncooked. Good-quality soya-bean meal (after oil extraction) used as a supplement to maize in pig rations was superior to the whole beans, but the quality, digestibility, and palatability of the meal varied considerably with the process of manufacture. Soft pork fat may result from feeding soya-bean oil or whole beans. The meal, having a lower oil content and correspondingly higher protein content, does not produce this effect.

A. G. POLLARD.

**Mechanical analysis of natural phosphates.** ALEXANDER and JACOB.—See VII. **Asphalts and bitumens on rubber estates.** SUTCLIFFE.—See XIV.

See also A., Jan., 132, **Influence of inorganic ions on seeds** (KURBATOV; GLÜCKMANN). 133, **Determination of acid-base balance in ash of plants** (FREAR). Cause of "wildfire" in tobacco (BÖNING). Rust diseases of cereals (TERÉNYI).

#### PATENTS.

**Fertilisers.** C. VILAIN (B.P. 336,651, 15.7.29).—Dolomite is treated with free phosphoric acid and the double phosphate produced is acted on by ammonia.

H. ROYAL-DAWSON.

**Manufacture of compound fertilisers and sodium bicarbonate.** SOC. CHIM. DE LA GRANDE-PARISSE, AZOTE ET PROD. CHIM. (B.P. 338,007, 19.11.29. Fr., 11.1.29. Addn. to B.P. 331,451; B., 1930, 924).—The double decomposition reaction (of the prior patent) between the potassium chloride and sodium nitrate is modified by effecting it in the mother-liquors of the process. Thus these salts may be added to the solution at that point in the manufacture at which the precipitation and separation of the bicarbonate have already taken place.

H. ROYAL-DAWSON.

**Manufacture of (A) concentrated, (B) [mixed], fertilisers.** A. HOLZ and T. V. D. BERDELL (B.P. 337,402 and 337,415, 30.7.29).—(A) Chlorine, prepared together with potassium hydroxide and hydrogen by the electrolysis of potassium chloride solution, is passed simultaneously with sulphur dioxide into water to yield a mixture of sulphuric and hydrochloric acids which is used to convert calcium phosphate into calcium sulphate, calcium chloride, and phosphoric acid; after filtration of the solution, the residual calcium sulphate is converted by treatment with the electrolytic potassium hydroxide solution and carbon dioxide into potassium sulphate and calcium carbonate, the latter being collected and the filtrate treated with sufficient calcium

hydroxide to precipitate dicalcium phosphate, which is removed, washed, and mixed with the potassium sulphate solution. The mixture is concentrated, granulated by spraying into warm air, and mixed with, e.g., ammonium nitrate. (B) Potassium chloride is treated below 200° with sufficient sulphuric acid to yield potassium bisulphate and to expel all the chlorine as hydrogen chloride, which is used for decomposing phosphatic ores in aqueous suspension with the formation of calcium chloride and phosphoric acid. After filtration the solution is treated with sufficient calcium hydroxide to precipitate the phosphoric acid as dicalcium phosphate, which is removed, dried at about 100°, and added to a mixture of potassium and ammonium sulphates prepared by treating the potassium bisulphate with ammonia.

L. A. COLES.

**Fumigating compound and its production.** K. F. COOPER, ASSR. to AMER. CYANAMID Co. (U.S.P. 1,754,148, 8.4.30. Appl., 24.5.23).—A mixture of 2–9 pts. of an inert material and 1 pt. of a cyanide, such as the crude material described in U.S.P. 1,359,257 (B., 1921, 9 A), capable of evolving gaseous hydrocyanic acid in presence of moisture is applied to the infected area as a dust. The inert diluent aids the uniform distribution of the cyanide and prevents damage, e.g., to the foliage of plants, which should also preferably be dry. If desired, an arsenical powder may also be employed capable of exerting a toxic effect after the escape of the hydrogen cyanide. The rate of application is 20–55 lb./acre.

C. JEPSON.

**Fungicidal product for seed treatment.** C. S. REDDY, ASSR. to QUAKER OATS Co. (U.S.P. 1,760,000, 27.5.30. Appl., 17.3.28).—Furan derivatives (e.g., furfuraldehyde, hydrofuranamide) are combined with mercury compounds after incorporation with an inert filler (e.g., talc).

A. G. POLLARD.

**Dressing seed.** H. RÖSSNER, A. STEINDORFF, and K. PFAFF, ASSRS. to FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (U.S.P. 1,783,200, 2.12.30. Appl., 8.10.25. Ger., 15.10.24).—See B.P. 241,568; B., 1927, 88.

#### XVII.—SUGARS; STARCHES; GUMS.

**Uba cane juice; [un-]trustworthiness of the Clerget method.** E. HADDON (S. African Sugar J., 1930, 14, 379).—Acidification with acetic acid of cloudy (lead-clarified) Uba cane juice causes further cloudiness due to an organic compound of silica. The Clerget method of analysis is inapplicable. The juice (100 c.c.) is boiled under reflux for 4 min. with barium hydroxide (2 g.); when cool it is treated with acetic acid (1 c.c.), lead-clarified, and polarised. CHEMICAL ABSTRACTS.

**Determination of the actual sugar content of filter-press cakes.** L. SHNAIDMAN (Nauk Zapiski Tzuk. Prom., 1930, 9, 161–166).—The cake is conveniently diluted with hot water, the calcium compound being decomposed with acetic acid.

CHEMICAL ABSTRACTS.

**Determination of invert sugar, particularly in presence of much sucrose.** G. BRUHNS (Deut. Zuckerind., 1930, 54, 1237, 1316–1317, 1337–1338; 55, 422–423, 486–487; Chem. Zentr., 1930, ii, 323).—Copper solution (10 c.c.), sodium potassium tartrate-sodium



hydroxide solution (10 c.c.), and sugar solution (20 c.c.) are well mixed in a 200-c.c. flask, heated rapidly, and boiled for exactly 2 min. over a small flame. During the heating a small quantity of finely-powdered talc is scattered over the surface of the liquid, which is not agitated. After addition of 50 c.c. of aerated distilled water the mixture is cooled to 15°; a solution of potassium thiocyanate (0.65 g.) and iodide (0.10 g.) and, after agitation, 5.5*N*-sulphuric acid (10 c.c.) are added and thiosulphate is run in until (in presence of starch) the precipitate is dark yellow to red and the liquid no longer becomes grey or blue in 5 min. A blank determination is performed with water instead of sugar solution. The difference corresponds with the copper precipitated. If the sugar solution contains substances which react with iodine, its "iodine titre" is determined instead of its "copper titre" by treatment of the copper-tartrate-sugar mixture with 50 c.c. of aerated distilled water without boiling, cooling to 15°, and titrating as above with thiosulphate. A. A. ELDRIDGE.

**Paper adhesives.** SHAW and others.—See V. **Temperature changes in sugar solutions.** JOSLYN and MARSH.—See XIX.

See also A., Jan., 47, **Inversion of sucrose by platinum-charcoal saturated with hydrogen** (BRUNS and WANJAN). 67, **Mesquite gum** (ANDERSON and OTIS).

#### PATENTS.

**Diffusion plant [for sugar beet etc.].** C. CAMUSET (U.S.P. 1,782,603, 25.11.30. Appl., 30.8.26. Fr., 25.9.25).—See B.P. 258,849; B., 1927, 664.

**Manufacture of crystalline dextrose.** W. B. NEWKIRK, ASSR. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,783,626, 2.12.20. Appl., 9.6.26).—See B.P. 254,729; B., 1927, 122.

### XVIII.—FERMENTATION INDUSTRIES.

**Degree of fermentation.** J. DE CLERCK (Bull. trimestr. Assoc. anc. Elèves Brass. Louvain, 1930, 30, 1—19; Chem. Zentr., 1930, ii, 324).—A discussion of differences in behaviour between fermentations in the vat and those in the flask. A. A. ELDRIDGE.

**Ester value, strength, and quality of rums and arracks.** H. WÜSTENFELD and C. LUCKOW (Mitt. Abtg. Trinkbranntwein- u. Likörfabr. Inst. Gärungsgewerbe, Berlin, 1930, 20, 2—6; Chem. Zentr., 1930, i, 3838).—The ester values of 83 rums lay usually between 200 and 800, but values up to 3000 were recorded; values for 23 arracks varied from 230 to 470. The requirements of control analysis are discussed. A. A. ELDRIDGE.

**Dough fermentation.** KARACSONYI and BAILEY.—See XIX.

See also A., Jan., 61, **Preparation of anhydrous alcohol and detection and determination of water in alcohol** (ADICKES). 122, **New enzymic oxidation [metaoxidase]** (BOAS). 124, **Determination of peptic activity** (GILMAN and COWGILL).

#### PATENTS.

**Distillation and rectification of alcohol.** E. A. BARBET (B.P. 338,569, 16.5.29).—Fermented wines are distilled and subjected to two successive rectifications,

the second of which is effected by using only the heat from the vapours from the distilling column. A greater economy of steam is obtained by carrying out the second rectification in a vacuum, the vacuum rectifier being heated by the vapours from the top of the first rectifier, and using the vapour from the distilling column solely for heating the primary rectifier. C. RANKEN.

**Preparation of acetylmethylcarbinol and diacetyl [by fermentation].** T. H. VERHAVE, SEN. (B.P. 337,025, 26.6.29. Addn. to B.P. 315,264; B. 1930, 1044).—In the process of the prior patent the aeration, insufficient to prevent formation of  $\beta$ -butylene glycol during fermentation, is continued until the glycol is oxidised. C. HOLLINS.

**Determining the degree of saturation of materials after immersion** (B.P. 338,282).—See I. **Food composition** (B.P. 318,522).—See XIX.

### XIX.—FOODS.

**Colloid chemistry of gluten.** III. H. L. B. DE JONG and W. J. KLAAR (Cereal Chem., 1930, 7, 587—591; cf. B., 1930, 787).—When an acid solution of gliadin is diluted with acetone-water mixtures of varying concentration of acetone and the viscosity determined, a maximum value is obtained with a mixture containing 44 vol.-% of acetone. E. B. HUGHES.

**Relation of the overgrinding of flour to dough fermentation.** L. P. KARACSONYI and C. H. BAILEY (Cereal Chem., 1930, 7, 571—587).—Samples of overground wheat flour showed a marked increase in diastatic activity, due probably to the easier accessibility of the starch. No substantial effect was apparent in the dough fermentation, and the gas production and gas retention were not appreciably altered. E. B. HUGHES.

**Relation between crude protein content and loaf volumes obtained by two different methods of baking.** R. H. HARRIS (Cereal Chem., 1930, 7, 557—570).—A large number of samples of hard red spring wheat were milled experimentally and subjected to baking tests by the standard and bromate-and-malt methods. Increase in loaf volume was found to correspond with increase in protein content in loaves baked by the latter method. Where the increase in volume over the standard method was greater than 10% the sample was rebaked with malt and arakady, and greater increases were obtained. On mathematical analysis the results showed that the coefficients of correlation between baking strength and protein content obtained appeared to justify Larmour's conclusion (B., 1930, 583) that commercial use of the protein test is justified as a factor in the classification of hard red spring wheat. E. B. HUGHES.

**Utility of protein peptisation by inorganic salt solutions as a means of predicting loaf volume.** W. F. GEDDES and C. H. GOULDEN (Cereal Chem., 1930, 7, 527—556).—Gortner's method (B., 1929, 262) for determining the peptisation of wheat protein by inorganic salt solutions has been applied to a large number of flours experimentally milled from hard red spring wheat and including immature and frosted wheat. Baking tests by the standard and bromate-and-malt methods and protein determinations were also made on all samples. From a mathematical analysis of the results, and of those



obtained by Gortner and by MacLeod, it was concluded that, on the whole, the total protein value was a better measure of quality than the percentage of protein peptised. E. B. HUGHES.

**Clarification of milk for American Cheddar cheese.** G. WILSTER (Iowa State Coll. J. Sci., 1930, 4, 181—213).—Effects of various methods of clarification of milk on the bacterial numbers and quality of cheese produced are examined. A. G. POLLARD.

**Does nickel dissolved from the container during pasteurisation catalyse the destruction of the vitamins of milk?** A. D. PRATT (J. Nutrition, 1930, 3, 141—155).—Pasteurisation in a nickel container introduced nickel ( $15 \text{ in } 10^6$ ) into milk. Pasteurisation in a glass or nickel container did not appreciably destroy vitamin-A. The antineuritic factor of the vitamin-B complex and vitamin-C were partly destroyed, but there was no evidence of catalysis of the destruction by nickel.

CHEMICAL ABSTRACTS.

**Testing sweetened condensed milk by a modified Babcock method.** W. D. SWORE (Ice Cream Trade J., 1930, 26, No. 8, 53—54).—In the Babcock method, 9 g. of a 1:1 mixture of water and sweetened condensed milk are mixed with concentrated ammonia solution (2 c.c.), butyl alcohol (3 c.c.), and sulphuric acid ( $d \ 1.73$ ;  $17.5 \text{ c.c.}$ ).

CHEMICAL ABSTRACTS.

**Large-scale experiments in sulphuring apricots.** E. M. CHACE, C. G. CHURCH, and D. G. SORBER (Ind. Eng. Chem., 1930, 22, 1317—1320).—Experiments have been made on a semi-commercial scale to investigate the effect of concentration, temperature, and period of sulphuring on the grade of the fruit and on the proportion of sulphur dioxide retained by it. Fruit from one district and during one season only was used. A method of grading devised for the experiments is described. Boxes having a capacity of 1000 litres of air-space and capable of holding 20 kg. of fresh fruit were used, these boxes being thermally controlled and equipped with mixing fans. Although the greater part of the absorption takes place in the first  $\frac{1}{2}$  hr., periods of less than 2 hrs. give unreliable results at concentrations below 3%. Concentrations above 3% lead to too great retention of sulphur dioxide; 2% concentration may be used if the sulphuring period is 3 hrs. at least, but concentrations below 2% are unreliable. Temperatures below  $26^\circ$  would probably lead to retarded absorption, but above  $50^\circ$  the fruit may develop a reddish or cooked appearance. W. J. BOYD.

**Carbon dioxide storage of fruits, vegetables, and flowers.** N. C. THORNTON (Ind. Eng. Chem., 1930, 22, 1186—1189).—The fruit etc. was held in storage for from 2 to 7 days, and the tolerance to carbon dioxide at six different temperatures ( $32$ — $77^\circ$ ) recorded. For some fruit ripening was retarded; pears were improved in flavour. Flowers in bud kept longer owing to later opening of the bud. Data are given for the common commercial fruits and vegetables and for a number of flowers. E. B. HUGHES.

**Heat transfer in foods during freezing and subsequent thawing.** I. Temperature changes in sugar solutions, sweetened fruit juices, and other liquids.

M. A. JOSLYN and G. L. MARSH (Ind. Eng. Chem., 1930, 22, 1192—1197).—When solutions containing sugar were cooled in air in an apparatus of a refined technical type, the temperature changes during freezing and thawing indicated that the process was one of conduction, and that convection was important only in the later stages of thawing. The viscosity of the sugar solutions apparently had very little effect and the chief factors affecting the rate of change in temperature were the sp. heat, the heat conductivity, the temperature of the first formation of ice, and the percentage of water present as ice. There is no simple relationship between the percentage of sugar present and the cooling rate. E. B. HUGHES.

**Vitamins-A and -D in fish oils.** NELSON and MANNING.—See XII. **Soya beans for pigs.** ROBISON.—See XVI.

See also A., Jan., 39, **Binding materials in cooking** (ZIEGELNAYER). 119, **Toxic action of baking-powders containing alum** (CERIOTTI).

PATENTS.

**Flour and dough.** J. DAPPER (B.P. 335,214, 19.6.29).—The baking property of flour or dough is increased by adding to the materials a mixture of persulphates and bromates, any tendency to evolve bromine thereby being prevented by the further addition of magnesium or calcium carbonate. H. ROYAL-DAWSON.

**Food composition and its production by fermentation.** P. L. MUSAE (B.P. 318,522, 4.9.29. U.S., 4.9.28).—A mixture of carob-bean and soya-bean flours is added to the dough of any fermented bakery product. The carob is rich in natural sugar which is released slowly during fermentation and the soya supplies protein, the result being to increase lactic fermentation and restrain the formation of butyric and acetic acids.

E. B. HUGHES.

**Stabilisation of cereal beverages.** D. COZZOLINO (U.S.P. 1,766,428, 24.6.30. Appl., 16.7.27).—The undesirable proteins of cereal beverages containing less than 0.5% of alcohol may be removed by the addition of 1—3 pts. of tannin and 2—6 pts. of silicious clay to 50,000 pts. of the beverage. After 5 days the precipitate may be filtered off, when the beverage will be found not to change in appearance on chilling or pasteurising. E. B. HUGHES.

**Filter of active adsorptive composition for freeing infusions of coffee from caffeine and roasting toxins.** W. PÄFFGEN (B.P. 339,543, 31.1.30. Ger., 18.9.29).—Coffee infusions are filtered through paper containing small amounts of highly active carbon.

E. B. HUGHES.

**Production of liquid food.** C. BAUER, Assr. to VET. PRODUCTS LABORATORY, INC. (U.S.P. 1,766,590, 24.6.30. Appl., 17.4.28).—Cows' milk is modified for the use of young animals (puppies and the like) by the addition of lactic acid and sugar. E. B. HUGHES.

**Evaporators for milk etc.** (B.P. 334,355). **Grinder** (U.S.P. 1,768,713). **Determining the degree of saturation of materials after immersion** (B.P. 338,282). **Sterilisation of liquids** (B.P. 338,422).—See I. **Lecithin preparations** (B.P. 317,730).—See III. **Working of butter etc.** (B.P. 338,053).—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Determination of quinine in tabloids and ampoules.** E. J. EMMANUEL (Z. anal. Chem., 1930, 82, 296—297).—The material is extracted with light petroleum to remove paraffin and the residue is dried, powdered, and digested with 50 c.c. of 1% hydrochloric acid at 50—60°. The clear solution is treated with 20 c.c. of 10% sodium hydroxide solution and the quinine removed by three extractions with 20 c.c. of chloroform. The resulting quinine solution is evaporated to dryness and the residual quinine weighed after heating to constant weight at 100°.

A. R. POWELL.

**Oil of amber.** T. T. COCKING (Perf. Ess. Oil Rec., 1930, 21, 477—478).—The analyses of a large number of samples of "Oleum succini rect." from three different English distillers indicate that the constants of these oils are very variable, especially the boiling range, and it is suggested that the Codex specification be replaced by the following:  $d$  0.850—0.875,  $\alpha$   $-12^\circ$  to  $+12^\circ$ ,  $n_D^{20}$  1.465—1.482, solubility in 90% alcohol 1 in 5—6 vols. Oil prepared from genuine amber chippings had:  $d$  0.935,  $\alpha$   $+20^\circ$ ,  $n_D^{20}$  1.5155, acid value 13.0, ester value 2.0, saponif. value 15.0, boiling range below 150° nil, 150—200° 15%, 200—250° 17%, 250—300° 43%, and it is shown from an examination of the products of distillation of ordinary colophony that distillation at a high temperature followed by careful fractionation is necessary to produce the oil known commercially as oil of amber.

E. H. SHARPLES.

**Three oil-bearing grasses from Burma.** D. RHIND (Agric. J. India, 1930, 25, 327—329).—Three species of *Cymbopogon* grass are described *C. clandestinus*, Stapf., on distillation gives 0.26% of oil (on dry grass) and *C. nov. spec.* a yield of 0.72% (for analyses cf. B., 1930, 166). Both oils have odours resembling that of ginger-grass oil. *C. virgatus*, Stapf., *nov. spec.*, on distillation gives 0.55% of an oil with an unpleasant odour having  $d_{25}^{20}$  0.9556,  $\alpha$  (100-mm. tube)  $-28^\circ 53'$ ,  $n_D^{20}$  1.49107, acid value 1.9, ester value 6.5, ester value after acetylation 72.8, alcohol content (as  $C_{10}H_{17}OH$ ) 21.2%.

E. H. SHARPLES.

See also A., Jan., 28, Periodates [of alkaloids] (ROSENTHALER). 55, Determination of orthophosphate ion (DEL CAMPO). 66, Bacterial oxidation of oxalates (SCHOLDER). 82, *p*-Aminophenylguanidine hydriodide (BRAUN). 88, Reactions of salicylic acid and salol (EKKERT). 94, Reactions of *g*- and *k*-strophanthin (EKKERT). 99, Reaction of antipyrine and pyramidone (RIBÈRE). 106, New sulphur derivatives of aromatic arsenicals (EVERETT). 119, Assay of strophanthin solutions (FASCHING). 120, Assay of aconitine solutions (BRANDT). 126, Pituitary gland preparations (ABEL). 127, Bovine parathyroid gland preparations (TWEEDY). 128, Technical insulin and its crystalline preparations (BÜRGER and KRAMER). 132, Paraffins of tobacco (KURILO). Microanalytical tobacco determinations (BODNÁR and others). 133, Cause of "wildfire" in tobacco (BÖNING).

## PATENTS.

**Increasing the biological value of fatty substances in ointments and like external therapeutic agents.** O. RIED (B.P. 316,264, 25.7.29. Austr., 26.7.28).—Fatty

substances in ointments, creams, etc., either alone or mixed with other substances, are mixed with powdered metals or metal oxides previous to irradiation with ultra-violet light or X-rays.

E. H. SHARPLES.

**Manufacture of ether derivatives of aminobenzoic alkamine esters [tert-aminoalkyl aminoalkoxybenzoates. Local anæsthetics].** SCHERING-KAHLBAUM A.-G. (B.P. 317,296, 12.8.29. Ger., 10.8.28).—The presence of a nuclear alkoxy group in aminoalkyl aminobenzoates enhances the anæsthetic activity. 4-Nitro-3-methoxybenzoyl chloride, m.p. 51°, reacts with  $\beta$ -diethylaminoethyl alcohol in boiling benzene (or with ethylene chlorohydrin, followed by diethylamine) to give  $\beta$ -diethylaminoethyl 4-nitro-3-methoxybenzoate hydrochloride, m.p. 143°, which is reduced to the amino-ester monohydrochloride, m.p. 156°. The following are also described:  $\beta$ -1-piperidinoethyl 4-amino-3-methoxybenzoate monohydrochloride;  $\beta$ -diethylaminoethyl 3-nitro-4-ethoxybenzoate hydrochloride, m.p. 133°, and the amino-ester monohydrochloride, m.p. 162—163°;  $\beta$ -diethylaminoethyl 3-nitro-4-isoamyloxybenzoate hydrochloride, m.p. 123°, and the amino-ester monohydrochloride, m.p. 115°;  $\beta$ -diethylaminoethyl 3-amino-4-methoxybenzoate dihydrochloride, m.p. 223°. New intermediates are: 3-nitro-4-ethoxybenzoic acid, m.p. 196—197° (chloride, m.p. 83—84°); 3-nitro-4-isoamyloxybenzoic acid, m.p. 178° (ethyl ester, m.p. 55—56°).

C. HOLLINS.

**Preparation of hydrogenated pyridine and piperidine derivatives.** A. BOEHRINGER (B.P. 314,019, 11.6.29. Ger., 21.6.29. Addn. to B.P. 312,919; B., 1930, 1058).—Pyridines having in 2- or 2:6-positions side-chains of the type  $CH_2 \cdot CO \cdot R$  ( $R$  = alkyl, aralkyl, or aryl) are hydrogenated in the side-chain, with or without hydrogenation of the nucleus. 2:6-Diphenacylpyridine hydrochloride in acetic acid with hydrogen and palladium at 15° gives 2- $\beta$ -phenylethyl-6- $\alpha$ -hydroxy- $\beta$ -phenylethylpyridine (hydrochloride, m.p. 190°), and at 40—50° 2:6-di-( $\beta$ -phenylethyl)pyridine, m.p. 153° (hydrochloride, m.p. 160°). With platinum oxide catalyst at 15° 2:6-di-( $\alpha$ -hydroxy- $\beta$ -cyclohexylethyl)piperidine, m.p. 70° (hydrochloride, m.p. 260°), is obtained.

C. HOLLINS.

**Manufacture of readily soluble salts of acridine bases substituted in the 9-[meso]-position by an amino-group.** I. G. FARBERIND. A.-G. (B.P. 337,136, 19.9.29. Ger., 8.10.28).—The acetates of 9-aminoacridines are more soluble in water than the lactates. Acetates of 3:9-diamino-7-ethoxy-, 9-amino-, 9-amino-7-methyl-, and 3:9-diamino-acridines are described.

C. HOLLINS.

**Manufacture of *N*-substituted benziminazolone-arsinic acids.** I. G. FARBERIND. A.-G. (B.P. 337,299, 28.1.30. Ger., 11.2.29. Addn. to B.P. 256,243; B., 1927, 670).—The arsinic acids of the prior patent are also obtainable by using a chloroformic ester in place of carbonyl chloride.

C. HOLLINS.

**Preparation of basic nitro-derivatives of 9-aminoacridine.** H. JENSCH and O. EISLEB, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,782,727, 25.11.30. Appl., 6.1.28. Ger., 11.1.27).—See B.P. 283,510; B., 1929, 577.

Synthesis of diacetyl-3 : 3-diamino-4 : 4'-di-hydroxy-5 : 5'-di-iodoarsenobenzene. A. D. MAC-ALLUM (U.S.P. 1,782,432, 25.11.30. Appl., 30.3.28. U.R., 4.5.27).—See B.P. 300,286; B., 1929, 150.

Manufacture of organic metalmercaptosulphonic [acid] compound. W. SCHÖELLER, E. BORWARDT, and A. FELD, Assrs. to CHEM. FABR. AUF AKT. (VORM. E. SCHERING) (U.S.P. 1,784,497, 9.12.30. Appl., 26.4.27. Ger., 5.5.26).—See B.P. 270,729; B., 1927, 860.

Caffeine and toxins from coffee (B.P. 339,543).—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Detection of sodium thiosulphate in motion-picture film. J. I. CRABTREE and J. F. ROSS (J. Soc. Motion Picture Eng., 1930, 14, 419).—Strips of processed film are placed in a solution containing mercuric chloride and potassium bromide. If the film contains an appreciable quantity of sodium thiosulphate the solution becomes opalescent, the turbidity being approximately proportional to the quantity present; 0.05 mg. can be detected. Factors determining the rate of fading of silver images have been outlined.

### CHEMICAL ABSTRACTS.

See also A., Jan., 48, Laying bare of ripening centres (LÜPPO-CRAMER). 58, Colorimetry without comparative standards (HOCK and MÜLLER). Measurement of hardness and absorption of X-rays (GREBE and SCHMITZ).

### PATENTS.

Photographic light-sensitive material. KODAK, LTD., Asses. of S. E. SHEPPARD and E. P. WIGHTMAN (B.P. 337,323, 19.2.30. U.S., 25.2.29).—Chemical fogging is minimised by incorporating in the emulsion a substance of the type  $R \cdot X \cdot NH \cdot Y \cdot R$ , where R is a nitrogen-free organic radical, and X and Y are carbonyl, sulphonyl, methylene, or, in cyclic compounds, methinyl or nitrogen. Examples are diacetamide, phthalimide, pyrrole, succinimide, saccharin, pyrazoles, pyrazolidones, 1 : 2 : 3-triazoles. C. HOLLINS.

Production of kinematograph film positives. J. E. THORNTON (B.P. 339,977, 10.8.29. Addn. to B.P. 316,338).

[Elimination of moiré effects in] kinematography and photography in colours. SOC. FRANÇ. DE CINÉMAT. ET DE PHOT. FILMS EN COULEURS KELLER-DORIAN, and I. KITROSER (B.P. 317,060, 9.8.29. Fr., 9.8.28).

Colouring of films. W. W. TRIGGS. From MULTI-COLOR FILMS, INC. (B.P. 339,971, 17.9.29).

Reproduction of artificial wood-grain by photographing a smooth board. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 340,131, 19.12.29. Ger., 26.1.29).

## XXII.—EXPLOSIVES; MATCHES.

### PATENTS.

Apparatus for digestion of nitrocellulose or purification of cellulose. HERCULES POWDER CO., Asses. of M. G. MILLIKEN (B.P. 338,941, 30.7.29. U.S., 29.5.29. Addn. to B.P. 301,267; B., 1929, 113).—In

the apparatus described in the prior patent, the valve may be employed in conjunction with, or replaced by, a standpipe about  $3\frac{1}{2}$  in. in diam. and 168 ft. high, so as to give a back-pressure on the coil of 72 lb./in.<sup>2</sup> The standpipe leads into the tank used for collecting the treated nitrocellulose. W. J. WRIGHT.

Purification of explosives [e.g., tetryl]. D. M. JACKMAN (U.S.P. 1,767,798, 24.6.30. Appl., 24.7.25).—The explosive is dissolved in a solvent which is insoluble in water and at a temperature near its b.p., and agitated with an equal volume of water at approximately the same temperature as that of the solution. W. J. WRIGHT.

Pure glycerin (B.P. 336,608).—See III. Pyroxylin solutions (U.S.P. 1,768,253).—See XIII.

## XXIII.—SANITATION; WATER PURIFICATION.

Determination of ethylene oxide in air disinfected with preparations containing the oxide. W. DECKERT (Z. anal. Chem., 1930, 82, 297—307).—A measured volume of the air is drawn at the rate of 250 c.c./min. through 0.1N- or 0.01N-hydrochloric acid containing 22% of sodium chloride. The solution is heated at 70° to convert the ethylene oxide into glycol chlorohydrin according to the equation  $C_2H_4O + HCl = OH \cdot CH_2 \cdot CH_2 \cdot Cl$ . The excess hydrochloric acid is titrated with 0.1N- or 0.01N-sodium hydroxide, using methyl-orange as indicator (1 c.c. of 0.1N-hydrochloric acid = 4.4 mg.  $C_2H_4O$ ). A. R. POWELL.

Volumetric determination of sulphate in water and its limits of accuracy. R. SCHMIDT (Z. anal. Chem., 1930, 92, 353—361).—Various modifications of the barium chromate method have been tested and the following procedure is recommended. The water (100 c.c.) is treated with 20 c.c. of a 0.5% solution of barium chromate in 0.2N-hydrochloric acid and, after shaking and settling for 30 min., the solution is treated with ammonia until the colour changes to a light greenish-yellow and is then diluted to 200 c.c. in a graduated flask; 100 c.c. of the solution are filtered through a dry paper, treated with 1 g. of potassium iodide and 10 c.c. of 25% hydrochloric acid, and titrated with thiosulphate. From the result 0.8 c.c. is deducted and the remainder multiplied by 5.33 gives the  $SO_3$  content of the water in mg./litre. The mean deviation of the results from those obtained gravimetrically is  $\pm 1.7$  mg./litre. A. R. POWELL.

See also A., Jan., 55, Rapid detection and determination of fluorine in mineral waters (CASARES and CASARES). 58, Condenser for low-temperature evaporation of water (EVANS and others). 126, Effect of electrolytic "chlorogen" on bacterial spores (AYYAR).

### PATENTS.

Composition for destroying rats and mice. A. ZIMMERMANN (B.P. 336,913, 5.2.30. Ger., 6.2.29).

[Means for supplying salts in] softening of water [by base-exchange]. UNITED WATER SOFTENERS, LTD., R. T. PEMBERTON, and H. S. LAWRENCE (B.P. 340,091, 7.11.29 and 24.2.30).

Separating dust from air (B.P. 339,514).—See I. Fumigant (U.S.P. 1,754,148).—See XVI.

## B.—APPLIED CHEMISTRY

FEB. 27 and MAR. 6, 1931.\*

## I.—GENERAL; PLANT; MACHINERY.

Use of mercury for power production. F. SAMUELSON (Proc. Inst. Mech. Eng., 1930, 765—779).—An account is given of the theory and development of the mercury-steam turbine and the plant at Hartford, Conn., is illustrated diagrammatically and its operation explained. The turbine carries loads of 10,300 kw. and requires 6 lb. of mercury per kw.; it operates at a mercury pressure of 70 lb./in.<sup>2</sup> and a steam pressure of 350 lb./in.<sup>2</sup> A. R. POWELL.

Theory of alternating heat regenerators. L. THIBAUDIER (Rev. Mét., 1930, 27, 544—559).—Mathematical. Expressions are derived for the rate of heat transfer and for calculating the optimum thickness of brick in various arrangements of alternating heat regenerators. A. R. POWELL.

Heat transmission in an experimental inclined-tube evaporator. C. M. LINDEN and G. H. MONTILLON (Ind. Eng. Chem., 1930, 22, 708—713).—The apparatus used consisted of an inclined copper tube surrounded by a jacket through which steam passed downwards. An entrainment separator was provided in the vapour space. Liquid flow was measured by the deflection of a copper screen bobbin attached to a coil spring. This value was independent of the temperature. Vapour and pipe temperatures were measured at temperature differences of 5—16° for evaporation at 82°, 91°, and 99°, and the overall and individual heat-transfer coefficients were calculated. The overall coefficients were greater than those recorded by other observers for vertical tubes under similar conditions, but less than with horizontal tubes. The liquid-film coefficients were found to increase with temperature difference and with evaporation temperature. The logarithmic mean velocity of liquid + vapour upwards within the tube also varies with the temperature difference, so that the liquid-film coefficient bears a definite relation to this velocity within about 10% deviation. The steam-film coefficients were calculated. C. IRWIN.

Heat transfer from a gas stream to a bed of broken solids. II. C. C. FURNAS (Ind. Eng. Chem., 1930, 22, 721—731; cf. B., 1930, 221).—This study was made in order to obtain quantitative thermal data on the working of the blast furnace, and is based on a development of theoretical curves obtained by Schumann for the temperature history of solids and gas at different points in the solid bed. The coefficients of heat transfer between a gas and a column of irregular solids were determined by forcing all the gas at a given plane through a relatively small orifice and determining its temperature. The apparatus used is described. Hot flue gases

(up to 1100°) were used in work with iron ore; with coal and coke the limit was 300°. Data obtained agreed closely with the computed curves. The procedure gives both the coefficient of heat transfer and the heat capacity of the charge. It was found that for beds of broken materials the coefficient of heat transfer varies with the 0.7 power of the mass velocity of the gas, at the (high) velocities studied. The effect of temperature on the coefficient was found to be small and within the range of experimental error. Considerable differences were found between different materials (iron ore, limestone, coke, and coal), and the effects of voids and particle size were also studied. The cooling of coke with air was found to give a much higher coefficient of transfer than its heating. This phenomenon is unexplained, and does not occur with iron ore. No effect of the heats of transition of ferric oxide could be detected. The calcination of limestone, whilst modifying the apparent heat capacity, does not affect the coefficient of heat transfer. Equations are developed covering the "steady state" in countercurrent flow. An example is given of the calculation of solid and gas temperatures within a cupola, the feed of iron, coke, and air and the composition of the exit gases being known. C. IRWIN.

Deposition of dust from air in motion and its application to the theory of the dust filter. F. ALBRECHT (Physikal. Z., 1931, 32, 48—56).—The theory of the deposition of dust from air currents in a cylinder is first discussed. The results obtained are tested experimentally by the examination of mists of frost deposited on cylinders. The conclusions arrived at through the theoretical investigation are applied to the examination of the properties of dust filters. Several of these are described and their action is analysed. The effect of turbulence is mentioned. A. J. MEE.

Purification of coal gas. THAU. Gas analysis apparatus. SCHMIDT.—See II. Evaporation of caustic soda. BADGER and others.—See VII. Protective coatings on iron. TILLMANS and others.—See X. Gas cleaning. LÉVÊQUE.—See XI. Fractional distillation. JANTZEN and TIEDCKE.—See XII. Water softening. MCGARIGLE.—See XXIII.

## PATENTS.

[Open-hearth] furnace. A. M. Gow (U.S.P. 1,769,220, 1.7.30. Appl., 17.6.21).—Natural or other rich gas cannot be preheated in an ordinary regenerator because of deposition of carbonaceous matter, hence combustion in an open-hearth furnace is delayed somewhat. In this invention highly preheated air is blown into the gas riser in line with the gas port, thus producing

\* The remainder of this set of Abstracts will appear in next week's issue.

a heated mixture. The heated air is driven by the injector action of a smaller quantity of compressed air.  
B. M. VENABLES.

**Rotary furnaces or dryers.** F. A. JOHNSON (B.P. 339,595, 4.9.29).—The total material is passed, together with a portion of the drying or heat-treatment medium, e.g., gases from a coke fire, through a first portion of a rotating cylinder, which is of smaller diameter than that of the remaining length. At the change of diameter provision is made for screening out the finer material which is fully dried, outlet of the gaseous medium, and admission of a fresh quantity which acts only on the over-size material while traversing the remaining part of the cylinder.  
B. M. VENABLES.

**Rotary heat-treatment furnace and lining.** E. H. MIX, ASSR. to REPUBLIC STEEL CORP. (U.S.P. 1,768,776, 1.7.30. Appl., 8.2.29).—A form of helically ribbed metallic lining for a rotary cylindrical furnace is described.  
B. M. VENABLES.

**Surface apparatus for heating or cooling liquids.** R. MORTON & Co., LTD., and P. ROBINSON (B.P. 339,869 and 339,870, 7.2.30).—Tubular heat exchangers are described. A number of inner tubes (conveniently 7) are arranged within a larger tube which carries the outer fluid, the space for the latter being divided by radial and cylindrical baffles; cored headers are provided at the ends of the inner tubes so that both liquids flow in a zig-zag course.  
B. M. VENABLES.

**Conducting catalytic exothermic gaseous reactions [under pressure].** R. WILLIAMS, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,769,873, 1.7.30. Appl., 6.11.25).—Various combinations of heat exchange between the entering and leaving gases, and between them and the catalyst mass, are claimed.  
B. M. VENABLES.

**Heat exchanger.** O. A. LABUS, ASSR. to AMER. CARBONIC MACHINERY Co. (U.S.P. 1,769,265, 1.7.30. Appl., 21.4.27).—A compact type of exchanger is described which has the passage for the outer fluid in the shape of a flat helix bounded by two cylindrical shells and a helical strip; the inner fluid flows in parallel through a number of helical tubes arranged abreast in the outer passage.  
B. M. VENABLES.

**Pulverising machine.** C. M. STONER, ASSR. to WOLF Co. (U.S.P. 1,769,867, 1.7.30. Appl., 5.11.27).—The construction of the machine is similar to that of an impact pulveriser with semi-circular screen round the lower half, but the action is mainly by the shearing action of hammers against cutting bars.  
B. M. VENABLES.

**Pulverising machinery.** W. J. LIBBEY (U.S.P. 1,769,352, 1.7.30. Appl., 7.7.28).—In a pulveriser of the centrifugal roll or other type a system of ploughs rotating at a lower speed than that of the pulveriser is provided to prevent accumulation of flat lumps of clogging material (e.g., compressed litharge).  
B. M. VENABLES.

**Grinding machine.** L. BORN (U.S.P. 1,769,823, 1.7.30. Appl., 26.11.28).—A form of ball or pebble mill comprising a closed cylinder easily removable from a driving frame is described.  
B. M. VENABLES.

**Mixing and separating or classifying apparatus.** K. S. VALENTINE, ASSR. to TURBO-MIXER CORP. (U.S.P.

1,768,870, 1.7.30. Appl., 4.4.29).—A tank is divided into three superposed zones by perforated horizontal screens, the central zone being maintained in active agitation, the upper one containing quiet lighter liquid suitable for decantation, and the lower one settled heavy liquid and sludge.  
B. M. VENABLES.

**Mixing apparatus.** J. PETERS, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,768,926, 1.7.30. Appl., 28.3.29).—A mixing device with motor is mounted on a counter-poised parallel motion so that it can be raised and lowered into a bowl which is itself mounted on wheels.  
B. M. VENABLES.

**Multi-stage mixing apparatus.** H. S. BEERS, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,768,735, 1.7.30. Appl., 13.4.29).—The mixer comprises alternate rings of fixed and moving blades similar to a radial-flow turbine, but the blades are provided with raised teeth acting as disintegrators.  
B. M. VENABLES.

(A) Mixer. (B) Mixing apparatus. (C) Mixing or emulsifying apparatus. J. JOHNSON, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,768,955—7, 1.7.30. Appl., [A] 14.11.28, [B] 10.1.29, [C] 7.1.29).—Mixing devices, somewhat similar to that described in U.S.P. 1,768,735 (preceding abstract), have a secondary circulation produced by a device higher up the shaft.  
B. M. VENABLES.

**Mixing and cooling apparatus.** J. PETERS, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,769,890, 1.7.30. Appl., 29.5.29).—A mixing tank is submerged in a tank containing cooling fluid and is easily removable therefrom. The mixing device is attached to the bottom of the inner tank and is driven by a packed shaft passing through the bottoms of both tanks from a motor and gearing below.  
B. M. VENABLES.

**Emulsifiers, mixers, and the like.** W. J. DAVY (B.P. 339,751, 8.11.29).—The materials to be emulsified are passed through sinuous passages contained in the thickness of the piston of a hand-operated pump. One pattern comprises simply a double-acting piston in a cylinder closed at both ends. In another type a single-acting piston operates in a cylinder with an inlet valve at the bottom and a closure at the top having small apertures (causing further emulsification); no outlet valve is necessary, and the pump is contained in a larger cylindrical reservoir from and to which the material is circulated until sufficiently emulsified.  
B. M. VENABLES.

**Rotary filters.** A. R. JAHN (B.P. 339,669, 4.9.29).—A pressure filter having rotating leaves is provided with means to remove thickened sludge from the leaves as fast as it is formed; thus the sludge falls to a sump below the prefilter compartment and passes to an additional filter of the archimedean type.  
B. M. VENABLES.

**Filtering of viscous material.** H. PRENTICE (U.S.P. 1,769,388, 1.7.30. Appl., 2.5.28).—The material is supplied to a central feed well, which is then closed and external heat is applied to the surrounding vessel. When the material is melted air pressure is applied to the top of the feed well and the fluid forced upwards through strata of filtering medium contained in the annular space between the feed well and the wall of

the vessel. The strata are supported by perforated diaphragms and may be heated by internal steam coils.

B. M. VENABLES.

**Centrifuge.** H. E. T. HAULTAIN (B.P. 1,768,761, 1.7.30. Appl., 9.8.29).—The upper bearing of a high-speed centrifuge is allowed movement which is restrained by spring fingers which are more than 60° but less than 180° apart. The bowl is formed of a tube which is given a corrugated interior surface by means of alternate rings of metal and compressible material. B. M. VENABLES.

**Separation of finely-divided impurities from liquids.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 339,636, 7.6.29).—Very fine solid or colloidal materials suspended in oleaginous or tarry liquids are removed by the use of a filter medium or filter aid composed of finely-divided, semi-solid, carbonaceous residues containing oily, tarry, or asphaltic material, such as those obtained by the heat treatment of carbonaceous material. B. M. VENABLES.

**Liquid separator.** J. KUHN (B.P. [A] 277,842, 28.12.26, and [B] 338,349, 6.11.29).—The apparatus comprises a closed vessel in which is situated a sliding valve operated by a ball float. In order to permit easy machining of the valve seat, in (A) the seat is inclined so that it can be reached through the end cover, in (B) the cover is inclined and the seat horizontal. B. M. VENABLES.

**Rotary spraying separator.** F. H. LUENSE, Assr. to ROTOSPRAY MANUFG. Co. (U.S.P. 1,769,586, 1.7.30. Appl., 3.12.25).—The material is sprayed by centrifugally rotating shallow bowls against a fixed screening surface through which liquid passes; the thickened material is lifted and re-sprayed, by means of a hollow truncated cone rotating below the bowls, until it is sufficiently dry. B. M. VENABLES.

**Distillation of liquid mixtures.** J. V. GAUDET and A. ABRAMSON (B.P. 339,663, 8.8.29).—A distillation system is controlled by the b.p. of the distillate, a sample of which is condensed, led to an assay flask, and reboiled; a thermometric device in the neck of the flask regulates through a controller and valve the flow of reflux liquid in the fractionating tower. B. M. VENABLES.

**Condensing apparatus.** G. L. MACNEILL (U.S.P. 1,769,746, 1.7.30. Appl., 4.6.23).—A main surface condenser is provided with an auxiliary ejector-condenser which is operated by some of the main condensate pumped specially for the purpose, the vapour inlet to the auxiliary condenser being between the main condenser and the source of vapour. It is stated that the temperature of the condensate may be raised to nearer the theoretical as determined by the vacuum. B. M. VENABLES.

**Evaporation of liquids or solutions.** R. M. WINTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 339,657, 14.6.29).—The liquid when flowing as a film is evaporated by direct or indirect radiant heat from plate-like elements that are maintained incandescent by surface combustion or other means. When the heating is indirect, good heat transmission is attained either by allowing the liquid to boil or, if that is not permissible, by keeping the film so thin (not exceeding 3 mm.) that the motion is turbulent. B. M. VENABLES.

**Crystalliser.** W. G. HALL, Assr. to R. C. PITCAIRN (U.S.P. 1,769,779, 1.7.30. Appl., 31.7.29).—A suitable tank is provided with a horizontal helical stirrer completely submerged in the liquid and with another of similar pitch, rotating synchronously, partly submerged so that it continually exposes liquid to the atmosphere. B. M. VENABLES.

**Gas-washing apparatus.** E. VON MALTITZ, Assr. to H. A. BRASSERT & Co. (U.S.P. 1,767,628, 24.6.30. Appl., 7.2.27).—Crude gas is cooled and washed by passing through a vessel filled with a number of rotating plates which are continuously wetted by water. The component discs are placed between baffles so that the maximum contact of gas with liquid is obtained; separately mounted vertical or horizontal shafts may carry plates which rotate in opposite directions. R. H. GRIFFITH.

**Preparation of contact mass.** K. B. JOHN, C. W. COSLOW, and C. J. SCHWINDT, Assrs. to SELDEN Co. (U.S.P. 1,764,024, 17.6.30. Appl., 14.2.29).—Claim is made for a machine for making small briquettes from powdered catalysts, e.g., kieselguhr, moistened with catalytically active binders. A. R. POWELL.

**Treating [spent] fuller's earth etc. [from filtration or decolorising processes].** H. J. HARTLEY, Assr. to NICHOLS COPPER Co. (U.S.P. 1,768,465, 24.6.30. Appl., 23.2.28).—The organic matter is burned out by passing the material downwards through a multiple-hearth furnace up which is passed a current of air preheated by the hot gases from the furnace. A. R. POWELL.

**Classifying apparatus [for granular materials].** A. FRANCE (U.S.P. 1,785,679, 16.12.30. Appl., 8.11.27. Belg., 2.4.27).—See B.P. 301,812; B., 1929, 191.

**Separation of intermixed divided materials.** K. DAVIS, Assr. to PEALE-DAVIS Co. (U.S.P. 1,787,340, 30.12.30. Appl., 10.7.22).—See B.P. 223,280; B., 1924, 999.

**Filter press.** L. REVEL (U.S.P. 1,785,494, 16.12.30. Appl., 6.6.28. Fr., 14.6.27).—See B.P. 292,117; B., 1929, 627.

**Production of filter sieves.** A. PAHL (U.S.P. 1,786,361, 23.12.30. Appl., 28.1.27. Ger., 20.5.25).—See B.P. 281,879; B., 1928, 144.

**Distillation apparatus.** L. J. SIMON (U.S.P. 1,787,399, 30.12.30. Appl., 13.6.27. U.K., 19.7.26).—See B.P. 278,815; B., 1927, 946.

**Lining vessels for carrying out operations with liquids and the like.** F. H. BRAMWELL, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,787,101, 30.12.30. Appl., 10.1.30. U.K., 9.1.29).—See B.P. 331,838; B., 1930, 887.

**Refrigerating apparatus of the absorption type.** G. ROOS (B.P. 340,689 and 340,695, [A] 5.11.29, [B] 6.11.29).

**Continuously acting absorption machines for producing cold or heat.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 340,659, 17.10.29. Ger., 26.10.28).

**Centrifugal condensers [compressors].** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 339,720, 10.10.29. Ger., 10.10.28).

[Link-connected plate] machine for drying and/or pressing [flat] material. E. and E. SIEMPELKAMP (G. SIEMPELKAMP & Co.) (B.P. 340,660, 18.10.29).

Furnace (B.P. 337,490). Heating of furnaces etc. (B.P. 339,641). Washing of sand filters [containing oil sludge] (U.S.P. 1,769,475—6).—See II. Un-oxidisable alloy (U.S.P. 1,771,773). Heat-absorbing and -emitting surfaces (B.P. 339,722).—See X. Conducting chemical reactions (G.P. 457,563 and 458,756).—See XI. Filter plugs (B.P. 339,761).—See XVIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Geological aspects of the formation of coal.** C. S. Fox (Fuel, 1930, 9, 548—559).—The mode of occurrence and the general characteristics of Indian coals are described, and the following conclusions are drawn relating to the origin and formation of these coals. The original plant material, leaves, dead twigs and trunks, etc., was rapidly submerged in fresh or salt water, and finally settled down in the stagnant aseptic water of swamps or lagoons. The attack by fungi and micro-organisms, however active in the plant material before and during the shallow stages of immersion, is considered to have ceased as the maturing mass sank in deeper water or was covered by further plant debris. The main processes in the maturing of peat, and its subsequent conversion into primary coal, were of a chemical character and involved all the plant components, cellulose, hemicellulose, lignin, lignocellulose, and suberin. These substances were slowly converted into a gelatinous substance of the nature of doppelrite. The spores, sheaths of sporangia, walls of wood cells, etc., which were particularly resistant, remained embedded in the gelatinous mass. This mass gradually hardened, forming primary vitrain. The further changes which the coal substances underwent, and which gave rise to the different classes of coal ordinarily met with, were brought about by the effects of heat and pressure, and involved the removal of part of the volatile matter of the material. A. B. MANNING.

**History and present position of coal investigation by oxidation.** W. FUCHS (Fuel, 1930, 9, 581—586).—Previous work on this subject, particularly that of Fischer, Bone, and of their collaborators, is critically discussed, and our knowledge of the chemistry of coal derived therefrom is summarised. The brown coals, in their ulmin constituents, are regarded as cyclic hydroxyoxycarboxylic acids, and the bituminous coals as reduced hydroxycarboxylic anhydrides. By mild oxidation both the ulmic acids and the ulmins are converted into "dehydrulmic acids," which contain OH, CO<sub>2</sub>H, and CH<sub>2</sub>·CO groups, and have mol. wts. of 1000—1400; these differ from ulmic acids in that they are soluble in organic solvents. Stronger oxidation converts these primary oxidation products into water-soluble, ether-insoluble acids which on further oxidation yield benzenecarboxylic acids and phenol. A. B. MANNING.

**X-Ray study of vitrains.** C. MAHADEVAN (Indian J. Physics, 1930, 5, 525—541. Cf. B., 1929, 965; 1930, 697).—Vitrains of varying composition and different

geological ages, examined by X-ray diffraction methods, gave two haloes, the spacings for the inner intense and outer faint haloes being 3·37 and 2·12 for the older and 3·50 and 2·21 Å. for the newer coals, respectively. General scattering between the direct spot and the haloes was observed; the effect of moisture content and volatile matter is discussed, and the effect on the haloes of the removal of the two last-named was examined. The sizes of the diffracting particles, calculated from Laue's formula, were of the order of colloidal particles. The spacings for the peak intensity of the halo were the same for the residues as for the original samples. The α-, β-, and γ-compounds obtained by extraction with pyridine and benzene were examined, and the spacings and widths of haloes compared with those for untreated coals. It is concluded that mineral matter present is in a colloidal state. N. M. BLIGH.

**Geipert [coal assay] apparatus.** F. SCHUSTER (Brennstoff-Chem., 1930, 11, 471—473; cf. B., 1926, 938).—From the results of analyses of the gas and coke produced in the Geipert apparatus it is concluded that (i) the rate of heating therein is greater than in the full-scale retorts; (ii) the gas escapes more rapidly therefrom, as is shown by its high content of unsaturated hydrocarbons (6%); (iii) the time of heating is longer and the final temperature higher than corresponds with large-scale practice, the coke produced having low hydrogen, sulphur, nitrogen, and oxygen contents. The apparatus, nevertheless, is useful for determining the thermal yield of gas, since the effects of the above-mentioned differences on the result to a great extent cancel one another. A. B. MANNING.

**Apparatus for determining the tendency of coal and other materials to self-ignition.** D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 690—694).—Analytical data are given showing the variation of the volatile and ash contents of coal according to the degree of fineness of the sample. The difficulties of accurate sampling are noted. Earlier types of apparatus for determining the lowest temperature at which self-ignition of coal takes place are criticised, and a new apparatus is described (cf. B., 1930, 847). H. F. GILLBE.

**Microbiological aspect of peat formation.** A. C. THAYSEN (Fuel, 1930, 9, 560—563; cf. Waksman, B., 1930, 933).—A study of the behaviour of the known types of cellulose-decomposing micro-organisms proves that the activity of these organisms in peat bogs is limited to the surface layers, that it decreases in intensity as soon as the debris has become waterlogged, and that, in acid peats at least, it ceases altogether long before the cellulose of the debris has been eliminated. A. B. MANNING.

**Evaluation of African torbanites.** P. NIEMANN (Brennstoff-Chem., 1930, 11, 469—471).—Torbanites from the Transvaal (moisture 1·0%; ash 23·4%) and Angola (moisture 4·0%; ash 43·5%) have been distilled in a current of superheated steam and the products examined. The yields of tar amounted to 35·5% and 24·0%, and of coke to 42·2% and 57·3%, respectively. The tars were distilled to coke, yielding (%): light oil (to 150°) 9·6, 0·3; heavy benzine 8·0, 3·3; fuel oil 51·1, 45·4; machine oil 11·0, 31·5;



paraffin wax 3.25, nil; and coke 12.6, 11.0%, respectively. The oils were of good quality. By extraction of the Transvaal tar with alcohol a creosote (10.5%) of high fungicidal value was obtained. The tar cokes were particularly suitable for the manufacture of electrodes. The torbanite cokes were difficult to ignite and could be utilised only in admixture with a good-quality fuel.

A. B. MANNING.

**Isolation of the humic acids.** G. THIESSEN and C. J. ENGELDER (Ind. Eng. Chem., 1930, 22, 1131—1133).—From a log of white cedar (*Thuja occidentalis*), which had decayed under conditions similar to those necessary for the formation of peat, after removal of the resins with acetone and of the lignin fraction with chlorine dioxide solution, a mixture of humic acids has been obtained by extraction with 4% sodium hydroxide solution. The acids, recovered from this extract by precipitation with dilute hydrochloric acid, may be separated into two fractions: humic acid (C 58.8%, H 5.1%), insoluble, and hymatomelanic acid (C 59.0%, H 5.5%), soluble in hot alcohol. A portion of the latter acid is soluble in acetone and ebulliscope determinations indicate *M* 800 for this fraction. Similar determinations made on nitro- and bromo-humic acid indicate *M* 1040 and 940, respectively. Solutions of humic acid in ammonia are almost completely transparent to red and yellow light, but the absorption increases with diminution in wave-length to almost complete opacity in the blue and violet regions of the spectrum. Absorption curves of a similar nature are recorded for solutions of nitro- and bromo-humic acids in acetone.

T. H. MORTON.

**Testing of coking coals.** W. MELZER (Arch. Eisenhüttenw., 1930—1, 4, 169—175; Stahl u. Eisen, 1930, 50, 1643—1644).—Comparative tests with numerous coals of the various methods which have been proposed for testing the coking qualities of coal showed that reliable results are obtained only by the method of Damm (cf. B., 1929, 382) and its modifications in which softening point is determined and the rate of evolution of the various gases measured during the coking process.

A. R. POWELL.

**Propagation of combustion in powdered coal.** H. E. NEWALL and F. S. SINNATT (Safety in Mines Res. Bd., 1930, Paper No. 63, 58 pp.).—A moulded column of the finely-powdered (200-mesh) coal was placed on a silica plate, one end of the "train" was heated with a small Bunsen burner flame, and the time required for the zone of combustion to travel 10 cm. was then determined. Preoxidation of the coal decreased the rate of propagation of the zone. Preheating of the coal in nitrogen at 100—300° increased the time of propagation; preheating at 300—400° decreased the time, but prolonged heating in nitrogen at these temperatures effected an increase in the time of combustion. An increase in the oxygen concentration above that of normal air decreased, and a reduction of the oxygen content increased, the time of propagation; no coal would burn in atmospheres containing less than 14% of oxygen. Additions of small amounts of potassium, sodium, and iron salts to the coal decreased the time, whilst additions of chlorides, hydrochloric, and sulphuric acids increased

the time of propagation. The temperature attained in the centre of a cone of coal was controlled by varying the rate of air supply, and the amount of hydrogen cyanide produced at various temperatures was determined; the maximum amount obtained was 0.1% by wt. of the coal, and none was found at combustion temperatures below 300°. The composition of the oxidation products produced from any coal is constant and is independent of the rate of propagation of the zone of combustion through the coal.

C. B. MARSON.

**Agglutination of coal and activation of its surface during coke formation, considered as two complementary phenomena.** W. SWIETOSLAWSKI (Fuel, 1930, 9, 564—566).—The coke produced by carbonising a non-caking coal under suitable conditions exhibits a surface of high activity, i.e., of high decolorising and adsorptive capacity. A highly developed surface, however, is disadvantageous in a non-caking coal or a semi-coke when it is desired to produce a strong coke from blends of these with a caking coal, owing to the difficulty of thoroughly impregnating such a surface with the plastic coal. Such impregnation may be accomplished by briquetting the mixture when it has attained a temperature within the plastic range of the caking constituent. Similar considerations apply to the briquetting and carbonising of mixtures of semi-coke and pitch.

A. B. MANNING.

**Coking a banded bituminous coal: the part played by each band.** C. P. FINN (Gas J., 1930, 192, 965—968).—Samples of vitrain, clarain, durain, and fusain obtained from the Parkgate seam in South Yorkshire have been examined to determine their influences on coke quality. Examination of the coke buttons obtained in the volatile matter test shows that vitrain gives the most swollen button, that obtained from durain is dull and shrunken, whilst fusain is non-caking. When the various banded ingredients were heated at 1°/min. in a laboratory coking test, vitrain and clarain gave the same degrees of contraction and swelling, durain contracted without swelling, and fusain was non-contracting and non-swelling. Quantities of clarain, durain, vitrain, and blends of these substances with each other and with fusain were coked in boxes, placed on the sole of a full-scale oven, and the resulting cokes tested for size and resistance to shatter; it was found that different samples of clarain, which forms the largest percentage of the banded ingredients present in a coking slack, varied considerably in their coking properties, but, in general, yielded cokes that were much fissured and of low shatter index. The presence of vitrain in a coking slack is proportionately more valuable than that of clarain, whilst durain, although so different in its coking properties from vitrain and clarain, exerts little effect. Fusain, although itself non-caking, when added uniformly in small, carefully regulated quantities, was found to effect marked improvements in the coke; an increase in the amount of "middlings" did not have any beneficial effect, and there were indications that their removal might be an advantage. Methods for increasing the amount of vitrain in a coking slack and for collecting and adding fusain in the required proportion are described.

C. B. MARSON.

**Action of catalysts on the course and products of the distillation of pine wood.** P. KUPFERBERG (Bull. Inst. Pin, 1930, 113—120, 137—144, 159—168, 185—191, 207—208).—The distillation curve for pine wood without catalyst shows a break about 300° corresponding with an exothermic decomposition at that temperature. In presence of catalysts this break disappears and with acid catalysts, especially phosphoric acid, is replaced by breaks at 180—200° (cellulose decomposition) and 330—350° (lignin decomposition). Metal chlorides (aluminium, iron, zinc, manganese, calcium, magnesium) postpone the decomposition and extend the distillation curve, an effect which is even more noticeable with alkaline catalysts (sodium carbonate, sodium hydroxide, calcium hydroxide). All the catalysts increase the yield of charcoal at the expense of the pyro-ligneous liquor and tar; the gas yield is increased by alkaline catalysts, and decreased by metal chlorides and especially by acids. In the pyro-ligneous liquor the amount of crude methyl alcohol is increased about five-fold by the use of catalysts, but acetic acid remains about the same; hydrochloric acid or sodium carbonate doubles the yield of furfuraldehyde, whilst that of acetone is similarly increased by sulphuric acid or lime. Much more furfuraldehyde is produced from the cellulose decomposition than from that of lignin. The charcoal of highest calorific value is obtained by the use of magnesium chloride, and all the catalysts increase its content both in hydrogen and in oxygen. The most important observation recorded is the effect of phosphoric acid, which on the one hand increases greatly the yield of charcoal, and on the other enables the distillation to proceed in two stages; at the end of the first stage (200°) a notable increase in calorific value is found, and the product may replace liquid fuels in gas producers. C. HOLLINS.

**Theory and practice of the biological method of rendering coal gas non-poisonous.** F. FISCHER, R. LIESKE, and K. WINZER (Brennstoff-Chem., 1930, 11, 452—455. Cf. Lieske and Hofmann, B., 1930, 803).—Various chemical reactions which can be brought about by bacterial action are briefly discussed. It has been found possible to free coal gas from carbon monoxide by passing it slowly through a tube packed with putrefied effluent sludge. The principal reactions which occur are:  $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$ ,  $\text{CO}_2 + 4\text{H}_2 = \text{CH}_4 + 2\text{H}_2\text{O}$ , and  $\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{H}_2$ , though doubtless a number of other reactions also take place. A reaction vessel of 100 litres capacity can free 200 litres of gas per day from carbon monoxide at 15—25°. The optimal temperature lies somewhat higher (24—32°). The activity of the bacteria falls off rapidly as the temperature is raised above 32° and ceases entirely at about 40°; at still higher temperatures, however, the reaction again sets in and exhibits a maximum at about 65°. The reaction at the higher temperature is due to thermophilic bacteria which appear to exist in most samples of effluent sludge. Attempts are being made to increase the rate of reaction. A. B. MANNING.

**Recent developments in plant for the mechanical purification of coal gas.** A. THAU (Brennstoff-Chem., 1930, 11, 416—418, 435—438, 455—459).—The methods of separating suspended solid and liquid particles from

gases are discussed, and the following plants are described in some detail: (a) the Smith tar separator, (b) the Bartling separator (cf. B.P. 282,840; B., 1929, 191), and (c) the Feld washer. A. B. MANNING.

**Humidity control in dry purification [of coal gas].** N. S. LOGAN (Gas J., 1930, 192, 970).—The important factors when revivification of the oxide in the purifier boxes is carried out *in situ* are discussed. Fresh oxide containing 35% of moisture was used in the purifiers, and water was introduced into the boxes, during revivification, to maintain this amount of moisture. C. B. MARSON.

**Interaction of methane with carbon dioxide and steam in the electric discharge.** K. PETERS and A. PRANSCHKE (Brennstoff-Chem., 1930, 11, 473—476).—Mixtures of methane or coke-oven gas with carbon dioxide or steam have been submitted to the action of an electric discharge under reduced pressure by the method described previously (B., 1930, 848). With high current densities the methane was completely converted into carbon monoxide:  $\text{CH}_4 + \text{CO}_2 = 2\text{CO} + 2\text{H}_2$ ;  $\text{CH}_4 + \text{H}_2\text{O} = \text{CO} + 3\text{H}_2$ . No deposition of carbon occurred. With medium current densities the greater part of the methane was converted into acetylene. The composition of the end gas could be varied within wide limits by varying the initial gas mixture and the current density. A. B. MANNING.

**Ignition of firedamp by the heat of impact of hand picks against rocks.** M. J. BURGESS and R. V. WHEELER (Safety in Mines Res. Bd., 1930, Paper No. 62, 21 pp.).—Experiments are described which show that firedamp can sometimes be ignited when a hand pick is struck against certain sandstones, especially those of a quartzitic or micaceous nature and which are fairly readily crushed by the impact of the pick point. No ignitions were obtained with iron stones and pyrites. C. B. MARSON.

**Properties of high-temperature gas from Estonian shale.** A. PUKSOV (Festschr. Jub. Ver. Estn. Chem., 1929, 76—80; Chem. Zentr., 1930, ii, 847).—The shale (1 g.) afforded gas (404 c.c.) of the following composition:  $\text{CO}_2$  8.1,  $\text{H}_2\text{S}$  0.5,  $\text{C}_2\text{H}_6$  15.3, O 1.9, CO 14.7, H 25.3,  $\text{C}_2\text{H}_4$  0.3,  $\text{CH}_4$  25.7, N 8.3%. A. A. ELDRIDGE.

**Apparatus for the exact analysis of gas mixtures in amounts down to 3—4 c.c.** A. SCHMIDT (Gas- u. Wasserfach, 1930, 73, 1137—1144).—The apparatus comprises a water-jacketed gas burette, a manometer, a levelling vessel, an absorption and combustion pipette with separate levelling vessel, and an oleum pipette. The quantity of gas before and after each operation is determined manometrically, i.e., by measuring the pressure when the gas is brought to a definite volume. The gas burette is divided into five bulbs, three being of 25 c.c. capacity each and two of 3—4 c.c., so that the volume to which the pressure measurements are referred can be varied according to the amount of gas available for analysis. The small bulbs are used for micro-analyses. The manometer is provided with a scale divided in mm. The absorption and combustion pipettes are connected to the gas burette through three-way taps in such a way as to leave no dead space.

The combustion pipette, which is provided with a small platinum spiral capable of being heated electrically, serves also for the absorption of carbon dioxide, oxygen, and carbon monoxide, the requisite reagents being introduced through the three-way tap. The oleum pipette is used for determining unsaturated hydrocarbons. With the exception of the determination of methane in the presence of its higher homologues the apparatus is applicable to the analysis of all gases occurring in ordinary practice. The technique is described in full. When suitable precautions are taken, the accuracy of an analysis amounts to 0.05% when only 3–5 c.c. of gas are available, and to 0.01% with 30–80 c.c.

A. B. MANNING.

**Titration of brown-coal-tar creosote with naphthalenediazonium chloride.** R. VON WALTHER and K. LACHMANN (Braunkohlenarch., 1930, No. 26, 1–11; Chem. Zentr., 1930, ii, 663–664).—The method is inapplicable, since various phenols present in the tar give values ranging from 36 to 103% of the theoretical.

A. A. ELDRIDGE.

**Treatment of oil sands.** K. KELLERMANN and A. VOGT (Petroleum, 1930, 26, 1141–1148, 1157–1164).—The possibility of completely separating the oil from oil sands by displacement with water or a suitable aqueous solution has been investigated. Although theoretical considerations, based on observation of the angles of contact of the oils and water respectively with sand, indicate that it should be possible to displace the oil by water, in practice the process fails, owing apparently to the adsorbed layer of oil on the sand particles effectively preventing any contact between the water and the sand. Nor was it possible to overcome the resistance of the oil layer by the use of heavy solutions, *e.g.*, concentrated calcium chloride. The application of an electric field in the presence of dilute solutions of sodium hydroxide etc. appeared to bring about some separation, but caused undue oxidation of the oil. The bromine, iodine, and oxygen values of the oils and their fractions were determined, and the surface tensions between the oils and some aqueous solutions, especially those of sodium carbonate, were measured. The lowering of the interfacial tension on the addition of sodium carbonate to water was shown experimentally to be associated with an adsorption of the salt on the interface. The separation of the oil from the sand was eventually effected by passing a solution of sodium bicarbonate through the material, thereby combining the effect of the lowered interfacial tension with the mechanical effect produced by the evolution of gas.

A. B. MANNING.

**Cracking of Californian kerosene and furnace oil at relatively low pressures.** G. EGLOFF (Petroleum, 1930, 26, 1263–1264).—Kerosene and furnace oil have been cracked at pressures of 14–21 atm. to give 53–60% of motor spirit of high anti-knock value. The oils were treated by a simultaneous cracking and coking process under pressure and the resulting distillate washed by the plumbite-sulphuric acid method and redistilled, the resultant spirit being washed with dilute caustic soda solution to eliminate traces of hydrogen sulphide. The motor spirit obtained was bright and of low gum and sulphur contents (1% or less). The proportions of unsaturated, aromatic, naphthene, and

paraffin hydrocarbons in the product from kerosene were 17.3%, 18.4%, 20%, and 44.3%, respectively. The residue from the cracking process is suitable for Diesel and heavy engine oil. The uncondensable gas has a heating value of about 1200 B.Th.U. per cub. ft., and may be used directly as fuel and for enriching water-gas or producer gas, or it may be cracked at high temperatures in the presence of steam in the producer, to give a gas of about 550 B.Th.U. per cub. ft. heating value. The coke obtained is practically ash- and sulphur-free, and suitable for the making of briquettes or carbon electrodes; its cellular structure renders it of great value for water-gas manufacture.

H. E. BLAYDEN.

**Isolation of the isomerides of hexane from petroleum.** J. H. BRUUN and M. M. HICKS-BRUUN (Bur. Stand. J. Res., 1930, 5, 933–942).—Fractional distillation of an Oklahoma crude petroleum concentrated the hexanes in a series of constant-boiling mixtures containing cyclic compounds. After addition of ethyl alcohol to these fractions, refractionation yields constant-boiling mixtures of alcohol and the hexanes, all of which boil below 58.7°, whereas similar mixtures with the cyclic compounds present all boil above 64°; the alcohol is removed by a water wash, and the hexanes are purified by equilibrium melting. The following isomerides were separated in this way:  $\beta\gamma$ -dimethylbutane,  $\beta$ - and  $\gamma$ -methylpentanes, and *n*-hexane. The f.p. of air-saturated  $\beta$ -methylpentane is  $-143^\circ$ , that of  $\gamma$ -methylpentane  $-118^\circ$ , and that of the eutectic of  $\beta$ -methylpentane and carbon dioxide  $-153^\circ$ . No evidence of the presence of  $\beta\beta$ -dimethylbutane in the petroleum examined could be obtained.

A. R. POWELL.

**Lubricating oils.** J. TAUSZ and A. STAAB (Petroleum, 1930, 26, 1117–1124, 1129–1140).—The change in viscosity produced when lubricating oils are diluted with a solvent, *e.g.*, toluene, has been compared with the variation in viscosity with rise of temperature. The former gives a better estimate of the cohesive forces within the oil, and therefore of its lubricating value, which is higher the less the change in viscosity produced by dilution. The change is expressed by the value of  $x$  in the equation  $\eta_1^x = \eta_2$ , where  $\eta_1$  is the viscosity of the original oil, and  $\eta_2$  that of the diluted oil; vegetable oils, on dilution with 20% of toluene, give values about 0.66, whilst mineral oils give 0.50–0.53. The mol. wts. of a number of lubricating oils have been determined, both ebullioscopically and cryoscopically, in different solvents; in general, the observed mol. wts. of the mineral oils rise with increasing concentration of the oil in the solvent, whilst those of the vegetable oils fall. On mixing a typical mineral oil ("Mobiloil") with benzene a marked increase in volume is observed; on mixing castor oil with benzene a small decrease is observed when the proportion of benzene is less than 50%, and a small increase with higher benzene concentrations.

A. B. MANNING.

**Formation of gum in motor fuels.** R. BRUNSCHWIG and L. JACQUÉ (Compt. rend., 1930, 191, 1066–1068).—The combined and individual effects of exposure to light and air on the colour and auto-oxidation ("gumming") of various samples of commercial benzene

motor spirits (containing unsaturated hydrocarbons) have been investigated by methods described previously (B., 1929, 931). Contrary to accepted ideas, the colour developed on ageing bears no relationship to the amount of gum formed, a sample (originally containing 1 mg. of residue/100 c.c.) which remained colourless, giving 47–147 mg. of residue/100 c.c. (according to the conditions of ageing), whilst one which became yellow even in the dark gave only 15–20 mg./100 c.c. Ageing in the presence of either light or air alone results in the formation of relatively small amounts of residue (light producing the larger effect), but the combined action of these two factors gives rise to a large quantity of residue, a sample which contained originally only 3 mg./100 c.c. containing 430 mg./100 c.c. after ageing under these conditions.

J. W. BAKER.

#### Refining of motor benzol by means of silica gel.

R. C. GEDDES (Gas World, 1931, 94, Coking Sect., 17–20).—For removal of gum-forming substances from crude benzol, simultaneous treatment with dilute sulphuric acid and silica gel is preferred to that with concentrated acid alone. The losses are greatly diminished as the simple olefine hydrocarbons are hardly attacked; the refined product can be regained in 93% yield. The process appears to be catalytic, and is carried out at about 150° under sufficient pressure to maintain a liquid phase.

R. H. GRIFFITH.

**Purification of used compressor oil.** L. PIATTI (Petroleum, 1930, 26; Motorenbezt., 3, 7).—The lubricating oil which is used in the compressors of systems for the long-distance transmission of coal gas, and which has become contaminated with constituents dissolved from the gas, has been satisfactorily purified by a simple steam-distillation at 120°.

A. B. MANNING.

**Extinction of benzine-air explosions by carbon tetrachloride.** A. H. BELINFANTE (Chem. Weekblad, 1931, 28, 2–19).—Ignition and explosion limits, and extinction by the vapour of an inert liquid, are considered in detail from the theoretical aspect. An account is given of the method of determining from the explosion and ignition limits of the single substances and the partial pressures of the components the behaviour of the vapour emitted by a binary liquid mixture when admixed with air. The explosion limits of mixtures of air, carbon tetrachloride, and two types of benzine, viz., of *d* 0.7942 and 0.651, have been determined by sparking mixtures of known volumes of air saturated with benzine vapour at a certain temperature with either air or air saturated with carbon tetrachloride vapour or with a mixture of carbon tetrachloride and benzine vapours. Owing to the relatively high b.p. of the tetrachloride a somewhat high concentration is necessary to inhibit explosion, and both within and without the explosion limits a fog of hydrochloric acid is formed on passing a spark. The vapour pressure-temperature curves of benzenes ranging from *d* 0.7942 to 0.651 and b.p. 50–202° to 32–67° have been determined, and the observed flash-points of a number of the specimens of benzene are compared with those obtained by other observers.

H. F. GILLBE.

#### Effect of anti-knock materials on the speed of

flame in a closed tube. Y. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 296–299 B).—The effects of lead tetramethyl, tin tetramethyl, and ethyl selenide on the speeds of flames in mixtures of air and hydrocarbon vapour in horizontal closed tubes has been investigated, the hydrocarbon used being the same as that described previously (A., 1930, 1255). Tin tetramethyl and ethyl selenide in concentrations up to 0.1 mol.-% diminish the speed, which increases again at higher concentrations. Lead tetramethyl reduces the velocity in concentrations up to 0.1 mol.-%. (Cf. Egerton and Gates, B., 1927, 738.)

C. W. GIBBY.

**Iowa coal problem.** H. L. OLIN. **Storage.** H. L. OLIN and C. E. SCOTT. **Coking.** H. L. OLIN, F. V. JOHNSON, JUN., and R. C. KINNE. **Washing.** H. L. OLIN, C. BARRETT, and H. D. ALLEN. **Weathering.** H. L. OLIN, J. D. WADDELL, and J. N. AMBROSE (Iowa Geol. Survey, Tech. Paper, 1930, No. 2).

**Heat transfer from gas stream to broken solids.** FURNAS.—See I. **Pyrolysis of methane.** WHEELER and WOOD. **Reduction and hydrogenation of phenols.** TROPSCH.—See III. **Ammonia catalysts.** GLUUD and SCHÖNFELDER.—See VII. **"Bitural" for roads.** BROADHEAD.—See IX. **Ammonia solution as fertiliser.** BIMSCHAS.—See XVI. **Carbon in water purification.** BEHRMAN and CRANE; NORCOM and DODD; BAYLIS.—See XXIII.

#### PATENTS.

**Concentration of minerals including coal.** J. F. C. FRIEND (B.P. 337,458, 2.8.29).—A layer of the material, in suitably subdivided form, e.g., below  $\frac{1}{2}$  in., is caused to travel over a horizontal screen in a series of intermittent advance movements, and is subjected to an alternating upward and downward movement of air through the material, whereby it is stratified into layers of different components. The movement of air through the layer is timed to occur between the horizontal advance movements. The stratified layers are then separated from one another.

A. B. MANNING.

**Preparation of oil and coal amalgam.** TRENT PROCESS CORP. (B.P. 337,920, 3.10.29. U.S., 20.10.28. Cf. U.S.P. 1,512,427; B., 1925, 62; also B.P. 262,302; B., 1927, 100).—The finely-divided coal is first treated with a light oil and water to form an amalgam and to facilitate the separation of the ash, and a heavy oil is then incorporated with the amalgam, which may then be briquetted and baked.

A. B. MANNING.

**Apparatus for treating coal.** F. B. DEHN. From MAUREL INVESTMENT CORP. (B.P. 337,600, 31.10.29).—The coal is briquetted with a waterproof asphalt or other binder, passed through a distilling zone in which steam and other volatile products are removed, then through a hardening zone, and finally through a retorting zone. The briquettes are carried through the first two zones on an endless conveyor; in the distilling chamber they are heated indirectly by the combustion gases from a furnace, and in the hardening chamber both directly and indirectly by these gases; the hardened briquettes are discharged from the conveyor into the retort, wherein they are further heated by passing the hot combustion gases through them. The conveyor passes into and

from the apparatus and from one chamber thereof to the next through water-seals. A. B. MANNING.

**Drying of peat fuels and the like.** D. W. BERLIN (B.P. 340,156, 22.1.30. Swed., 6.2.29).—Wet-carbonised peat or a similar low-grade fuel is subjected to a two-stage centrifugal separation, first in a vessel with water-tight walls, and then in one with porous walls. The product from the first stage may be suitably subdivided before being dried in the second stage by heating.

R. H. GRIFFITH.

**Manufacture of firelighters.** BRIQUETTING & CARBONISING SYND., LTD., and T. A. GOSKAR (B.P. 339,985, 17.9.29).—Finely-ground coke, anthracite, or other smokeless fuel is mixed with pulverised peat and briquetted, preferably with the addition of sodium silicate; sawdust, waste paper, and similar combustible substances may also be added. The finished pieces are coated with wax or resin.

R. H. GRIFFITH.

**Treatment of activated charcoals.** C. H. LANDER, F. S. SINNATT, J. G. KING, and W. E. BAKES (B.P. 337,348, 23.7.29).—Charcoal which has been used for absorbing substances, particularly sulphur compounds, is reactivated by being subjected to the action of superheated steam containing small proportions of air and a volatile basic substance such as ammonia, aniline, pyridine, etc.

A. B. MANNING.

**Regeneration of activated charcoal used for dechlorinating drinking water.** H. PICK (B.P. 316,269, 26.7.29. Czechoslov., 26.7.28. Cf. Adler, B., 1929, 700).—The exhausted charcoal is treated with a hot alkaline solution, e.g., dilute sodium hydroxide, with stirring, and is then washed.

A. B. MANNING.

**Production of lampblack.** A. J. PARIS, JUN. (U.S.P. 1,756,877, 29.4.30. Appl., 4.10.21).—Hydrocarbon gases are decomposed to give high yields of lampblack by bubbling through a molten mass of hot metal such as lead. The lampblack floats on the surface of the liquid and is removed by a stream of gas.

R. H. GRIFFITH.

**Purification of graphites.** E. RABETRANO (B.P. 337,738, 25.6.29. Fr., 9.11.28).—The finely-ground material is added to water containing a colloid adapted to retard the rate of settling of the particles. After separation of the denser impurities by gravity the fraction containing the bulk of the graphite in suspension is subjected to further separation by selective electro-osmotic action (or cataphoresis) in suitable apparatus, which is described.

A. B. MANNING.

**Coke ovens.** N.V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & Co. G.M.B.H. (B.P. 337,667, 18.12.29. Ger., 4.5.29).—A coke oven with adjacent upflow and downflow heating flues, which are in open connexion above and below so that a certain amount of the downflow gas is returned into circulation in the incandescent upflow flues, has the brick slides which control the gas and air supplies to the upflow flues arranged below the level of the lower openings connecting the upflow and downflow flues. The cross-section of these openings can be varied by brick-slides operated from the chamber below the regenerators.

A. B. MANNING.

**Cylindrical oven for distillation of coal or lignite at a low or high temperature.** H. HARDY (B.P. 338,047, 31.12.29. Belg., 3.1.29).—The oven comprises an outer rotatable drum, subdivided by transverse partitions into series of combustion, distillation, and suction chambers, and a hollow, fixed, central shaft divided longitudinally into three conduits for the admission of gas and air for heating and for the evacuation of the combustion products, respectively. Each distillation chamber is divided into wedge-shaped compartments, into which fit moulds charged with the coal to be distilled. As the drum slowly rotates, the temperature of each charge of coal rises as the corresponding compartment approaches the section of the combustion chamber in which the burners are situated. The volatile products of distillation pass into the suction chambers, which are also subdivided into compartments so that the products evolved at different stages of the heating may be withdrawn separately; for this purpose the corresponding compartments of the different suction chambers are connected together in axial rows.

A. B. MANNING.

**Coal-carbonising apparatus for extraction of bituminous materials.** K. BERGFELD (U.S.P. 1,756,969, 6.5.30. Appl., 7.3.27. Ger., 30.11.23).—Coal to be carbonised is packed in a vertical vessel round a central hollow pier in which combustion of heating gases takes place, and from which hot waste gases pass through the coal. The volume of circulating gas can be increased by addition of some of the distillation gases, which are preheated in an annular space surrounding the primary combustion zone.

R. H. GRIFFITH.

**[Heating of] furnaces, carbonising chambers, retorts, etc.** WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 339,641, 6.8.29).—The gas and air required for heating coke ovens etc. are passed under pressure through a multitubular recuperator, which ends in a nozzle; from this nozzle the combustion products ascend centrally to the top of a vertical heating flue. At this point their direction of flow is necessarily reversed and they descend, along the walls of the carbonising chamber, to escape by way of the outer part of the recuperator. Great flexibility is attained by varying the nozzle design or dimensions of the flue, and the recuperator is constructed so that it can easily be withdrawn from the setting.

R. H. GRIFFITH.

**Recovering lost heat in coke ovens and like thermic apparatus.** H. E. G. J. J. HELIN (B.P. 310,756, 29.4.29. Belg., 28.4.28).—In coke ovens, furnaces, or similar apparatus using regenerators or recuperators for heating the air for combustion, an additional air preheater is provided heated either by a source of heat independent of the apparatus, or, in the case of a coke oven, by the distillation gases. The residual heat in the combustion products leaving the regenerators is utilised for the production of steam in a separate waste-heat recovery plant.

A. B. MANNING.

**Distilling and gasifying solid fuels.** METALLGES. A.-G. (B.P. 337,721, 24.4.30. Ger., 2.5.29).—The fuel travels by gravitation down a vertical shaft, passing successively through a drying, a distilling, and a cooling

zone, these processes being effected by the circulation of suitably preheated gases through the fuel. A part of the circulating gases is passed through the cooling zone and thence through the upper part of the distillation zone. Distillation is effected by passing highly heated gases, principally steam, to which some oxygen may be added if desired, through the intermediate zone. These gases are heated in a chamber through which refractory heating bodies are continuously circulated; the upper part of the chamber is traversed by a current of hot combustion gases, which are subsequently utilised in drying the fuel, and the lower part by the gases which are to be heated. The temperature of the gases effecting distillation may be sufficiently high to bring about interaction between the fuel and the steam with the production of some water-gas.

A. B. MANNING.

#### Distillation of bituminous shales, cannel coals, torbanites, and similar carbonaceous substances.

F. ESLING (B.P. 339,963, 12.9.29).—Shales or other substances rich in sulphur are mixed with a petroleum oil and, if necessary, with a vegetable oil also, before carbonisation or distillation. If only comparatively low temperatures are employed, the sulphur remains largely in the retort or still in combination with asphaltic compounds, and the high-boiling residue can be used for road dressing.

R. H. GRIFFITH.

**Making carbonaceous material.** A. OBERLE (U.S.P. 1,763,063, 10.6.30. Appl., 18.8.24).—Lamp-black, petroleum coke, or similar material is prepared for use in moulding (as electrodes etc.) by grinding it to a fine powder and injecting this with superheated steam into a heated expansion chamber. Heavy particles first separate by gravity and volatile impurities are then carried off. These vapours and gases may be used as a fresh source of carbon black.

R. H. GRIFFITH.

**Coking of bituminous material.** AMER. TAR PRODUCTS Co., Assees. of E. O. RHODES (B.P. 337,800, 9.7.29. U.S., 9.2.29).—Pitch or similar material is coked in externally heated retort ovens of the by-product type, into which it is introduced in the liquid condition together with an inert gas, *e.g.*, steam. The material is sprayed into the oven at such a rate that the charge gradually accumulates without there being any quantity of liquid material in the oven at any time.

A. B. MANNING.

**Destructive hydrogenation of carbonaceous materials.** STANDARD OIL DEVELOPMENT Co., Assees. of J. M. JENNINGS (B.P. 337,671, 20.12.29. U.S., 21.12.28).—Heavy fluid asphalt-containing materials are hydrogenated by maintaining the material at the decomposition temperature, *e.g.*, 420–520°, in the presence of hydrogen under pressures in excess of 25 atm., and in contact with a solid catalyst immune to sulphur poisoning, the latter being present in such amount that the ratio asphalt:catalyst is held below 1.5, and preferably below 0.5. The volatile products are removed as they are formed and led to condensers, fresh material being supplied to the reaction chamber as required. Fresh catalyst is added from time to time by diverting the flow of fresh material before it enters the reaction chamber through a vessel containing the catalyst.

A. B. MANNING.

**Destructive hydrogenation of carbonaceous materials.** W. R. TATE, H. P. STEPHENSON, J. F. LEHMANN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 339,479, 29.11.29).—An apparatus is employed, for treatment of liquid hydrocarbons etc. with hydrogen under pressure, in which the level of liquid in a jacketing vessel is controlled by an external pneumatic indicator. Hydrogen for reaction bubbles at a controlled rate through oil which completely fills an inner vessel and which overflows from the top into the jacketing chamber.

R. H. GRIFFITH.

**Destructive hydrogenation of carbonaceous material.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 339,875, 13.2.30. Holl., 11.3.29).—The catalyst for treatment of carbonaceous material with hydrogen under pressure consists of a simultaneously precipitated mixture of iron and aluminium oxides  $\text{Fe}_2\text{O}_3 : \text{Al}_2\text{O}_3 = 5 : 1$  (approx.).

R. H. GRIFFITH.

**Treatment of solid and fluid carbonaceous materials.** F. UHDE (B.P. 339,317, 5.9.29. Ger., 3.10.28).—Destructive hydrogenation of substances such as coal, petroleum, tar, etc. is achieved by heating at about 200 atm. pressure to above 400° in the presence of finely-divided iron and steam. The nascent hydrogen produced serves for the conversion of heavy hydrocarbons into those of lower b.p. Any iron oxide remaining after the process is completed can be reduced for further use. Catalysts aiding hydrogenation may be added, or other substances (*e.g.*, alkalis) may be dissolved in the water.

R. H. GRIFFITH.

**Separation of oils, tars, and the like from solid constituents.** I. G. FARBENIND. A.-G. (B.P. 312,228, 1.5.29. Ger., 22.5.28).—The oils obtained by the destructive hydrogenation of coal etc. are separated from the solid residues by adding sawdust, brown coal dust, or ashes to the product, which may also be diluted with a suitable solvent, and centrifuging or filtering the mixture. The flocculating action of the sawdust etc. on the suspended solids greatly reduces the time required for filtration.

A. B. MANNING.

**Separation of oil from solid carbonaceous material.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,681, 13.9.29).—Mixtures of oil with solid carbonaceous material, such as products from destructive hydrogenation, are filtered at raised temperature through either a layer of granulated substance or a number of superimposed wire sieves. The time of passage through the filter is reduced by applying pressure or suction to opposite sides of the effective layer. The residue may be treated by distillation, by extraction with a low-boiling solvent, or with an aqueous liquid containing an emulsifying agent.

R. H. GRIFFITH.

**Manufacture of hydrogen or gases containing hydrogen from methane and similar hydrocarbons or gases containing these hydrocarbons.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 340,050, 3.10.29).—A water-gas generator with deep fuel bed is filled with coke or with a refractory material and is supplied alternately with streams of air and of a gas rich in hydrocarbons. The latter is decomposed, and the resulting deposit of carbon is consumed during



the following blow period in which the vessel is again raised to the working temperature. The air is preheated by passage through a regenerator, and enters the generator at several different levels; the hydrocarbon gases are also preheated in a second regenerator or the system may be arranged so that a single recuperator serves two units in turn. R. H. GRIFFITH.

**Simultaneously producing light hydrocarbon motor fuel oil and carburetted water-gas.** F. A. HOWARD, ASST. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,767,455, 24.6.30. Appl., 5.11.23).—By selection of suitable conditions in the carburettor and superheater of an ordinary carburetted water-gas plant, appreciable amounts of light spirit, as well as permanent gas, can be obtained from gas oils. A plant, having a generator 7 ft. in diam. and 15 ft. high, was supplied with 20.3 gals. of oil per min.; this was cracked at about 690° and provided gas of 665 B.Th.U. per cub. ft. and an 18% yield of oil boiling below 200°. The rate of oil feed is appreciably higher, and the temperature lower, than that required to give the best yield of permanent gas. R. H. GRIFFITH.

**Generation of [water]-gas.** POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 337,825, 9.8.29).—Water-gas is generated in a number of units each consisting of a generator, a waste-heat boiler, a generator jacket boiler, and a superheater. The units are arranged in two sets, alternately on "blow" and "run," the "blow" air in one set and the "run" steam in the other being superheated by the waste heat of the gases after these have passed through the waste-heat boiler. The gas passes through the latter preferably in a downward direction. A. B. MANNING.

**Continuous manufacture of water-gas.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,807, 6.8.29).—The fuel, in granular form and preheated to about 500°, is passed down externally heated, narrow, brickwork chambers or cylindrical vertical retorts, which are subdivided into several cells, lying one above the other, by inclined plates which constrict the diameter of the retort. Steam, preferably preheated to 800°, is passed into each cell and the water-gas produced is led away through a collecting chamber in the upper part of the cell; this chamber serves also as a deflector for forcing the fuel in the thinnest layer practicable against the heating walls so that it is raised rapidly and uniformly to the required temperature before it passes downwards into the next gasifying zone. A. B. MANNING.

**Cooling of combustible gas for removal of condensable vapours.** HUMPHREYS & GLASGOW, LTD., ASSECS. of J. S. HAUG (B.P. 339,885, 24.2.30. U.S., 6.5.29).—The relief holder on the gas stream from a carburetted water-gas plant is connected between the inlet and outlet of a condenser in which the hot gases are cooled. Cooling water in this condenser is circulated countercurrent to the gas stream. R. H. GRIFFITH.

**Removal of sulphurous compounds from gases containing them.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, and SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.)

(B.P. 317,015, 7.8.29. Fr., 8.8.28).—Water-gas is freed from organic sulphur compounds by adding sufficient coke-oven gas thereto and subjecting the mixture to progressive liquefaction under such conditions that the gas is washed by the liquefied ethylene and methane so produced. The purified gas may then be separated into mixtures of carbon monoxide and hydrogen, or nitrogen and hydrogen etc. suitable for various industrial syntheses. A. B. MANNING.

**Purification of gases.** A. O. JAEGER (ASST. to SELDEN Co.) and J. A. BERTSCH (U.S.P. 1,765,869, 24.6.30. Appl., 28.8.25).—Volatile metalloid compounds, particularly those of sulphur, are removed from gases or gaseous mixtures by treatment with halogens, especially chlorine and bromine. The reactions which take place may be assisted by physical means such as pressure, radiation, etc., or by catalysts. The resulting compounds of the substance to be eliminated are easily removed from the gas stream, e.g., by washing with a high-boiling organic solvent (tetralin) followed by treatment with solid absorbents. R. H. GRIFFITH.

**Manufacture of gases and the like.** I. G. FARBENIND. A.-G. (B.P. 319,657, 31.7.29. Ger., 25.9.28).—The gases leaving the apparatus in which coal, oil, etc. is undergoing destructive hydrogenation are subjected to fractional condensation in stages by strong cooling or by high compression. A. B. MANNING.

**Treatment of coke-oven gas.** COMP. DE BETHUNE (B.P. 314,432, 22.6.29. Fr., 27.6.28).—A mixture of hydrogen, nitrogen, and carbon monoxide suitable for the direct synthesis of methyl alcohol and ammonia in successive stages is produced by the incomplete combustion of coke-oven gas, from which a large proportion of the hydrogen has already been removed, and the re-addition of the requisite amount of hydrogen after the elimination of the unsaturated hydrocarbons and carbon dioxide. The combustion of the gas is so regulated that the carbon dioxide content of the product lies between 4% and 5%, and no oxides of nitrogen are formed. The unsaturated hydrocarbons are removed by heating the gas in the presence of a catalyst containing iron, nickel, and/or chromium, and the carbon dioxide by washing with a solution of ammonia. A. B. MANNING.

**Adjusting the calorific value and density of coal-distillation gases.** A. POTT (B.P. 312,348, 26.4.29. Ger., 25.5.28).—The coal gas is diluted with a gas produced by the interaction of part of the original gas with steam and/or air at high temperatures, e.g., 1100—1200°. This diluent gas is cheaper than water-gas and has a lower carbon monoxide content; moreover, its density can be varied within wide limits by varying the proportions of air and steam used in its production. A. B. MANNING.

**Manufacture of fuel gas.** J. B. GARNER, ASST. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,767,357, 24.6.30. Appl., 30.3.23).—Natural gas is passed through a heated vessel in which it is decomposed to give a gas of low calorific value, and carbon black. This gas is mixed with the untreated natural supply in such amount that the calorific value is reduced to about 800 B.Th.U. per cub. ft. R. H. GRIFFITH.



**Catalytic desulphurisation of gases.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,016, 16.9.29).—Organic sulphur compounds present in carbonisation gases are converted into hydrogen sulphide by passage, with an excess of hydrogen, over a catalyst consisting of an oxide or sulphide of elements in groups II—VII, together with metals of group VIII or heavy metals of group I. *E.g.*, gas containing 0.44 wt.-% of carbon disulphide is passed at 280° and under normal pressure over a catalyst obtained by precipitating molybdenum sulphide on Florida earth, and adding nickel carbonate. The exit gas contains sulphur in the form of hydrogen sulphide only. R. H. GRIFFITH.

**Purifying from naphthalene the distillation gases of coal.** GEWERKSCHAFT M. STINNES (B.P. 311,326, 29.4.29. Ger., 9.5.28. Cf. B.P. 333,297; B., 1930, 1055).—The gas is treated at above 30° with a solvent oil comprising a mixture of low-boiling oils with higher- or high-boiling oils, and the gas, charged with the vapour of the lower-boiling constituents of the solvent, is then cooled. The solvent vapour is precipitated together with the naphthalene, and, after separation from any condensed water and from at least a part of the absorbed naphthalene, is returned to the solvent oil.

A. B. MANNING.

**Recovery of naphthalene from gases.** N.V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & CO. G.M.B.H. (B.P. 337,723, 19.5.30. Ger., 17.5.29).—A solvent for naphthalene, *e.g.*, xylene, is introduced into the gas in a finely-subdivided form, and the gas containing the solvent in suspension is passed through an electrical gas purifier, wherein the solvent containing the dissolved naphthalene is precipitated. The solvent is then separated from the naphthalene and returned to the current of gas before it enters the electrical purifier.

A. B. MANNING.

**Tar for use on roads.** THERMAL INDUSTRIAL & CUEM. RES. CO., LTD., and C. O. CONDRUP (B.P. 339,640, 6.8.29).—Coal tar is treated with sulphuric acid in small amounts, at about 50–80°, and the oils which separate from the acid layer are fractionated, particularly for the removal of waxy substances. Road-making material of any desired consistency may be obtained by blending different fractions of this oil with products recovered from the acid sludge. In some cases further heat treatment of the mixtures is necessary in order to give a tar of the requisite viscosity. R. H. GRIFFITH.

**Conversion of petroleum oils.** PETROLEUM CONVERSION CORP., Assees. of A. P. SACHS and E. W. BEARDSLEY (B.P. 340,021, 19.9.29. U.S., 6.10.28).—The hydrocarbon oil is fractionally vaporised and the fractions are separately led off and added to the reaction zone at separated points along the path of the reacting products, so that the lighter fractions are subjected to a longer time of conversion than the heavier fractions. The temperature of the reacting gases is controlled by introducing heated carrier gas at the same points as the various oil fractions are admitted. H. S. GARLICK.

**Cracking of hydrocarbon oils.** J. E. POLLAK. From PETROLEUM CONVERSION CORP. (B.P. 340,030, 19.9.29. Cf. B.P. 340,030; preceding abstract).—The oil is vaporised and passed into the first of a series of re-

action chambers (preferably three) in which it is mingled with a heated gaseous carrier, the volume of which is approx. one half the total amount of gaseous carrier added at all stages, and at such a temperature that the temperature fall through the chamber is less than 55°. The mixture is passed to a second and thereafter a third reaction chamber, in each of which it is mingled with about an equal quantity of the remaining heated carrier gas, the amount added being sufficient in each case to raise the temperature of the reaction mixture substantially to its conversion temperature. A mean reaction temperature level is thus maintained throughout the operation, and the exit temperature of the last stage is not substantially more than 55° below the desirable cracking temperature. H. S. GARLICK.

**Fractionation of [hydrocarbon] oils.** W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,298, 24.6.30. Appl., 12.1.25).—Oil is passed through a cracking tube and digestion chamber to a fractionating column maintained under approximately the same pressure as the cracking apparatus. The composition of the vapours leaving the top of the column is controlled by means of a cooling coil or by adding reflux. Reflux is taken from the column by means of a tray situated above the point of entry of the oil from the digestion chamber. The heavy oil in the bottom of the column is stripped of light products by means of a current of inert gas and then returned to the cracking cycle. T. A. SMITH.

**Condensation of hydrocarbon vapours.** N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,299, 24.6.30. Appl., 12.7.27).—Oil vapours are indirectly cooled with water or other low-boiling liquid which is vaporised under reduced pressure. The final cooling and also the condensation of the steam or other vapour produced is carried out by bringing the oil condensate and the vapours in indirect contact with salt water. T. A. SMITH.

**Purification of [water-free] gasoline.** W. S. BAYLIS, Assr. to FILTROL Co. OF CALIFORNIA (U.S.P. 1,766,211, 24.6.30. Appl., 19.9.27).—Gasoline is decolorised by treatment with water-free clay to which 6% by wt. of anhydrous sulphuric acid has been added. The mixture of clay and gasoline is then passed through a filter press. T. A. SMITH.

**Low-boiling hydrocarbon oils.** STANDARD OIL DEVELOPMENT Co., Assees. of H. G. M. FISCHER and C. E. GUSTAFSON (B.P. 319,362–3, 19.9.29. U.S., 21.9.28).—Gum and acid formation in low-boiling hydrocarbon oils, *e.g.*, gasoline and naphthas, especially those from cracking processes, is inhibited or decreased by the addition of one or more stabilising agents in total quantity not exceeding 1 g. per litre. Suitable agents consist of an aromatic hydrocarbon with condensed nuclei having an  $\alpha$ -substituted group which is able to combine with acids, *e.g.*,  $\alpha$ -naphthol, either (A) alone, or (B) with an aromatic hydrocarbon with condensed nuclei, *e.g.*, anthracene. Small quantities of anti-detonating agents may also be present. H. S. GARLICK.

**Absorption of olefines in sulphuric acid.** STANDARD OIL DEVELOPMENT Co., Assees. of H. E. BUC (B.P.

340,098, 14.11.29. U.S., 15.11.28).—Olefine mixtures are treated with sulphuric acid in three successive stages, the concentration of the acid for the first stage being below 65%, for the second 78–82%, and for the final stage above 90%, the spent acid from each stage being collected separately. The temperature is kept at 32–49° during the first two stages. The quantity of acid supplied to the second stage is not less than one mol. per mol. of olefine. Where gaseous olefines are being treated it is advantageous to add an absorption oil along with the acid. H. S. GARLICK.

**Refining of hydrocarbons.** T. T. GRAY, ASSR. to GRAY PROCESSES CORP. (U.S.P. 1,759,814, 20.5.30. Appl., 9.8.26).—A hydrocarbon oil is cracked and the vapours are subjected to partial condensation to obtain a vapour fraction of definite boiling range, *i.e.*, motor fuel, which while still in the vapour phase is brought under pressure into contact with solid, absorptive, catalytic material, thereby polymerising constituents which tend to discolour on exposure to light or to form deposits. Condensate containing polymerides is continuously drained from the vicinity of the treating zone from which the vapours of the treated fraction are continuously withdrawn and separately condensed.

H. S. GARLICK.

**Production of fuels for internal-combustion engines.** R. T. HURLEY (B.P. 315,706, 3.7.29. U.S., 16.7.28).—The use of iron carbonyl as an anti-knock agent has the disadvantage that a conducting layer of iron oxide is deposited on the spark-plug insulators. By the addition to the fuel of lead tetraethyl or nickel carbonyl in a proportion not exceeding 10% of the iron carbonyl present the deposited layer becomes non-conducting.

A. B. MANNING.

**Reconditioning of sand filters [containing mineral oil sludge]. Continuous acid-washed sand filter.** C. S. TEITSWORTH, ASSR. to GEN. PETROLEUM CORP. (U.S.P. 1,769,475–6, 1.7.30. Appl., 9.8.29).—A sand filter of which all the grains are coated with acid sludge removed from petroleum is cleaned: in (A) by mechanically displacing part, and hydrolysing the remainder, of the sludge by means of a comparatively small quantity of water, and then removing the water and hydrolysed sludge by one or more small washes with oil; and in (B) by washing with 90–100% sulphuric acid. Any oil present at the beginning or end of flushing is easily settled in a clean state. B. M. VENABLES.

**Revivification of fuller's earth and other [mineral oil] filtering clays.** H. LOWERY, ASSR. to STANDARD OIL Co. (U.S.P. 1,763,167, 10.6.30. Appl., 25.5.27).—Filtering clay containing mineral oil is heated at 85–100° in a solution of sodium carbonate (*d* 1.06), which is vigorously agitated until the oil separates from the clay particles; these are then allowed to settle, washed free from soda, dried, and ignited to remove the last traces of oil.

A. R. POWELL.

**Utilisation of [low-compression] motor fuels.** T. MIDGLEY, JUN., ASSR. to GEN. MOTORS CORP. (U.S.P. 1,787,419, 30.12.30. Appl., 22.1.26).—See B.P. 196,237; B., 1924, 165.

**Purification of hydrocarbon oils.** H. G. M. FISCHER, ASSR. to STANDARD OIL DEVELOPMENT Co.

(U.S.P. 1,767,356, 24.6.30. Appl., 19.8.27).—See B.P. 295,728; B., 1930, 231.

**Regeneration of lubricating oils.** H. BENSMANN (U.S.P. 1,787,353, 30.12.30. Appl., 2.11.26. Ger., 25.3.26).—See B.P. 268,284; B., 1927, 516.

**Preparation of petrolatum and the like.** F. LAVIROTTE, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,785,270, 16.12.30. Appl., 30.9.25. Fr., 4.10.24).—See B.P. 240,803; B., 1926, 40.

**Hardening of paraffin, waxes, and the like.** W. PUNGS and M. JAHRSTORFER, ASSRS. to I. G. FARBEIND. A.-G. (U.S.P. 1,786,263, 23.12.30. Appl., 22.12.27. Ger., 23.12.26).—See B.P. 306,053; B., 1929, 314.

**Furnace for burning fuel mixtures.** G. HOFMANN (B.P. 337,490, 12.8.29).

**Doors for horizontal coking chambers.** W. W. GROVES. From C. OTTO & Co., G.M.B.H. (B.P. 340,453, 18.3.30).

**[Burner devices for] furnaces burning liquid, gaseous, or pulverised fuel.** J. G. MCKEAN and R. F. JONES (B.P. 340,630, 8.10.29).

**Catalysing devices, particularly for internal-combustion engines.** D. BALACHOWSKY, P. CAIRE, and M. LEVY (B.P. 339,251, 26.7.29).

**Separation of finely-divided impurities from liquids (B.P. 339,636). Distillation of mixed liquids (B.P. 339,663). Gas-washing apparatus (U.S.P. 1,767,628).—See I. Unsaturated hydrocarbons (B.P. 337,434). Products from olefines (B.P. 316,951).—See III. Hydrogen (U.S.P. 1,756,934).—See VII. Bituminous emulsions (B.P. 333,303). Road material (U.S.P. 1,769,442).—See IX. Coated electron metal [for engine cylinders] (B.P. 339,722). Conversion of carbonaceous materials into hydrocarbons (B.P. 339,276).—See X. Impregnating fabric (B.P. 338,538).—See XIII. Indicator of atmospheric pollution by gases (B.P. 340,195).—See XXIII.**

### III.—ORGANIC INTERMEDIATES.

**Recent experiments on the pyrolysis of methane.** R. V. WHEELER and W. L. WOOD (Fuel, 1930, 9, 567–574. Cf. B., 1929, 88).—Some further experiments, both of the "static" and "flowing" type, have been carried out on the pyrolysis of methane. The decomposition temperature, even in the presence of a catalyst, *e.g.*, iron, which accelerates the decomposition at a higher temperature, lies between 650° and 675°. The rapid initial decomposition of methane in a silica bulb is chiefly a surface effect, its rate being increased by packing the bulb with silica; but the reaction does not proceed to a true equilibrium owing to the formation of a protective layer of adsorbed hydrogen on the surface (cf. Holliday and Exell, A., 1929, 773). Under "flowing" conditions with periods of heating of the order of 0.3 sec. at 1050°, in a silica tube, 84% of the methane is converted into higher hydrocarbons. With an increase in the duration of heating more of the methane is decomposed into its elements. With the shortest periods of heating the higher hydrocarbons consist essentially of molecules with two carbon atoms, which, with slightly increased periods of heating,

condense to form aromatic hydrocarbons. Butylene and butadiene are formed during the reaction, but neither propylene nor acetylene could be detected in the products. The use of an iron tube leads to the complete decomposition of the methane into its elements at a comparatively low temperature, the carbon formed being of a bulky gritty nature. The carbon deposit on the silica or chrome-iron tubes is of a lustrous foil-like variety, and its accumulation does not appear to affect the course of the reaction. No difference can be detected between chrome iron and silica as regards the formation of liquid hydrocarbons from methane. Moreover, an extensive increase in the area of silica exposed per unit volume of gas has no appreciable effect on the rate or extent of the decomposition. It is concluded that the reactions which are of importance under "flowing" conditions are those which occur throughout the volume of the gas when the surface reaction has been inhibited by the adsorbed hydrogen layer. It appears probable that with iron this protective layer is not formed. The formation of benzene is regarded as taking place through the intermediate formation of ethylene and butylene and/or butadiene.

A. B. MANNING.

**Ether: use of potassium hydroxide in testing for aldehydes.** L. W. GREEN and R. E. SCHOETZOW (Amer. J. Pharm., 1930, 102, 652).—In the U.S.P. X. test, 50% aqueous or solid potassium hydroxide gives more distinct yellow colour with ethyl ether containing 0.01% of aldehyde than does the more dilute solution prescribed.

H. E. F. NOTTON.

**Catalytic reduction and hydrogenation of phenols by hydrogen under pressure.** H. TROPSCHE [with B. HLAVICA and O. WEINSTEIN] (Brennstoff-Chem., 1930, 11, 449—452).—The influence of catalysts on the hydrogenation of tricresol has been studied. The experiments were carried out at 460—480° in small rotating autoclaves, capacity 1.9 or 4.5 litres, with a maximum hydrogen pressure of about 200 atm. and a reaction time of 1—2 hrs. Zinc oxide, copper chromate, tungstic oxide, and ferric hydroxide had no catalytic effect. Zinc chloride, aluminium hydroxide, ammonium vanadate, chromium hydroxide, uranium oxide, cobalt hydroxide, and nickel sulphide all had an appreciable effect, and gave yields of hydrocarbons up to 25% of the starting material. The most efficient catalysts were molybdenum oxide and sulphide, which gave yields of hydrocarbons of about 80%, leaving only about 4% of unchanged cresol in the product. Nickel hydroxide was also active as a catalyst, giving 56% of hydrocarbons, but was distinguished from molybdenum oxide by its sensitivity towards sulphur; nickel sulphide was much less active than the hydroxide. About 1% of the initial material appeared as gaseous hydrocarbons. The greater part of the hydrocarbons produced boiled within the range 95—125°. Analysis of this fraction by Kattwinkel's method (B., 1928, 78) gave approx. 20% of unsaturated hydrocarbons and 40% of aromatic hydrocarbons. A partial hydrogenation of the aromatic hydrocarbons had therefore taken place; this was more marked with nickel hydroxide than with molybdenum oxide as catalyst. No cyclic alcohols were formed.

A. B. MANNING.

**Humic acids.** THIESSEN and ENGELDER. **Products of distillation of pine wood.** KUPFERBERG. **Hexane isomerides from petroleum.** BRUUN and HICKS-BRUUN.—See II. **Differentiation between *p*-phenylene- and *p*-tolylene-diamines.** GRIEBEL.—See IV. **Effect of carbon tetrachloride etc. on metals.** FORMÁNEK.—See X. **Citric acid fermentation.** FREY. **Detection of isopropyl alcohol.** BODENDORF.—See XVIII. **Amyl acetate in sensitometry.** KIESER.—See XXI.

## PATENTS.

**Manufacture of oxygenated organic compounds [(A) from carbon oxides and hydrogen, (B) from methane and steam].** H. DREYFUS (B.P. 337,409 and 337,410, 24.7.29).—(A) Alcohols are obtained by hydrogenation of carbon oxides in presence of borates, silicates, or phosphates of iron, nickel, or cobalt, especially the acid salts, and preferably in association with alkalis or alkaline-earths at 250—400°/50—200 atm. The carbon oxides may be partly or wholly replaced by the alcohols produced by hydrogenation. (B) Methane and steam are heated at 200—500°, preferably in presence of metals or metal compounds at 50—100 atm. Hydrogen, carbon oxides, or diluent gases may be added to the mixture. Suitable catalysts are potassium dichromate with zinc oxide, zinc chromate with cobalt oxide, and zinc chromate. The products are mainly methyl and other alcohols.

C. HOLLINS.

**Manufacture of [unsaturated] hydrocarbons.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,434, 30.5.29).—A Cowper tower packed with material of high heat capacity and preferably having catalytic properties is heated by burning (producer) gas with enough oxygen to remove carbon deposited from a preceding reaction. When the temperature reaches, e.g., 900° at the base of the tower the burning gas is led down the tower at a rate which is decreased as the tower cools. Olefines are obtained at 700—900°, benzene at 800—1100°.

C. HOLLINS.

**Manufacture of condensation products from olefines and unsaturated [aromatic] hydrocarbons.** I. G. FARBENIND. A.-G. (B.P. 316,951, 6.8.29. Ger., 4.8.28).—Olefines over C<sub>2</sub> are condensed alone or with an aromatic hydrocarbon by heating at 120—180°/10—20 atm. in presence of a highly porous hydrosilicate (fuller's earth), with or without addition of another catalyst, e.g., metal halide or hydrogen halide. A solvent may be employed. Propylene and naphthalene give tetraiso-propylnaphthalene, m.p. 126°; propylene alone yields a middle oil, b.p. 120—200°/16 mm., and a lubricating oil, b.p. 200—250°/16 mm.

C. HOLLINS.

**Manufacture of products [derivatives of higher hydrocarbons] containing nitrogen.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,962, 11.9.29).—Halogenated derivatives of paraffin hydrocarbons (above C<sub>8</sub>) are heated with ammonia in aqueous or alcoholic solution or with agents yielding ammonia, e.g., ammonium carbonate or urea, with or without the addition of copper or copper compounds as catalysts, preferably under increased pressure.

H. S. GARLICK.

**Manufacture of acetone from isopropyl alcohol.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROL-

EUM MAATS. (B.P. 337,566, 10.10.29).—In the catalytic oxidation of isopropyl alcohol at 400° in presence of copper, manganese peroxide, barium peroxide, a platinum metal, zinc oxide, cadmium oxide, uranium oxide, blue tungsten oxide, vanadium pentoxide, magnesia, beryllium oxide, or zirconium oxide there is added to the catalyst about 1% of zinc oxide, thorium oxide, sodium carbonate, cerium oxide, or zirconium oxide, or more than one of these. C. HOLLINS.

**Polymerisation of 1 : 3[ $\alpha\gamma$ ]-butadiene, homologues and analogues thereof.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,460, 2.8.29).—Rubber-like products are obtained by polymerisation of butadiene etc. by means of alkali metal placed in a small glass or metal container provided with a few perforations or slits. Polymerisation is complete within the small container in about 24 hrs., but only commences in the bulk of the liquid after several days. The process may be performed in 2 stages, the contents of the small container being polymerised before it is introduced into the bulk. C. HOLLINS.

**Manufacture of fatty acid derivatives [wetting, emulsifying, and cleaning agents].** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,368, 26.7.29).—Fatty acids (lauric, palmitic, stearic, ricinoleic, wool fat acids) or their esters are heated at 150–200° with diethylenetriamine or other polyalkylenepolyamine; the products become water-soluble on alkylation, giving emulsifying and wetting agents not precipitated by alkali. C. HOLLINS.

**Manufacture of aminohydroxyanthraquinones and substitution products thereof.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,047, 26.7.29).—Phthalic anhydride etc. is condensed with an acylaminophenol, e.g., in presence of aluminium chloride, and the product, after hydrolysis, is cyclised by treatment with sulphuric acid. Di-*o*-anisylcarbamide gives an intermediate acid, m.p. 188°, which is hydrolysed and cyclised by 96% sulphuric acid at 125–135° to form 2-amino-3-hydroxyanthraquinone. The preparation of 1-amino-4-hydroxyanthraquinone from 4-benzamidophenol, of 5-chloro-2-amino-3-hydroxyanthraquinone (decomp. 333°) from di-*o*-anisylcarbamide (intermediate acid, m.p. 274–276°), of 4-chloro-2-amino-1-hydroxyanthraquinone (m.p. 254–255°) from phosgenated 4-chloro-*o*-anisidine, and of 2-amino-1-hydroxy-4-methylantraquinone (m.p. 204–205°) from phosgenated cresidine, is described. C. HOLLINS.

**Catalytic oxidation of organic compounds [hydrocarbons].** A. WOHL, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,787,416–7, 30.12.30. Appl., [A] 11.8.21, [B] 8.8.29. Ger., [A, B] 22.6.16).—See B.P. 156,244; B., 1922, 407 A.

**Manufacture of triaryl phosphates.** W. GIBSON, C. R. HENSHAW, and J. B. PAYMAN, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,785,951, 23.12.30. Appl., 7.9.29. U.K., 27.9.28).—See B.P. 322,036; B., 1930, 95.

**Production of thymol.** W. SCHOELLER and H. JORDAN, Assrs. to CHEM. FABR. AUF AKT. (VORM. E. SCHERING) (U.S.P. 1,786,922, 30.12.30. Appl., 20.6.27. Ger., 29.6.26).—See B.P. 273,685; B., 1930, 136.

**Preparation of 4- and 2-halogeno- and 4- and 2-amino-*p*-phenyl-*o*-benzoylbenzoic acid, of 4- and 2-amino-3-phenylanthraquinone by amination, and of *p*-phenoxy-*o*-benzoylbenzoic acid.** P. H. GROGINS (U.S.P. 1,786,526–1,786,532, 30.12.30. Appl. [A–F] 8.8.29, [G] 2.10.29).—See A., 1930, 1186.

**Preparation of hydroxythionaphthens.** E. HOFFA and F. MÜLLER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,785,813, 23.12.30. Appl., 2.8.28. Ger., 17.8.27).—See B.P. 295,694; B., 1930, 276.

**Purification of crude anthracene.** A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP. (Re-issue 17,902, 16.12.30, of U.S.P. 1,693,713, 4.12.28).—See B., 1929, 350.

**Catalytic oxidation of anthracene.** A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,786,950, 30.12.30. Appl., 29.3.28).—See B.P. 295,270; B., 1929, 806.

**Coke-oven gas (B.P. 314,432). Naphthalene from gases (B.P. 311,326 and 337,723). Absorption of olefines (B.P. 340,098).**—See II.

#### IV.—DYESTUFFS.

**Fastness of dyestuffs in ultra-violet light. III.** G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 292–298; cf. B., 1930, 896).—A number of dyestuffs of the azo, pyrazolone, di- and triphenylmethane, xanthene, acridine, quinoline, azine, oxazine, and thiazine groups are classified, according to their resistance to the action of ultra-violet irradiation, into four groups, showing: (1) little or no change in colour after about 3 hrs., (2) appreciable change within 1–3 hrs., (3) marked change in 1–2 hrs., or (4) intense change of colour or pronounced decolorisation after 1 hr. T. H. POPE.

**Differentiation of *p*-phenylenediamine and *p*-tolenylenediamine in hair dyes.** C. GRIEBEL (Apoth.-Ztg., 1930, 45, 318–320; Chem. Zentr., 1930, ii, 593).—Both substances give a yellow colour with a reagent consisting of vanillin (0.05 g.), alcohol (1 c.c.), and 25% hydrochloric acid (4 c.c.); with the former, however, the colour soon becomes red.

A. A. ELDRIDGE.

#### PATENTS.

**Manufacture of substitution products of sulphur dyes.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,061, 27.7.29. Addn. to B.P. 317,776; B., 1930, 10).—The process of alkylation or aralkylation in substance or on the fibre is applied to the products of B.P. 325,519 (B., 1930, 455). The sulphur dyes from tetra- and penta-chlorinated dinaphthylene dioxide, treated on the fibre with phenylbenzyltrimethylammonium chloride, give orange shades. C. HOLLINS.

**Manufacture of wool dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,021, 21.6.29).—The copper compound of a 4-halogeno-1-hydroxyanthraquinone-2-sulphonic acid, precipitated by adding copper sulphate to a solution of the sodium salt, is condensed with amines or amides, R.NH<sub>2</sub>, where R is an alkyl, cycloalkyl, aralkyl, aryl, or acyl group. *p*-Aminoacetanilide gives a navy-blue wool dye, 4-acetamido-*o*-toluidine a bluish-violet, *m*-phenylenediamine a bluish-grey. C. HOLLINS.

**Manufacture of derivatives [vat dyes] of the acridone series.** J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 337,374, 26.4. and 12.6.29 and 13.1.30).—Orange-red to red-brown vat dyes are obtained by chlorination and/or bromination of complex quinonoid acridones. Examples of starting materials are: 1:2:6:7-diphthaloylacridone ( $N=5$ ); diacridone from benzidine and 2 mols. of 1-chloroanthraquinone-2-carboxylic acid; diacridone from dibromo-1:2:6:7-dibenzopyrene-3:8-quinone and 2 mols. of 2-aldehydo-1-aminoanthraquinone; acridone from bromo-1:2:6:7-dibenzopyrene-3:8-quinone and 2-aldehydo-1-aminoanthraquinone. C. HOLLINS.

**Manufacture of pyranthrone derivatives.** A. SHEPHERDSON and S. THORNLEY, ASSRS. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,785,600—1, 16.12.30. Appl., [A] 29.9.27, [B] 9.5.29. U.K., [A] 3.10.26, [B] 8.10.26).—See B.P. 282,913; B., 1928, 152.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Determination of small amounts of copper in textiles.** R. E. RUPP (Proc. Amer. Assoc. Textile Chem. Colorists, 1930, 215—217).—The sample (10—20 g.) is digested with concentrated nitric (25 c.c.) and sulphuric (25 c.c.) acids and potassium sulphate (2 g.), the residue being rendered alkaline with ammonia and filtered through asbestos. The copper is then determined colorimetrically as ferrocyanide.

CHEMICAL ABSTRACTS.

**Determination of copper, manganese, and iron in fabrics.** W. C. SMITH (Proc. Amer. Assoc. Textile Chem. Colorists, 1930, 217—219).—The sample (10 g.) is digested with fuming nitric acid (2.5 c.c.), sodium sulphate (2.5 g.), and potassium sulphate (2.5 g.), digestion being completed after addition of sulphuric acid (25 g.). The residue is diluted, rendered alkaline with ammonia, and then acid to Congo-red with 10% sulphuric acid, 5 c.c. in excess being added. Aliquot portions are employed for the determination of copper (ethyl xanthate), manganese (Willard and Greathouse), and iron (thiocyanate, with addition of nitric acid).

CHEMICAL ABSTRACTS.

**Pine wood.** M. SOUM (Bull. Inst. Pin, 1929, 173—182, 253—264, 277—291, 313—316).—Analyses of various samples of maritime pine wood are given, and celluloses and lignins prepared from them are compared. Under the action of weathering, micro-organisms, and parasites, cellulose is degraded, whilst lignin is in great part unattacked. The lignocelluloses thus yield lignin soluble in alcohol, acetone, and pyridine. The attacked lignin contains fewer methoxyl and hydroxyl groups and has a smaller phloroglucinol number. Under certain conditions, after felling of the tree the stumps remaining in the soil undergo conversion into "light wood" ("bois gras"), becoming very rich in resin (up to 40%), whilst the cellulose and lignin retain their mechanical and chemical properties. Under the influence of certain reducing diastases ("resinases") the cellulose of the stump is almost completely converted into oleo-resins, and the lignin approaches in composition and properties the humic acids of lignites. Protected from air the wood undergoes "mummification" in the soil,

the ligneous elements being preserved and the resins converted into retene. A fossil coniferous wood, rich in free humic acids but containing only light cellulose, has been identified in the ligniferous strata of the Lalueque basin. The valuable constituents of "light wood" suggest its artificial production by the action of resinases on wood that has not yet been long enough in the soil. The resinases appear to be secreted by the wounded cells.

C. HOLLINS.

### Semi-chemical pulp prepared from spruce wood by the calcium bisulphite-steam cooking process.

C. G. SCHWALBE and N. F. HAGSTRÖM (Cellulosechem., 1930, 11, 233—249).—About 4 kg. of the wood in the form of chips is impregnated with 28 litres of a solution of calcium bisulphite (4.8%  $\text{SO}_2$ , CaO 1.0%) in an autoclave at a temperature below that necessary to cause dissolution of the lignin; the excess liquor is then drained away and the residue cooked at a higher temperature by admission of steam. The drainage liquid contains about one third of the original sulphur dioxide and a small quantity, usually  $< 3\%$ , of organic matter. When the bisulphite treatment is conducted at  $120^\circ$  for 4—7 hrs., and the steaming at  $145^\circ$  for 0.5—1.5 hrs., fibre (yield 45%; lignin content 10%) is obtained which can easily be pulped and is bleachable. On the other hand, bisulphiting at  $105^\circ$  for 6 hrs. followed by steaming at  $130^\circ$  for 3 hrs. yields 60% of fibre, but the product is less readily pulped, contains 20% of incrusting materials (chiefly lignin), cannot be bleached with normal quantities of reagent, but yields paper of good quality. The liquid from the steaming process contains, besides ligninsulphonic acids, most of the hemicelluloses of the wood. The concentration of fermentable sugars (arising from hydrolytic degradation of the cellulose) is very low. During the imbibition stage sulphur dioxide is used up in the formation of insoluble lignin-sulphonic acids, which partly dissolve during the second stage of the process. The disintegration of a hard fibre into pulp is aided considerably by treatment with a 1% solution of magnesium hydroxide for 24 hrs. at ordinary temperatures; treatment with solutions of hydrochloric and sulphuric acids, calcium bisulphite, and calcium hydroxide is without marked action. Disintegration is also aided by treatment with a solution of chlorine, which is rapidly absorbed, followed by an equivalent amount of sodium hydroxide solution.

T. H. MORTON.

**Physical properties of artificial silks and their relations to the prime materials and methods of preparation.** E. VIVIANI (Giorn. Chim. Ind. Appl., 1930, 12, 519—536).—Methods are described for examining the covering power, elasticity, sections, regularity of filament, etc. Experiments made show that the maturation of alkali-cellulose in presence of oxygen is characterised by diminution in viscosity of the viscose prepared with it and of the solutions in Schweitzer's reagent. At normal temperatures (20— $30^\circ$ ), maturation in complete absence of oxygen results, not in diminution, but rather in increase of the viscosity. Maturation in presence of oxygen leads to the formation of soda-soluble derivatives (? oxycellulose), the proportion of which increases with the amount of oxygen absorbed, until finally the cellulose is converted completely into

soluble product. No such soluble products are obtained in absence of oxygen at 20—30°. As regards its practical applications, maturation of alkali-cellulose must be regarded essentially as an oxidation phenomenon, this view explaining the necessity of using sodium hydroxide free from impurities, such as metallic oxides, which accelerate absorption of oxygen. T. H. POPE.

**Physical and chemical nature of nitrocellulose.** A. BECK, L. CLÉMENT, and C. RIVIÈRE (*Chim. et Ind.*, 1930, 24, 1068—1072).—By the precipitation of a 10% acetone solution by acetone containing 20% of water nitrocellulose may be separated into four fractions. The final fraction is obtained as a colloidal suspension; the solute is recovered by addition of sodium or barium chloride, or, better, by removal of the solvent under reduced pressure. The four fractions have a nitrogen content identical with that of the original material, but the viscosity of their solutions falls, the initial fraction giving the highest and the final the lowest value. A similar series of fractions is obtained when to an acetone solution of nitrocellulose there is added a quantity of water insufficient to cause precipitation and then the acetone is gradually removed by distillation in a vacuum. T. H. MORTON.

**Properties of nitrocellulose made from jute, with special reference to its stability.** R. C. BAGCHI (*J. Indian Chem. Soc.*, 1930, 7, 863—874).—The yield of nitrocellulose from jute is considerably smaller than that from cotton, mainly owing to the presence of a considerable amount of  $\gamma$ -cellulose, which gives water-soluble nitro-compounds. Uniform nitration is also more difficult unless impurities are first removed from the jute. The jute nitrocellulose cannot be stabilised by boiling with 1% hydrochloric acid. If the product, after 4 boilings with acid, is boiled twice with 0.3% sodium carbonate solution, and then 6 times with water, a marked improvement in the stability of the nitrocellulose results. A still further increase in stability is found when the product is treated with alcohol during 3 days after the dilute acid treatment. Small amounts of sugars, gum, or hemicellulose cause the nitrocellulose to be markedly unstable. The effect of other impurities is studied, and it is found that the nitrogen content of the nitrocellulose decreases as the impurities increase. The viscosity of solutions of jute nitrocellulose in ether-alcohol is much less than that of similar solutions of cotton nitrocellulose. H. BURTON.

**Removing printer's ink from paper.** SCHWALBE.—See XIII.

#### PATENTS.

**Working up materials containing lignin and cellulose.** O. A. MÜLLER (B.P. 340,164, 10.2.30. Ger., 9.2.29).—Undesired lignin is removed from a mixture with cellulose by treatment with chlorine and then with an organic solvent, such as alcohol, which is miscible with water and contains a mineral acid. Chlorolignin may be recovered from the extract by dilution with water or by addition of caustic alkali. Other products, which may be tanning agents, often remain in the liquid and can be obtained by removal of the solvent or addition of an alkaline-earth hydroxide. R. H. GRIFFITH.

**Manufacture of cellulose fatty acid esters.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,366, 25.7. and 13.11.29 and 7.3.30).—Methylene chloride or ethylene chloride is used as solvent in the manufacture of aliphatic esters (including mixed aliphatic esters) of cellulose; 0.5% of sulphuric acid is sufficient catalyst. An undegraded product of high viscosity is obtained in 5 hrs. at 50°. The alkylene chloride assists the concentration of the dilute acetic or other acid when wet precipitation is employed. For mixed esters one acid and one anhydride may be used in the desired proportions, no excess of acid being needed for solvent purposes. C. HOLLINS.

**Treating [spinning] artificial silk made from viscose.** K. LEUCHS (U.S.P. 1,770,412, 15.7.30. Appl., 10.10.27. Ger., 5.7.26).—See B.P. 298,688; B., 1929, 125.

**Desulphurising articles consisting of regenerated cellulose.** W. ELLER and T. MADLUNG, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,786,941, 30.12.30. Appl., 7.5.28. Ger., 13.7.27).—See B.P. 293,833; B., 1929, 714.

**Manufacture of transparent papers, particularly those used for the packing of edible articles.** O. KLOTZ (U.S.P. 1,786,911, 30.12.30. Appl., 25.2.27. Ger., 9.10.26).—See B.P. 283,751; B., 1928, 229.

**Beaters for paper pulp.** W. KIRCHNER and G. STRECKER (B.P. 340,713, 14.11.29. Ger., 14.11.28).

**Sulphite-cellulose liquors (U.S.P. 1,765,560). Reflecting material (U.S.P. 1,767,285).**—See XIII.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### PATENTS.

**Bleaching and dyeing of jute, hemp, and like fibrous materials.** T. E. ROBERTSON (B.P. 316,265, 25.7.29. Austral., 26.7.28).—Superior bleaching is obtained, and the bleached material is more amenable to dyeing in fast shades with direct dyes, if the bleaching process consists of successive treatments with a dilute solution of chlorine and a solution of sulphur dioxide or other similar reducing agent without intermediate treatment. It is preferred to use a dilute acidified solution of bleaching powder and a boiling solution of sodium hyposulphite in the bleaching process.

A. J. HALL.

**[Ozonising] treatment of textile threads and fabrics.** O. HOFFMANN (B.P. 337,305, 31.1.30. Ger., 9.2.29).—Linen fabric is passed in open width through a chamber having upper and lower series of guiding rollers and through which is circulated ozonised air containing 0.2—0.4 g. O<sub>3</sub>/m.<sup>3</sup>; the treated linen can be more easily and thoroughly bleached subsequently by the usual methods. The process is suitable for other textile materials. A. J. HALL.

**Mordanting, weighting, and dyeing [of textile materials].** BRIT. CELANESE, LTD., W. A. DICKIE, and F. B. HILL (B.P. 337,813, 7.8.29).—Films and textile materials are treated with a high-speed stream of metal oxide or of oxidisable metal, the metal on the fabric being subsequently oxidised, whereby the oxide so thoroughly impregnates the material that mordant dyeing is rendered possible. The process is particularly

suitable for treating materials consisting of cellulose esters and ethers, and is conveniently carried out by means of the Schoop metallising apparatus. Suitable metals include zinc, aluminium, tin, and alloys of these.

A. J. HALL.

**Production of [mordanted or weighted] cellulose derivative [ester or ether] materials.** H. DREYFUS (B.P. 336,621, 9.7.29).—Cellulose acetate (etc.) materials containing fixed metal residues are produced by spinning a solution of cellulose acetate in acetone containing a soluble metal salt and then treating either directly in the coagulating bath (in the case of wet-spinning) or subsequently with substances capable of precipitating the metal. Suitable salts include stannic or ferric chlorides and thiocyanates, aluminium thiocyanate, zinc chloride, chromium thiocyanate, as precipitants tannic acid, ammonia, sodium monohydrogen phosphate, and sodium carbonate may be used. The weighted or mordanted silk may afterwards be dyed by means of direct or mordant dyes.

A. J. HALL.

**Naphthol dyeing.** F. KARRER (B.P. 338,012, 25.11.29).—In dyeing yarn wound on cops or bobbins by the "packed" system, in which the dye liquors are pumped through the material being dyed, the formation of loose colour on the surface of the yarn is avoided by drying the yarn between its treatments with a naphthol solution and with a diazo solution. The yarn is packed in a truck which is run into a dyeing chamber capable of being hermetically sealed by an airtight door. Naphthol solution is then pumped through the yarn, and excess liquor is removed by connecting the dyeing chamber to a vacuum pump; by admitting hot air the yarn is subsequently dried. Afterwards a diazo solution is pumped through the dry yarn and almost all the dye thus formed is within the fibre substance.

A. J. HALL.

**Dyeing of cellulose acetate fibres, films, etc. with aniline black and the like.** SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 337,746, 27.7.29).—Cellulose acetate silk is treated with an aqueous suspension or solution of aniline containing a small proportion (about 10%) of *p*-aminodiphenylamine or *p*-phenylenediamine and the resulting silk containing the absorbed amines is then treated with a hot acidified solution of a dichromate saturated or nearly so with sodium chloride; the silk thereby rapidly acquires a fast black shade. It is advantageous to add to the oxidising bath small amounts of copper sulphate and sodium chlorate.

A. J. HALL.

**Production of fast tints on materials [azo couplings of leuco-compounds].** SOC. CHEM. IND. IN BASLE (B.P. 318,833, 9.9.29. Switz., 7.9.28).—Textiles are padded with the leuco-compound of a vat or sulphide dye, and are then pressed and passed through a diazo solution. Fast browns and olives are produced. Examples are: Ciba blue 2B, vatted and coupled with diazotised benzeneazo- $\alpha$ -naphthylamine (black-brown); Cibanone yellow 3G, vatted and coupled with diazotised aniline  $\rightarrow$  cresidine (full brown); Pyrogene green 3G vatted in sodium sulphide and coupled with benzeneazo- $\alpha$ -naphthylamine (olive).

C. HOLLINS.

**Applying pictures in fast colours to textile fabrics.** A. DENGLER (B.P. 337,877, 30.8.29. Ger., 3.11.28).—Pictures which may be transferred to fabrics by hot-ironing are formed by suitably spraying paper with a solution containing dyes, mordants (e.g., sodium bisulphate, milk of lime), oxidising agents (e.g., potassium, percarbonate), and thickening agents (e.g., glue, soap, and particularly a resin soap).

A. J. HALL.

**Reducing or counteracting the attacks by alkaline liquids on straining cloths.** ALUMINIUM-IND. A.-G. (B.P. 337,559, 9.10.29. Ger., 17.1.29).—Cotton has increased resistance to alkaline liquors, such as those resulting from the treatment of bauxite in the manufacture of aluminium, if it is impregnated with a manganese-oxygen compound such as manganous hydroxide and particularly manganese dioxide. Such impregnation may be effected by adding manganese compounds to the alkaline liquors which come in contact with the cotton, or by treating the cotton with potassium permanganate.

A. J. HALL.

**[Laundry] washing processes.** A. MARQUARDT and E. WALTER (B.P. 338,121, 8.8.29).—In washing textile materials the detergent liquor is prepared by adding together or successively the separate components of a soap-forming mixture such as saponifying agents and saponifiable substances, without previously pressing the detergent liquor into or out of the articles being washed; additional substances such as alcohols, hydrocarbons, protective colloids, and bleaching agents may also be added.

A. J. HALL.

**Dye jigs.** MASCHINENFABR. RAPPERSWIL A.-G. (B.P. 340,431, 31.1.30. Switz., 2.2.29).

**Steaming of dyed or printed fabrics.** I. G. FARBENIND. A.-G. (B.P. 336,590, 15.7.29. Ger., 18.3.29. Addn. to B.P. 333,873).

**Wetting etc. agents** (B.P. 337,368).—See III. **Impregnating fabric** (B.P. 338,538).—See XIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Evaporation of caustic soda to high concentrations by means of diphenyl vapours.** W. L. BADGER, C. C. MONRAD, and H. W. DIAMOND (Ind. Eng. Chem., 1930, 22, 700—707).—A semi-works-scale plant was designed to study the evaporation of caustic soda, using as heating medium diphenyl vapour (b.p. 256°/1 atm., latent heat 138 B.Th.U./lb., m.p. 69°, sp. heat 0.4—0.6 according to temperature). The evaporator used was a nickel tube surrounded by an iron jacket down which the diphenyl vapour passed, returning to the boiler when condensed. On account of the corrosion of iron parts by caustic soda it was found necessary to use a nickel-cast iron impeller in the circulating pump, monel metal piping, and a nickel-cast iron vapour head. The pump was packed with laminated copper. No great difficulty occurred in handling the diphenyl. The pump was rendered tight by the use of a bronze, water-cooled stuffing box. Diphenyl pipe lines were traced with high-pressure steam lines and heavily insulated. A large number of determinations are given of heat-transfer coefficients for condensing diphenyl and boiling



caustic soda. The former film offered the greater resistance to heat transfer. The diphenyl coefficient tended to increase with increasing temperature difference, but was scarcely affected by vapour temperature. Caustic soda was concentrated in the apparatus up to 98.2% NaOH, and it is believed that a commercial apparatus on these principles is possible. C. IRWIN.

**Ammonia synthesis on a large laboratory scale.** W. GLUUD, R. SCHÖNFELDER, and W. REISE (Ber. Ges. Kohlentechn., 1930, 3, 52—95; Chem. Zentr., 1930, ii, 599—600).—Three series of experiments, employing pressures of 1, 100, and 300 atm., respectively, are described; various iron catalysts were employed. A. A. ELDRIDGE.

**Increasing the activity of iron [ammonia] catalysts.** W. GLUUD and R. SCHÖNFELDER (Ber. Ges. Kohlentechn., 1930, 3, 96—99; Chem. Zentr., 1930, ii, 594).—The catalyst containing iron is treated with carbon monoxide in order to form  $\text{Fe}_5\text{C}$ , which is then reduced by hydrogen. A. A. ELDRIDGE.

**Chemical and physical composition of certain finely-divided natural phosphates from Florida.** W. L. HILL, K. D. JACOB, L. T. ALEXANDER, and H. L. MARSHALL (Ind. Eng. Chem., 1930, 22, 1392—1396).—Tests on the particle size of waste-pond phosphates showed negligible quantities coarser than  $50\ \mu$ , 9—19.5% of  $50\text{--}5\ \mu$ , and 80.7—91.9% finer than  $5\ \mu$ . Soft phosphates were considerably coarser. Both waste-pond and soft phosphates may be heated up to  $500^\circ$  without physical change if the time of heating is not more than  $\frac{1}{2}$  hr. The average sp. gr. of waste-pond phosphates is 2.93, and of soft phosphate 3.09. The chemical compositions of several waste-pond and soft phosphates, and the variations in chemical composition with particle size, are tabulated. The citrate-soluble  $\text{P}_2\text{O}_5$  content of 26 samples varied from 0.4 to 11.1% (av. 3.8%) of the total  $\text{P}_2\text{O}_5$ ; citric acid-soluble  $\text{P}_2\text{O}_5$  varied from 19.7 to 40.3% (av. 28.7%). W. J. WRIGHT.

**Composition of citrate-insoluble residues from superphosphates and ammoniated superphosphates.** K. D. JACOB, W. L. HILL, W. H. ROSS, and L. F. RADER, JUN. (Ind. Eng. Chem., 1930, 22, 1385—1392).—The citrate-insoluble phosphate in ammoniated superphosphates and tricalcium phosphate, but not in non-ammoniated phosphates, decreased progressively with the weight of sample tested. The phosphoric acid dissolved from ordinary, triple, and ammoniated triple superphosphate was directly proportional to the weight of the sample. Solubility of water-insoluble phosphates in ammonium citrate solution depends on their chemical and physical nature and on the total amount present; it is reduced by the presence of other citrate-soluble salts, such as calcium sulphate. Citrate-insoluble residues from ammoniated and non-ammoniated superphosphates and triple superphosphates contain variable amounts of iron, aluminium, calcium, phosphoric acid, and fluorine, but little sulphate, whilst those from tricalcium phosphate consisted wholly or partly of calcium hydroxyphosphate. W. J. WRIGHT.

**Reactions occurring during the ammoniation of superphosphate.** F. G. KEENEN (Ind. Eng. Chem., 1930, 22, 1378—1382).—When the proportion of  $\text{NH}_3$

reaches 1 mol. per mol. of water-soluble  $\text{P}_2\text{O}_5$ , the reaction (1) is  $\text{CaH}_4(\text{PO}_4)_2 + \text{NH}_3 \rightarrow \text{NH}_4\text{H}_2\text{PO}_4 + \text{CaHPO}_4$ , and no loss of available  $\text{P}_2\text{O}_5$  occurs. With more than 1 mol.  $\text{NH}_3$ , and no control of temperature or moisture, the dicalcium phosphate disappears more rapidly than the monoammonium phosphate. Thus with 2 mols.  $\text{NH}_3$ , the temperature is  $70\text{--}90^\circ$  and the reaction (2), viz.,  $2\text{CaHPO}_4 + \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{NH}_3 \rightarrow \text{Ca}_3(\text{PO}_4)_2 + (\text{NH}_4)_2\text{SO}_4 + 2\text{H}_2\text{O}$ , occurs. As ammoniation increases the temperature rises to  $90\text{--}100^\circ$ , and when all the dicalcium phosphate has reacted, the reaction (3) proceeds as:  $\text{NH}_4\text{H}_2\text{PO}_4 + \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{NH}_3 \rightarrow (\text{NH}_4)_2\text{SO}_4 + \text{CaHPO}_4 + 2\text{H}_2\text{O}$ . Since monoammonium phosphate gradually decreases in amount as the ammonia absorption increases, reactions (2) and (3) must occur simultaneously. If a low temperature and moisture content are maintained the reaction may be controlled thus:  $\text{CaH}_4(\text{PO}_4)_2 + 2\text{NH}_3 \rightarrow \text{CaHPO}_4 + (\text{NH}_4)_2\text{HPO}_4$ . A review of patent and other literature is given. W. J. WRIGHT.

**Manufacture of lithium carbonate from the lepidolites of U.S.S.R.** E. S. BURKSER, I. S. SHTOKMAN, and A. M. BAUMAN (J. Chem. Ind. Russia, 1930, 6, 1436—1438).—Plans are described, and analyses of minerals are given. CHEMICAL ABSTRACTS.

**Arsenic determination in bismuth and barium salts.** L. W. GREEN and R. E. SCHOETZOW (J. Amer. Pharm. Assoc., 1930, 19, 1310).—Salts of barium and bismuth to which 2 pts. of arsenic ( $\text{As}_2\text{O}_3$ ) per million had been added, gave by the Gutzeit test stains indicating slightly more than 2 p.p.m.; this test was much more sensitive than the Bettendorf test, which gave negative results in the above cases. E. H. SHARPLES.

**Hydrolysis of arsenious iodide.** W. J. HUSA (J. Amer. Pharm. Assoc., 1930, 19, 1287—1291).—A dilute, aqueous solution of arsenious iodide is essentially a solution of arsenious acid and hydriodic acid in equilibrium with a small quantity of arsenious iodide (cf. Cocking, B., 1930, 348). In freshly prepared solutions equilibrium is reached within a few minutes (cf. B., 1930, 585). E. H. SHARPLES.

**Production of oxygen from lime and chlorine.** O. R. SWEENEY, J. W. HUSSEY, and W. RALSTON (Ind. Eng. Chem., 1930, 22, 716—718).—If dry bleaching powder is heated, 88% of the theoretical yield of oxygen is obtained. If chlorine is passed into hot milk of lime containing cobalt nitrate the yield is 99%. Oxygen is also produced by passing chlorine over lime at  $850^\circ$ . The second method, employing catalysts, was studied. The optimum temperature for cobalt was  $94^\circ$ , and cobalt and nickel were the most efficient catalysts tested. A design for a commercial plant, using a 0.001M-solution of nickel sulphate, is given and costs are discussed. Even in cases where chlorine is almost a waste product the process is not likely to compete with the usual method of manufacture of oxygen. It is, however, capable of small-scale operation, and might be employed in exceptional localities or perhaps in laboratories. C. IRWIN.

**Aluminium alloys.** STERNER-RAINER; RÖHRIG. **Corrosion of lead by sulphuric acid.** MAHN and WILHELM.—Sec. X.

## PATENTS.

**Neutralisation of acid waste waters.** A. M. HAGEMAN and W. L. SULLIVAN, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,765,424, 24.6.30. Appl., 25.4.29).—The liquors are diluted until they contain 0.3% of free sulphuric acid and then circulated through a bed of (a) granular dolomite to neutralise most of the acid, and (b) limestone or marble to neutralise the remainder. This procedure avoids the deposition of calcium sulphate in the neutralising tanks. A. R. POWELL.

**Production of (A) ammonia, (B) hydrogen-nitrogen mixture in the manufacture of synthetic ammonia.** L. C. JONES, Assr. to CHEM. ENG. CORP. (U.S.P. 1,765,534—5, 24.6.30. Appl., [A] 24.3.25, [B] 28.10.25).—(A) The gas mixture is treated with about 0.2% of ammonia prior to compression, whereby the aqueous condensate obtained on compression contains all the catalyst poisons present in the gases, *e.g.*, chlorine, sulphur, and phosphorus compounds. (B) A mixture of air, carbon monoxide from the manufacture of aluminium or calcium carbide, and steam is passed over iron (or chromium) oxide catalyst at 325–500°, sufficient steam being added to prevent the temperature exceeding 500°. The greater part of the resulting carbon dioxide is removed, and the residual gases are again mixed with steam and passed over a second similar catalyst. A. R. POWELL.

**Synthetic production of ammonia.** R. S. RICHARDSON, Assr. to CHEM. ENG. CORP. (U.S.P. 1,765,541, 24.6.30. Appl., 26.2.27).—The gas mixture from the converters is cooled to 30° and passed upwards through a tower packed with glass or porcelain rings and down which is circulated a stream of liquid ammonia at or below 0°. In this way the greater part of the ammonia in the gas mixture is condensed and the liquid ammonia, after cooling, can be used again. A. R. POWELL.

**Preparation of adsorptive [silica] gels.** H. N. HOLMES (U.S.P. 1,762,228, 10.6.30. Appl., 12.9.25).—A dilute solution of sodium silicate is treated with sufficient ferric chloride or sulphate solution completely to precipitate the silica. After keeping for 60 hrs., the gelatinous mass is filtered off and dried in a warm place until the moisture content falls to 35–70%; the lumps are roughly broken and preserved in an airtight vessel until syneresis causes them to sweat, then boiled with 9*N*-sulphuric acid until the iron is extracted, washed free from iron salts and acid, and dried. The product has a greater pore volume and higher adsorptive power than have gels produced in the usual way. A. R. POWELL.

**Production of titanic oxide.** M. SCHNETKA, Assr. to TITANIUM PIGMENT CO., INC. (U.S.P. 1,758,472, 13.5.30. Appl., 25.6.28. Ger., 6.9.27).—Hydrolysis of titanium sulphate solutions containing ferric sulphate is effected by boiling in the presence of a small quantity of hydrofluoric acid or a soluble fluoride. The precipitated titania is free from iron and more granular than that obtained by known methods. A. R. POWELL.

**Production of titanium dioxide.** W. MECKLENBURG (U.S.P. 1,758,528, 13.5.30. Appl., 1.2.29. Czechoslov., 5.5.28).—A suspension of titanium dioxide in sodium sulphate solution is prepared by treating

a solution of titanium sulphate at 80° with sodium hydroxide solution until the  $p_H$  of the solution is 4–4.5. A small quantity of this suspension is added to a boiling solution containing 20%  $TiO_2$  and 40–45%  $H_2SO_4$ , and the mixture is boiled for 3 hrs., whereby 95% of the titania present is precipitated as a dense white flocculent precipitate which settles and filters readily. A. R. POWELL.

**Production of oxides of titanium, thorium, or rare metals of the fourth group.** IMPERIAL CHEM. INDUSTRIES, LTD., and F. L. CLARK (B.P. 339,608, 7.9.29).—The ore, *e.g.*, ilmenite, is fused with alkali carbonate or hydroxide and the product ground and suspended in a 40% solution of sodium hydroxide. On electrolysis the solution at 90–120° under a pressure of 20 atm. of hydrogen, using insoluble anodes, the iron and other metallic impurities are removed and a relatively pure titania suspension remains. The process is also applicable to the purification of zirconia, hafnia, and thorina. A. R. POWELL.

**Manufacture of salt [from brine].** W. F. DOWNING, JUN., Assr. to DIAMOND CRYSTAL SALT CO. (U.S.P. 1,768,399, 24.6.30. Appl., 29.5.24).—Brine is evaporated until saturated with sodium chloride, then passed through a series of open-pan evaporators in which the salt is deposited. Part of the brine with suspended salt crystals is passed back from a late stage in the evaporation to an earlier stage so as to control the size of the crystals formed. A. R. POWELL.

**Recovery of borax from brine.** H. D. HELLMERS, Assr. to WEST END CHEM. CO. (U.S.P. 1,756,122, 29.4.30. Appl., 24.10.25).—Borax is recovered from Searles Lake brine by saturating the brine with carbon dioxide above 25°, removing the precipitated sodium hydrogen carbonate, and cooling the solution or mixing it with a proportion of the original brine. A. R. POWELL.

**Manufacture of alkali carbonates and bicarbonates and soluble magnesium salts.** H. E. POTTS. From Soc. ANON. ALCALINA (B.P. 339,330, 1.8.29).—A double carbonate of magnesium and potassium is prepared by causing nascent magnesium carbonate to react with a solution of a potassium salt, such as the chloride, saturated with carbon dioxide under a pressure equal to or greater than the critical pressure, and then to be decomposed by steam. Magnesium carbonate is precipitated and may be used again, and alkali carbonate passes into solution in the water of crystallisation. From the mother-liquors obtained in the preparation of the double carbonate magnesium chloride is recovered. W. J. WRIGHT.

**Recovery of sodium sulphite from spent sulphite [cellulose] liquors.** P. A. BARBOU, Assr. to Soc. BARBOU & CIE. (U.S.P. 1,765,560, 24.6.30. Appl., 12.1.28. Fr., 21.1.27).—The spent liquors from the digestion of straw with sodium sulphite solution are treated with sulphuric acid equivalent to the sodium present, then with calcium hydrogen sulphite equivalent to the sulphuric acid added, whereby a precipitate of calcium sulphate and lignin derivatives is obtained together with a solution of sodium hydrogen sulphite which is neutralised with sodium carbonate for re-use in the digestion process. A. R. POWELL.

**Manufacture of alkali sulphydrates [hydrogen sulphides].** H. P. BASSETT (U.S.P. 1,766,810, 24.6.30. Appl., 24.3.27).—Sodium sulphate is reduced with coke at 650–850° and the resulting mass is extracted with water. The filtered solution is evaporated to  $d$  1.4, whereby most of the unreduced sodium sulphide separates as the anhydrous salt, this being collected and returned to the first stage. The mother-liquor is diluted to  $d$  1.3 and allowed to cool to deposit sodium sulphide crystals; some of these are dissolved in water and converted into sodium hydrogen sulphide by treatment with the hydrogen sulphide evolved by treating the remaining crystals and the mother-liquor with sodium hydrogen sulphate (nitre cake). The sodium sulphate from the gas generator is recovered and used in the preparation of further quantities of sulphide.

A. R. POWELL.

**Manufacture of fertiliser salts [ammonium sulphate and phosphate].** H. B. KIPPER (U.S.P. 1,768,075, 24.6.30. Appl., 14.1.28).—A suspension of finely-ground calcium sulphate or calcium phosphate in hot water is beaten into a mist in a closed reaction vessel into which is passed a mixture of carbon dioxide, ammonia, and steam to convert the calcium into carbonate and leave a solution of ammonium sulphate or phosphate.

A. R. POWELL.

(A) Treatment of phosphate rock and the like.  
(B) Production of mixtures suitable for use as fertilisers.  
(C) Production of mixtures comprising ammonium nitrate and calcium carbonate. ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 339,340, Addn. B.P. 339,500, and B.P. 339,562, [A] 4.9.29, [B] 12.12.29, [C] 24.2.30. Nor., [A] 27.10.28, [B] 14.12.28, [C] 4.3.29).—(A) Phosphate rock is treated with nitric acid of 50% concentration in such amount and under conditions that, by suitable cooling, more than 80% of the lime is obtained as  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ , the phosphoric acid and the rest of the lime remaining in the mother-liquor. (B) The mother-liquor, obtained as described in (A), is precipitated with ammonia or ammonium liquor (20% N), and the dicalcium phosphate produced is mixed with calcium nitrate and urea, together with other nitrogen compounds and potassium salts, if desired. (C) A solution of calcium nitrate is treated with ammonia and carbonic acid, and the mixture of ammonium nitrate and calcium carbonate is obtained in a solid, granular form by evaporation, the temperature of the mass being maintained at 80–100° after the water content is reduced to about 5%.

W. J. WRIGHT.

**Manufacture of barium sulphate.** J. B. PIERCE, JUN. (U.S.P. 1,765,737, 24.6.30. Appl., 10.11.24).—A solution of barium sulphide is treated with the carbon dioxide produced by the interaction of sodium carbonate and sodium hydrogen sulphate, the precipitate of barium carbonate is removed, and the filtrate, containing barium hydrogen sulphide, is treated with the sodium sulphate solution from the carbon dioxide generator, whereby barium sulphate (blanc fixe) of good quality is precipitated, leaving a solution of sodium hydrogen sulphide which is evaporated to 30% for marketing.

A. R. POWELL.

**Preparation of by-product calcium sulphate for**

**plaster.** R. S. EDWARDS, ASSR. to RUMFORD CHEM. WORKS (U.S.P. 1,756,637, 29.4.30. Appl., 9.4.27).—Calcium sulphate obtained as a by-product in the treatment of phosphate rock and fluorspar with sulphuric acid may contain acid calcium phosphate and fluoride, which interfere with the setting of plaster made from it. This difficulty may be overcome by treating the calcium sulphate with hot dilute sulphuric or hydrochloric acid and neutralising the solution with milk of lime. The calcium sulphate thus obtained is neutral and contains calcium fluosilicate which acts as an accelerator in the setting of the plaster prepared from it. A. R. POWELL.

**Manufacture of cyanates and cyanamides.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 339,371, 13.9.29, 12.4. and 8.7.30).—A mixture of urea and an oxide or carbonate of a bivalent metal is heated to at least 130°, preferably in a current of a gas free from oxygen to remove the water formed, whereby cyanate is obtained, the temperature being then raised to 400–600° to produce cyanamide. Instead of urea, ammonia and carbon dioxide may be used, in which case the reaction with the metallic oxide or carbonate is carried out at a pressure not less than 50 atm., or a mixture of ammonium carbonate and carbamate may be employed.

W. J. WRIGHT.

**Preparation of silicious materials.** A. S. BEHRMAN, ASSR. to GEN. ZEOLITE CO. (U.S.P. 1,755,496, 22.4.30. Appl., 29.7.25).—A dilute solution of sodium silicate is treated with sufficient sulphuric acid to combine with the soda and produce a colloidal solution of silicic acid. Addition of ammonia to this solution results in the separation of a silica gel which may be dried for use as an adsorptive. Addition of aluminium sulphate and sodium carbonate to the colloidal acid solution precipitates sodium aluminium zeolite for use as a base-exchange substance. The aluminium salt may be replaced wholly or partly by salts of other metals, and ammonia may be used instead of soda as a precipitant, to produce gels containing iron, chromium, or other metals for use as catalysts. A. R. POWELL.

**Manufacture of a base-exchange material.** E. M. PARTRIDGE and E. W. SCARRITT, ASSRS. to PERMUTIT CO. (U.S.P. 1,766,350, 24.6.30. Appl., 5.1.26).—A solution of sodium silicate and trisodium phosphate is treated with a solution of aluminium sulphate and the resulting gel is dried at 95°. The dried mass is thrown into water and thereby caused to break up into small particles which can be readily washed free from soluble salts.

A. R. POWELL.

**Production of hydrogen [from carbon monoxide and steam].** J. S. BEEKLEY, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,756,934, 6.5.30. Appl., 19.12.27).—Steam is mixed with 33–50% of the quantity of water-gas with which it will react and the mixture, preheated to 420°, is passed over a catalyst. The issuing gases are mixed with a further quantity of water-gas to reduce the temperature again to 420° and once more passed over a catalyst. If desired, the process can be repeated a third time. It is claimed that the amount of methane formed and the quantity of carbon deposited in the catalyst chambers are reduced to very small proportions by working in this manner. A. R. POWELL.

**Manufacture of [solid] compounds of hydrogen peroxide.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,332, 6.8.29).—Solid substances capable of reacting with hydrogen peroxide are subjected to the action of gaseous mixtures poor in hydrogen peroxide so that no liquid is condensed on the substances. Stabilisers, such as starch, amines, ethyl alcohol, or neutral pyrophosphates, may be present. Gaseous mixtures produced by treating hydrogen-oxygen mixtures by means of the silent electric discharge or photochemically are suitable. W. J. WRIGHT.

**Production of nitrogen.** W. B. VAN ARSDEL, ASSR. to BROWN Co. (U.S.P. 1,765,781, 24.6.30. Appl., 10.11.21).—Purified air and hydrogen are passed in correct proportions through a magnesite-lined tube furnace packed with a porous refractory. Automatic means are provided for regulating the supplies of the two gases so that just sufficient hydrogen is supplied for the complete combustion of the oxygen in the air.

A. R. POWELL.

**Composition for the generation of chlorine.** G. J. HOWITZ, ASSR. to LEER. SEIDELL (U.S.P. 1,767,676, 24.6.30. Appl., 11.8.24).—A mixture of sodium chloride and chlorate with a solid acidic substance, e.g., citric acid, boric acid, or sodium hydrogen sulphate, is claimed. The components are used in powdered form, the chloride-chlorate mixture being packed separately from the acidic substance; for use the two powders are mixed dry, whereby a slow stream of chlorine is evolved suitable for disinfecting and medical purposes.

A. R. POWELL.

**Manufacture of carbon disulphide.** F. W. VOGEL, ASSR. to BROWN Co. (U.S.P. 1,765,809, 24.6.30. Appl., 22.12.24).—The gases resulting from the action of sulphur vapour on wood charcoal at 1000° are treated with sulphur dioxide in amount equivalent to their hydrogen sulphide content and cooled to 150°, whereby the resulting sulphur is condensed for further use in the process. The remaining gases are cooled to the ordinary temperature to condense the carbon disulphide.

A. R. POWELL.

**Production of hypochlorite compositions.** R. B. McMULLIN and M. C. TAYLOR, ASSRS. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,787,080 and 1,787,048, 30.12.30. Appl., [A] 6.3.30, [B] 16.8.28. Ger., [A] 11.5.29).—See B.P. 317,437; B., 1930, 711.

**Transforming barium and strontium sulphates.** F. ROTHE and H. BRENEK, ASSRS. to RHENANIA VER. CHEM. FABR. A.-G. (U.S.P. 1,772,269, 5.8.30. Appl., 3.11.25. Ger., 12.11.24).—See B.P. 242,996; B., 1926, 787.

**Preparation of pure alumina.** T. SUZUKI, H. TANAKA, and T. KURITA, ASSRS. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,785,464, 16.12.30. Appl., 6.7.27. Jap., 3.2.27).—See B.P. 284,661; B., 1928, 927.

**Detergents and their preparation.** C. A. JENSEN. From ELECTRIC SMELTING & ALUMINUM Co. (B.P. 339,355, 7.9.29).—See U.S.P. 1,745,844; B., 1930, 662.

**Hydrogen from hydrocarbons (B.P. 340,050). Sulphurous compounds from gases (B.P. 317,015).**

**Purification of gases (U.S.P. 1,765,869). Fuller's earth (U.S.P. 1,763,167). Coke-oven gas (B.P. 314,432).—See II. Straining cloths [for bauxite treatment] (B.P. 337,559).—See VI. Unoxidisable alloy (U.S.P. 1,771,773). Oxides (B.P. 339,645).—See X. Reflecting material (U.S.P. 1,767,285). Red lead (B.P. 340,082).—See XIII. Fertilisers (B.P. 316,583).—See XVI. Filter plugs (B.P. 339,761).—See XVIII.**

## VIII.—GLASS; CERAMICS.

### PATENTS.

**Manufacture of abrasive or refractory articles.** P. H. WALKER and S. S. KENYON, ASSRS. to CARBORUNDUM Co. (U.S.P. 1,785,102, 16.12.30. Appl., 1.5.28).—See B.P. 310,841; B., 1931, 65.

**Continuously drawing glass tubes or rods.** P. A. FAYRE (B.P. 319,361, 18.9.29. Belg., 22.9.28).

**Saggars, setters, or similar containers [in one or more parts] for use in the manufacture of ceramic wares.** G. W., J. F., W., and C. E. NAYLOR (B.P. 340,376, 18.11.29).

## IX.—BUILDING MATERIALS.

**Sparingly soluble cement from Portland cement and arsenic.** D. WERNER and S. GIERTZ-HEDSTRÖM (Tekn. Tidskr., 1930, 60, 41—49; Chem. Zentr., 1930, ii, 443).—A mixture of Portland cement (70%) and arsenious oxide (30%), after being heated at 200°, affords a hydraulic cement of normal setting time and of smaller solubility than ordinary cement.

A. A. ELDRIDGE.

**Manufacture of "Bitural" as a road builder.** C. F. BROADHEAD (Chem. Eng. Min. Rev. [Chem. Sec.], 1930, 23, 68—73).—Owing to the lower viscosity of continuous vertical-retort tar it is necessary to remove more of the light oils to obtain the same consistency as that of distilled horizontal tar. The higher temperature necessary results in a brittle pitch content of the resultant road tar. This and the presence of waxes (b.p. 280—300°) and of a higher content of unsaturated hydrocarbons are the causes of the inferiority of continuous vertical-retort road tar. A method for improving this type of tar to produce "Bitural" is outlined, in which 10 gals. of formalin and 5 gals. of ammonia (d 0.880) are intimately mixed with 1000 gals. of tar and digested at 71° for 8 hrs., whereby formaldehyde condensation products are formed *in situ* with the phenols, cresols, xylols, etc. Heated air is then passed over or through the tar until the desired consistency is obtained, the temperature of the tar being gradually raised during this period to 193°. The penetration test of a satisfactory product yields the value 220. The addition of 8% of asphaltic-base petroleum to the product is advantageous. A description of a large-scale plant incorporating two digester units of 2500 and 5000 gals. capacity, erected in Victoria, is given.

H. S. GARLICK.

**Heat transfer from gas stream to broken solids. FURNAS.**—See I. Pine wood. SOUM.—See V.

## PATENTS.

**Colouring of granulated slate, mineral granules, etc.** H. C. FISHER, Assr. to PHILIP CAREY MANUF. CO. (U.S.P. 1,766,891—2 and 1,766,814—5, 24.6.30. Appl., [A] 29.4.25, [B] 14.5.26, [C, D] 13.10.26).—(A) Granulated slate, free from dust, is mixed with a small quantity of sodium silicate solution containing in suspension a suitable mineral pigment and the coated particles are heated gradually in a rotating kiln until an adherent, glassy, coloured film is formed on the particles without causing them to stick together. (B) Slate is treated with a mixture of sulphuric acid and sodium dichromate to convert the mineral chlorite in the slate into ferric sulphate and gelatinous silica. The mass is then heated until the sulphate is converted into ferric oxide, whereby the particles are coloured red to brown. (C) The particles are coated with a film of sodium silicate, borax, and ferric oxide and heated until a reddish-brown fused glaze is formed on the surface. (D) The particles are coated with a slurry containing ferric oxide and fluxes such as litharge, silica, magnesia, borax, and sodium carbonate and the mass is heated to cause the flux to melt and fix the pigment on the surface of the particles.

A. R. POWELL.

**Manufacture of bituminous emulsions.** J. PARKER (B.P. 333,303, 14.5.29).—Tar macadam for surfacing roads etc. is prepared by heating 1—3 pts. of casein, 1—3 pts. of rosin, 0.15—0.45 pt. of sodium hydroxide, and 30—60 pts. of water at 100° for 30 min. to saponify the rosin, cooling to 95°, heating for 30 min. at this temperature with 5—10 pts. of crude naphtha under reflux, and stirring in slowly 60—120 pts. of coal tar, pitch, and/or bitumen, with or without a proportion of animal and/or vegetable oils. The resulting emulsion is stable down to -6° and may be diluted with water as required.

A. R. POWELL.

**Manufacture of bituminous road material.** F. O. W. LOOMIS and D. M. LOOMIS (U.S.P. 1,769,442, 1.7.30. Appl., 12.10.29. Can., 10.11.28).—A road-making aggregate is cleaned and dried by a stream of air, then mixed with a small amount of kerosene, and an aqueous emulsion of asphalt agitated violently with the mixture so that the emulsion is broken without heating and a film of bituminous material uniformly deposited.

R. H. GRIFFITH.

**Producing [printing of] veneer woods.** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 340,483, 20.9.29. Ger., 4.10.28).

**Furnace drum** (U.S.P. 1,768,964). Combined crusher-separator (U.S.P. 1,769,310).—See I. Distillation [products] of bituminous shales (B.P. 339,963). Tar for roads (B.P. 339,640).—See II. Calcium sulphate for plaster (U.S.P. 1,756,637). Reflecting material (U.S.P. 1,767,285).—See VII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Course of reduction of iron ore.** II. B. STÅLHANE and T. MALMBERG (Jernkontorets Annaler, 1930, 114, 1—26; Chem. Zentr., 1930, ii, 972).—The reduction proceeds by way of ferrosoferric and ferrous oxides to the metal, the end-product being a sponge of 95% purity.

The reaction  $\text{Fe}_3\text{O}_4 \rightarrow \text{FeO}$  is fairly rapid and somewhat retards the reaction  $\text{FeO} \rightarrow \text{Fe}$ . The reaction time depends on the speed of the reaction  $(\text{FeO})_n + n\text{CO} \rightarrow n\text{Fe} + n\text{CO}_2$  in the reaction zone and the speed with which the carbon dioxide so formed can diffuse away.

A. A. ELDRIDGE.

**Manufacture and heat treatment of sheet [iron and steel] for the automobile industry.** A. T. KATHNER and P. DAMIRON (Rev. Mét., 1930, 27, 615—629).—An account is given of modern mass-production methods of manufacturing and heat-treating steel sheet for motor bodies.

A. R. POWELL.

**Hardening of steel.** H. ESSER and W. EILENDER (Arch. Eisenhüttenw., 1930—1, 4, 113—144; Stahl u. Eisen, 1930, 50, 1616—1617).—Annealing and rapid air-quenching experiments on thin sheets of steel have afforded evidence that martensite is a heterogeneous mixture of  $\alpha$ -iron and cementite and not a solid solution, and that all iron-carbon alloys, even those with a very small carbon content, are capable of being hardened provided that they are quenched extremely rapidly from a suitable annealing temperature. The authors conclude that the hardening of steel by quenching is controlled by the degree of dispersion of the precipitated iron carbide and by the considerable distortion of the  $\alpha$ -iron lattice produced by the separation of carbide in the  $\gamma \rightarrow \alpha$  transformation at a temperature at which the plasticity of the metal is very low. This theory is shown to be in accordance with the phenomena observed during the reheating of quenched steels, and also accounts for the changes which take place in the physical properties of the metal during heat treatment.

A. R. POWELL.

**Iron-aluminium-carbon alloys.** O. VON KEIL and O. JUNGWIRTH (Arch. Eisenhüttenw., 1930—1, 4, 221—224; Stahl u. Eisen, 1930, 50, 1643).—The effect of aluminium on the iron-carbon system has been investigated. The eutectic composition is reduced by 0.16% C by the addition of 1% Al, the pearlite transformation point is slightly reduced by addition of aluminium up to 3.5%, but with 3.5—8% Al it is raised again, and with more than 8% Al the intensity of this transformation is infinitely small, and in alloys with a high carbon content the  $\gamma$ -field ceases to exist. With hypoeutectic alloys containing less than 5% Al the finely granular distribution of the graphite, which corresponds with metastable freezing and spontaneous decomposition, is rendered more prominent; with 5—9.5% Al the edges of the primary solid solution crystals show a decreased tendency to decompose, and these zones become more readily soluble in acids, and with more than 9.6% Al the solid solution becomes stable and readily soluble in acids. Addition of aluminium to iron with a high carbon content results in complete separation of the carbon as graphite with 2.3% Al, complete disappearance of the graphite with 11% Al, and complete precipitation again with 18% Al.

A. R. POWELL.

**Determination of carbon in high-sulphur steels by direct combustion.** H. A. BRIGHT and G. E. F. LUNDELL (Bur. Stand. J. Res., 1930, 5, 943—949).—The recommended combustion train is as follows: The porcelain combustion tube is 48 cm. long and 2.5 cm.

internal diam. tapering to 1 cm. for the last 14 cm. of length, and finally to about 6 mm. for a short length. Beyond the combustion boat the tube contains only the usual oxidised copper gauze in the zone which is heated at 700–800°. The exit gases pass consecutively through 50% chromic acid solution, asbestos coated with iron oxide, 97% sulphuric acid, more asbestos, 20-mesh anhydrous, and a tube packed with ascarite and anhydrous. The copper gauze becomes coated with copper sulphate and ferric oxide and requires occasional renewal.

A. R. POWELL.

**Influence of high temperatures and of addition of salts on the equilibrium in solution between calcium carbonate and bicarbonate, and the lime-rust-protective coating [on iron].** J. TILLMANS, P. HIRSCH and W. R. HECKMANN (Gas- u. Wasserfach, 1931, 74, 1–9).—The equilibrium between calcium carbonate and bicarbonate has been investigated at temperatures from 40° to 100° by removing carbon dioxide from a saturated calcium bicarbonate solution until equilibrium is attained and determining the free and combined carbon dioxide content of the solution. The concentrations being expressed in mg./litre, the equilibrium constant  $K = [\text{HCO}_3]/[\text{H}_2\text{CO}_3]$  falls from  $2.05 \times 10^4$  at 40° to  $3.976 \times 10^3$  at 100°, and at any temperature,  $t$ , is given by  $K_t = 66,300 \times (0.9714)^t$ . Sodium salts, and calcium salts at concentrations below 60 mg./litre, do not influence the equilibrium; calcium salts at high concentrations produce a neutral salt effect. Addition of magnesium chloride markedly reduces the free carbon dioxide content of the solutions. Study of the action of water containing calcium bicarbonate and oxygen on iron, with the formation of a protective coating at temperatures up to 100°, shows that although at the higher temperatures visible corrosion appears to take place more rapidly, the time needed for the formation of the protective layer remains unchanged. It has been shown experimentally that dissolved oxygen is essential for the formation of the layer, as in its absence the coating of ferric hydroxide, upon which carbon dioxide and calcium carbonate are adsorbed, is not produced. The significance of the results for hot-water engineering is indicated.

H. F. GILLBE.

**Mechanism of corrosion of copper-steel.** C. CARIUS (Z. Metallk., 1930, 22, 337–341).—During the rusting of copper-steel (0.25–1.1% Cu) in the air or in distilled water the metal becomes covered with an adherent film of metallic copper the surface of which is converted into cupric oxide. Under sea-water four layers of corrosion products are produced: the lowest consists of spongy copper, above this is a thicker layer of a greenish mixture of ferrous and ferric hydroxide, then follows a thin black film of hydrated ferrosiferrous oxide, and finally there is a brownish-red, thick, outer layer of the hydroxide,  $\text{FeO}(\text{OH})$ . The formation of the copper layer in both cases is shown to be a secondary reaction, both metals first going into solution and the copper then being reduced to metal by the unchanged steel with the formation of various oxidation products of iron; the deposited copper and the unchanged steel form a local element with a relatively high *E.M.F.* and hence the rusting of the steel is accelerated. A

complete electrochemical explanation of the formation of the various corrosion layers is given.

A. R. POWELL.

**Corrosion of copper. I. Behaviour towards chemical agents. II. Electrochemical behaviour and formation of a protective film.** L. W. HAASE (Metall-Wirt., 1930, 9, 503–506; Chem. Zentr., 1930, ii, 797–798).—Corrosion by steam and gaseous sulphur dioxide, hydrogen chloride, fluoride, bromide, or iodide, and carbon dioxide; by neutral salts (ammonium chloride in particular); by carbonates; by magnesium salts of sea-water; and by acids is discussed. With the exception of sulphurous acid, dilute acids corrode copper only in presence of oxygen. Alkali hydroxide solutions corrode little, calcium hydroxide not at all, and ammonia solution only in presence of oxygen. Liquid hydrocarbons dissolve superficial oxide; vegetable oils are more harmful than saturated aliphatic compounds.

A. A. ELDRIDGE.

**Methods of corrosion testing.** E. K. O. SCHMIDT (Z. Metallk., 1930, 22, 328–333).—The methods used at the German Aeronautical Research Institute in testing the behaviour of light metals and alloys in corrosive media are described and illustrated. The apparatus includes mechanical devices for the alternate wetting and drying test in sea-water, for exposing the specimens to circulating streams of sea-water, and for carrying out the salt-spray test. The decrease in elongation and tensile strength with progressive corrosion is shown to be a satisfactory method of comparing the behaviour of metals under corrosive conditions. Characteristic curves for various aluminium alloys, showing the percentage reduction in various mechanical properties when subjected to the action of sea-water under different conditions, are reproduced.

A. R. POWELL.

**Use of aluminium [in chemical laboratories].** P. DROSSBACH (Hausz. V.A.W. Erftw. Aluminium, 1930, 2, 39–44; Chem. Zentr., 1930, ii, 976).—The influence of oxygen on the corrosion of aluminium (always anodic) was investigated. The amount of corrosion depends on the nature of the cathode in the local element.

A. A. ELDRIDGE.

**Effect of soda and soap solutions on aluminium.** H. BORNER (Hausz. V.A.W. Erftw. Aluminium, 1930, 2, 48–49; Chem. Zentr., 1930, ii, 977).—The corrodibility of aluminium and its alloys, alone or in presence of copper, brass, tin, zinc, iron, or magnesium, by alkaline soap solutions at 20° or 50–60°, has been measured. In general, the corrodibility is in the order of the potential series, but with sodium carbonate solution zinc is an exception, having a protective action on aluminium.

A. A. ELDRIDGE.

**Resistance of aluminium to fatty acids at 80–100°.** W. CLAUS (Hausz. V.A.W. Erftw. Aluminium, 1930, 2, 44–47; Chem. Zentr., 1930, ii, 977).—Under the working conditions employed aluminium was almost completely resistant to the action of solid and liquid fatty acids.

A. A. ELDRIDGE.

**Behaviour of aluminium, copper, zinc, and iron foil in contact with moist lithopone.** ANON. (Hausz. V.A.W. Erftw. Aluminium, 1930, 2, 52; Chem. Zentr.,

1930, ii, 977; also *Farben-Chem.*, 1931, 2, 88).—Aluminium is least affected. A. A. ELDRIDGE.

**Annealing and quenching of duralumin.** MATTER (Rev. Mét., 1930, 27, 560—562).—Air-quenching of duralumin sheets not thicker than 2 mm. followed by ageing at the ordinary temperature produces the same mechanical properties as does quenching in water. With increasing thickness, however, the tensile strength of aged air-quenched sheets becomes progressively lower. Further tests have shown that the critical rate of cooling for obtaining a tensile strength above 30 kg./mm.<sup>2</sup> after ageing is about 150°/hr. measured at 400°. Quenching from 500° in a salt bath at 200° produces metal equally as good as that obtained by quenching in water.

A. R. POWELL.

**Effect of heat treatment on aluminium and its light alloys.** J. SUHR (Rev. Mét., 1930, 27, 563—569).—Examples are given of the variation of the physical and mechanical properties of aluminium and some of its alloys with the heat treatment to which they have been subjected.

A. R. POWELL.

**Flowability of the light alloys of aluminium: influence of chemical composition.** A. COURT (Compt. rend., 1930, 191, 1128—1130).—By the method (cf. B., 1930, 563) described previously (temperature of mould 15°, of pouring 750°), the flowability of alloys of aluminium with (a) silicon (0—21.72%), (b) copper (0—22.05%), and (c) zinc (0—30%) has been determined. Both (a) and (b) show a sharp drop to about 2% of addition, and then a steady rise, the flowability for 6—7% being about the same as for aluminium itself; after 18% it remains nearly constant at some 60% above that of aluminium; (b) is throughout slightly lower than (a). Alloy (c) shows a slow but steady drop to little more than half that of aluminium for 12% Zn, followed by a similar rise to 30% Zn, for which it is still slightly below the initial figure. C. A. SILBERRAD.

**Electrical conductivity and tensile properties of light magnesium-aluminium alloys as affected by atmospheric exposure.** E. WILSON (J. Inst. Electr. Eng., 1930, 69, 89—94).—Comparative atmospheric corrosion tests have been made on stressed conductor wires of high-purity aluminium, aldreys, duralumin, and a 0.75% nickel-aluminium alloy. The change in resistance with time was least with the nickel alloy and greatest with duralumin; after two years the ratio  $R_0/R_t$  was 0.96 for the nickel alloy, 0.93 for aluminium, 0.90 for aldreys, and 0.85 for duralumin. Inter-crystalline corrosion in the duralumin was very marked, but occurred to only a slight extent in aldreys and not at all in the nickel alloy or aluminium. The loss in weight and the increase in sp. resistance were the same for the nickel alloy after exposure for 24 yrs. as for aldreys after exposure for 1.5 yrs. All four conductors showed an average loss of 7.5% in the breaking load after 9 months, but there was a tendency for the more tightly stressed wires to suffer a smaller decrease in strength than that of similar wire under smaller stresses. The most marked deterioration caused by corrosion was in the elongation, especially in the case of duralumin, which after 8 yrs. failed to show any elongation in the tensile test.

A. R. POWELL.

**Corrosion and protection of aluminium rolling alloys in aircraft construction.** P. BRENNER (Z. Metallk., 1930, 22, 348—355).—The various phenomena, such as local pitting, intercrystalline corrosion, etc., associated with the corrosion of duralumin and alutal in sea-water are discussed and illustrated with relation to their bearing on the use of aluminium alloys in aircraft construction. The causes of intercrystalline corrosion in duralumin are too low a quenching temperature, inefficient quenching (e.g., in oil or hot water), and too high an ageing temperature, all of which leave the copper in such a condition as to produce local elements. The best resistance to corrosion is obtained by quenching in cold water from above 500° followed by ageing at the ordinary temperature, and comparative tests with various aluminium alloys have shown that duralumin treated in this way behaves better than any of the other commercial aluminium alloys on exposure to sea water or atmospheric corrosion. Comparative tests on a new German and a new American alloy of the duralumin type are shown graphically. "KS sea-water" alloy in the hardened state has too low an elongation, and in the annealed state too low a tensile strength for aircraft work, but is definitely more resistant to corrosion than duralumin. The value of various protective coatings—paints, varnishes, electrolytically and chemically produced films, and pure aluminium—on hard aluminium alloys is briefly discussed.

A. R. POWELL.

**Corrosion of aluminium casting alloys.** R. STERNER-RAINER (Z. Metallk., 1930, 22, 357—361).—The reduction in tensile strength and elongation of pure aluminium, commercial aluminium, and aluminium with small quantities of manganese, titanium, antimony, magnesium, iron, silicon, magnesium silicide,  $MgZn_2$ , cadmium, bismuth, zinc, or copper has been determined after 1—10 days' immersion in 3% sodium chloride solution. Of the alloying constituents tested, copper and  $MgZn_2$  have the greatest accelerating effect on the corrosion of commercial aluminium, whereas magnesium up to 1%, antimony up to 2%, bismuth up to 2%, and  $Mg_2Si$  up to 2.5% appear to improve the resistance of commercial aluminium to corrosion. The behaviour of typical aluminium casting alloys during prolonged exposure to sea water has confirmed the superiority of "KS sea-water" alloy; apparently this alloy develops a highly resistant protective film in the early stages of exposure. A table showing the behaviour of pure aluminium, KS alloy, silumin, and the German and American light alloys towards numerous salt solutions, organic acids, and various foods is included.

A. R. POWELL.

**Micrographical researches on the corrosion of aluminium and its alloys.** H. RÖHRIG (Z. Metallk., 1930, 22, 362—364).—Microscopical examination of polished sheets of aluminium and various light aluminium alloys after immersion in 50% nitric acid, 0.1N-hydrochloric acid, 0.1N-sodium hydroxide, and 3% sodium chloride solution containing 1% of hydrogen peroxide has shown that corrosion starts in the ground mass in the vicinity of a particle of a cathodic constituent and spreads either along the grain boundaries or concentrically outwards over the surface according to the nature of the corroding medium. Annealing tests on



99.8% aluminium show that the number and magnitude of the etching pits which form after immersion for 1 min. in aqua regia decrease rapidly with the temperature of annealing, owing to the impurities present entering into solid solution. A. R. POWELL.

**Effect of physical state of small amounts of copper on rate of corrosion of lead by sulphuric acid.** E. G. MAHIN and E. J. WILHELM (Ind. Eng. Chem., 1930, 22, 1397—1404).—The amount of copper retained in solution in lead-copper alloys is less than 0.0025%; alloys which have been quenched in water retain 0.065—0.08% in solid solution. Copper is precipitated if the alloy is annealed for 50 hrs. at 280°, but annealing at 100° for 500 hrs. precipitates copper only when its content reaches 0.06%. Rapid cooling increases the corrosion of lead by sulphuric acid owing to induced stresses, but the addition of up to 1% of copper reduces the attack, provided the alloy is quenched. When lead is to be subjected to annealing, the amount of copper present should not exceed 0.2%, as above this amount copper crystals separate and cause electrolytic action, whereby the protective coating of lead sulphate becomes ruptured. W. J. WRIGHT.

**Corrosion of lead and its importance for the general knowledge of corrosion.** M. WERNER (Z. Metallk., 1930, 22, 342—346).—Pure electrolytic lead melted *in vacuo* has a relatively fine-grained structure and an extraordinarily high resistance to corrosion by sulphuric acid and hydrochloric acid. Addition of small quantities of other metals may cause grain refinement or grain growth together with increased or diminished resistance to corrosion, but there is no relation between grain size and resistance to corrosion as stated by Brenthel (B., 1930, 286). Lead, which is rapidly attacked by sulphuric acid at 280°, is completely resistant thereto after addition of 0.1% Pt, but addition of a small quantity of bismuth renders the metal more readily soluble in sulphuric acid in spite of its action in refining the grain size. The presence of small quantities of elements more noble than, and insoluble in, lead may increase its resistance to corrosion by producing passive surface layers. On the other hand, elements which form a eutectic with lead tend to decrease its resistance to corrosion, as also does the presence of lead oxide.

A. R. POWELL.

**Physical and radiative properties of tantalum carbide [filaments].** K. BECKER and H. EWEST (Z. tech. Physik, 1930, 11, 148—150, 216—220; Chem. Zentr., 1930, ii, 597).—Tantalum carbide filaments are prepared by carburisation of tantalum wire in an atmosphere containing carbon. Such filaments are stable up to about 3600° Abs. in a vacuum or in an argon (85%)–nitrogen mixture. Decarburisation is effected at high temperatures by hydrogen or tungsten; the carbide is attacked by oxygen and aqueous vapour with formation of oxide. The carbide has m.p. 4150° Abs., and coefficient of thermal expansion  $8.2 \times 10^{-6}$ . The specific electrical resistance at 20° is  $1.02 \times 10^{-4}$  ohm per cm. The electron emission is smaller than with tantalum, and the total radiation greater than with tungsten. A. A. ELDRIDGE.

**Behaviour of metals towards disinfectant and**

**cleansing media.** W. GUERTLER, T. LIEPUS, MOHR, and OSTERBURG (Metall-Wirt., 1930, 9, 447—449; Chem. Zentr., 1930, ii, 798).—The behaviour of metals and alloys towards various disinfectants at optimal concentration is recorded. A. A. ELDRIDGE.

**Effect of carbon tetrachloride, trichloroethylene, and tetrachloroethane on metals.** J. FORMÁNEK (Chem. Obzor, 1930, 5, 57—59; Chem. Zentr., 1930, ii, 976).—The corrosion of copper, brass, nickel, tin, lead, aluminium, zinc, iron, and iron coated with ferrosferic oxide is negligibly small. Trichloroethylene and tetrachloroethane attack copper, brass, tin, iron, and iron coated with ferrosferic oxide more markedly, but still to a relatively small extent. A. A. ELDRIDGE.

**Influence of high-frequency oscillations on the treatment of metallurgical products.** G. MAHOUX (Compt. rend., 1930, 191, 1328—1330).—Round bars of austenitic and nickel-chromium-molybdenum steels were subjected to the action of high-frequency oscillations and heated, simultaneously, for 10 hrs. at 500° in a current of ammonia. Comparison with test pieces treated similarly, but with the omission of the discharge, showed considerable increases in the Brinell-Vickers hardnesses of the surface and interior, in the magnetic susceptibility, and in the rate of nitridation, and a small increase in resilience. Both the penetration of an electrolytic deposit of chromium into a soft steel, and the diffusion of carbon from the centre to the outside of an iron casting heated for 9 hrs. at 530° were accelerated by the action of the discharge (cf. following abstract). J. GRANT.

**[Influence of high-frequency oscillations on the treatment of metallurgical products.]** L. GUILLET (Compt. rend., 1930, 191, 1331—1332).—The experiments of Mahoux (preceding abstract) have been confirmed. The possibilities of the process are indicated. J. GRANT.

**Effect of surface conditions and electrodeposited metals on the resistance of materials to repeated stresses.** R. H. D. BARKLIE and H. J. DAVIES (Proc. Inst. Mech. Eng., 1930, 731—750).—The fatigue limit of steel is seriously reduced by electroplating it with nickel, owing to the formation of a crack in the nickel layer under the combined influence of its own tensile stress and of the stress applied in the test and the resulting concentration of stress at the base of this crack. This effect of nickel-plating exceeds that produced by a circular groove in the test piece of equal depth to the depth of the crack as the internal stress in the nickel tends to widen the crack. The difference between the effects of the groove and the crack decreases with decreasing thickness of the deposit and depth of groove. The deleterious effects of nickel-plating may be avoided by depositing the nickel at 50°, using a superposed alternating current, and treating the iron anodically in 10% sulphuric acid prior to plating. Copper plate free from internal stress reduces the fatigue limit of steel as the deposit cracks at a lower range of stress than the steel; zinc and lead deposits, on the other hand, have no effect on the fatigue limit of steel, and the deposition of a very thin layer of lead, e.g.,

$1 \times 10^{-7}$  in. thick, is sufficient to insulate the steel from the effect of stressed nickel deposits. A. R. POWELL.

**Contamination of solutions (in cleaning and electroplating).** F. J. LISCOMB (Metal Ind., N.Y., 1930, 28, 427–429).—A discussion of the introduction of iron into alkaline cleaning and electroplating solutions, which should be free from chlorides and used in rubber-lined tanks. Sodium ferrocyanide does not interfere with anodic corrosion in acid copper solutions except when the free cyanide is low and the current density high. CHEMICAL ABSTRACTS.

**Determination of cathodic yield in chromium-plating baths.** O. MACCHIA (Ind. chim., 1930, 5, 561–565; Chem. Zentr., 1930, ii, 975).—The chromium is deposited on copper or nickel-plated copper strips which have previously been immersed for 1 min. in boiling 10% potassium cyanide solution, dried with filter paper, then dried for 5–6 min. at 50–60° and weighed. With the nickel-plated strips the deposit of chromium is dissolved anodically in 10% sodium hydroxide solution against a lead cathode.

A. A. ELDRIDGE.

**Plating of rolled zinc and zinc-base die-castings.** E. A. ANDERSON and C. E. REINHARD (N.J. Zinc Co. Res. Bull., 1929, Nov., 12 pp.).—Successful methods, together with modifications, are described.

CHEMICAL ABSTRACTS.

**Electrodeposition of lead on metals.** N. A. ISGARISCHEV and N. N. IVANOV (Min. Suir. Tzvet. Met., 1929, 4, 1048–1052).—White, finely crystalline deposits of lead were obtained on copper or iron from a bath containing lead acetate (25 g.), acetic acid (80%, 32 g.), glue (1 g.), and pyridine (2 c.c.) in water (1 litre); the current density could be increased to 0.33 amp./in.<sup>2</sup>

CHEMICAL ABSTRACTS.

**Mercury for power production.** SAMUELSON. **Heat transfer from gas stream to broken solids.** FURNAS.—See I. **Gas-cleaning plant.** LÉVÊQUE.—See XI.

PATENTS.

**Furnaces for the heat treatment of metals [iron] etc.** A. SMALLWOOD and J. FALLON (B.P. 337,362, 25.6.29).—The furnace comprises a muffle chamber for heating the billets for rolling and one or more secondary chambers heated by waste gases from the muffle and adapted for the heat treatment of the sheets produced in the rolling of the ingots.

A. R. POWELL.

**Slag-reducing process [for steelworks' slag].** T. F. BALLY and L. G. PRITZ (U.S.P. 1,754,845, 15.4.30. Appl., 28.1.28).—Basic and acid slags from steel furnaces are reduced with carbon in an electric arc furnace to yield ferromanganese containing silicon and phosphorus and a slag containing lime, alumina, and silica; after addition of scrap iron this slag is heated further with more carbon to produce ferrosilicon and a highly basic aluminatous slag which can be used again in the steel furnaces.

A. R. POWELL.

**Reduction of metal oxides [iron ore] without melting.** W. H. SMITH (U.S.P. 1,759,173, 20.5.30. Appl., 10.5.28).—A mixture of iron ore and carbon is passed downwards through a long vertical retort divided

into four zones: a preheating zone at the top followed by a reducing zone, a nodulising zone, and finally a cooling zone at the bottom. The first three are electrically heated at 425°, 870°, and 1040°, respectively, and provision is made for withdrawing evolved gases at points between succeeding zones. A stream of carbon monoxide is passed upwards from the bottom of the cooling zone to drive out carbon dioxide and steam therefrom and thus prevent re-oxidation of the reduced iron sponge.

A. R. POWELL.

**Manufacture of steel having good machining properties.** F. BORGGRAFE (B.P. 318,177 and 339,946, [A] 28.8.29, [B] 12.9.29. Ger., [A] 29.8.28, [B] 22.12.28).—(A) Scrap steel or iron is melted in a cupola with silicon-iron, ferrosilicon, or hæmatite pig iron low in phosphorus, and the resulting metal is mixed with Thomas pig iron so that the mixture contains about 0.25% P. The product is refined in a small acid-lined Bessemer converter until the required carbon content (0.04–0.10%) is obtained, deoxidised with ferromanganese, and cast into ingots which contain 0.2–0.5% P, 0.3–0.6% Mn, 0.04–0.1% C, 0.05–0.15% S, and 0.1–0.25% Si. (B) The refining is effected in an acid-lined open-hearth furnace.

A. R. POWELL.

**Removal of the last traces of oxygen from iron alloys.** A. GLAZUNOV (B.P. 317,493, 17.8.29. Czechoslov., 18.8.28).—The metal is deoxidised with an alloy of lead containing sodium and calcium or magnesium. The deoxidiser is introduced into the iron bath in a perforated sheet-iron cage of such a weight that the cage and alloy are sufficiently heavy to sink through the slag into the bath before the alloy melts.

A. R. POWELL.

**Heat treatment of manganese steel.** H. WADE. From TAYLOR-WHARTON IRON & STEEL Co. (B.P. 339,521, 31.12.29).—The grain structure of austenitic manganese steel is refined by annealing at 450–525° for 72 hrs. to cause partial transformation of the austenite, cooling slowly to 20°, reheating at 1060° to reconvert the martensite etc. into austenite, and quenching in cold water.

A. R. POWELL.

**Production of magnetic materials [heat treatment of permalloy].** P. S. MCCANN, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,765,436, 24.6.30. Appl., 6.11.24).—Permalloy tape is annealed at 1200°, cooled slowly to 800°, quenched, and reduced in thickness at least 50% by rolling.

A. R. POWELL.

**Refining and working of copper.** H. H. STOUT (B.P. 317,400, 12.8.29. U.S., 15.8.28).—Broken copper cathodes, clean scrap, or copper shot are pressed into briquettes which are heated below the m.p. in an atmosphere of hydrogen or carbon monoxide to effect reduction of the surface oxide films and removal of dissolved oxygen, then subjected to pressure at the same temperature to consolidate the briquettes into a homogeneous mass which is finally extruded into rods.

A. R. POWELL.

**Unoxidisable [manganese-copper-nickel] alloy.** C. CONTAL (U.S.P. 1,771,773, 29.7.30. Appl., 17.7.29. Fr., 21.6.29).—The alloy comprises 40 (40–50)% Ni, 31 (30–40)% Cu, 18 (10–20)% Mn, 9 (5–15)% Sb, and 2 (1–10)% Sn. It has  $d$  8.28, m.p. about 1000°.

a white colour and homogeneous microstructure, and is highly resistant to the action of sulphuric and nitric acids, steam, ammonia, and caustic alkalis.

A. R. POWELL.

#### Manufacture of porous metals or metal oxides.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,645, 5.9.29).—The metal oxide is made into a stiff foam with an organic reagent, the foam is dried at 100°, and the resulting porous mass heated at a suitable temperature in a reducing atmosphere to produce a porous sinter of the metal. *E.g.*, 120 pts. of cupric oxide are beaten up with a foam made from 100 pts. of a 2% solution of sodium diisopropyl-naphthalenesulphonate and the dried foam is heated at 900–1000° in hydrogen. The copper oxide may be replaced by the oxides of silver, iron, lead, or nickel, or by powdered chromium, molybdenum, aluminium, or silicon, and the sulphonate by other similar compounds or by saponin, with or without binders such as glycol, glycerol, or glue. The products are suitable for use as catalysts.

A. R. POWELL.

**Aluminium alloy.** H. J. GARRARD (B.P. 338,923, 30.5.29).—A hardener alloy containing 37% Al, 17% Mg, and 46% Zn is prepared and cast into ingots. Pure aluminium is then melted in an electric furnace and 3–20% of the hardener added, followed by 0.5–5% Bi. The resulting castings have an ultimate strength of 15–18 tons/in.<sup>2</sup>, which may be increased to 30 tons/in.<sup>2</sup> by cold-work.

A. R. POWELL.

**Production of ingots from metals of high crystallising speed, *e.g.*, aluminium and its alloys.** H. RÖHRIG (B.P. 339,624, 8.7.29).—The metal is cast into covered moulds the bottoms of which consist of a metal having a high heat conductivity and the sides and covers of a heat-insulating material into which may be embedded heating means so that cooling takes place only at the bottoms of the ingots. [Stat. ref.]

A. R. POWELL.

**Increasing the resistance to corrosion of aluminium alloys.** DURENER METALLWERKE A.-G. (B.P. 318,999, 16.8.29. Ger., 14.9.28).—Duralumin containing 0.25–1% Mg, 3.5–5% Cu, and 0.2–1% Mn is coated on one or both sides with a thin layer of another duralumin alloy, containing, *e.g.*, 0.2–1% Mg and 0.2–2% Mn, but no copper, and the combined alloy is heat-treated in the usual way.

A. R. POWELL.

**Increasing the radiation constant of or heat-absorbing and -emitting capacity of metallic surfaces. [Providing electron-metal engine cylinders with an oxidised aluminium coating.]** W. W. TRIGGS. From VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 339,722, 11.10.29).—Cylinders of electron metal for use in a combustion engine are provided with a thin coating of aluminium or an aluminium alloy and the surface of the coating is treated in known manner to produce thereon an adherent film of alumina. [Stat. ref.]

A. R. POWELL.

**Blast-roasting or sintering of [zinc] ores and metallurgical materials.** NAT. PROCESSES, LTD., and T. B. GYLES (B.P. 338,886, 27.8.29).—Furnace gases with a low content of sulphur dioxide (*e.g.*, 0.5%) are passed through a heap of zinc oxide sinter until the

sulphur content of the latter rises to 7–8%; 3 pts. of this material mixed with 1 pt. of raw blende are then roasted on a Dwight-Lloyd sintering grate, whereby a good sinter containing only 0.2% S should be obtained. Alternatively the gases may be passed through a mixture of raw and partly calcined blende and the product blast-roasted as before.

A. R. POWELL.

**Zinc-base die-casting alloy.** NEW JERSEY ZINC Co., Assees. of E. A. ANDERSON (B.P. 340,104, 20.11.29. U.S., 5.6.29).—The alloy contains 2–10% Al and 0.1–0.3% Mg; the zinc used contains more than 99.99% Zn, < 0.003% Pb, < 0.003% Cd, and < 0.001% Fe. This alloy is not subject to growth or intercrystalline oxidation.

A. R. POWELL.

**Simultaneous reduction of [nickel] ores and conversion of carbonaceous materials into hydrocarbons of low b.p.** J. L. FOHLEN (B.P. 339,276, 25.7.29).—The gases from the destructive distillation of lignite are passed through a heated mass of garnierite and calcium sulphide and the resulting nickel matte is separated from the slag, crushed, roasted to oxide, and reduced at 150–300° by means of the vapour of the liquid products obtained in the lignite distillation, whereby nickel powder and cracked hydrocarbon products of low b.p. are obtained. The combustible gases obtained in the process are utilised in heating the retorts and the rotary furnace in which the matte is produced.

A. R. POWELL.

**[Silver-copper-nickel] alloy.** M. E. BARKER, Assr. to H. H. SEMMES (U.S.P. 1,757,507, 6.5.30. Appl., 5.9.28).—An alloy of 55–65% Ni, 27–37% Cu, and 3–13% Ag is claimed. The preferred composition is 60% Ni, 32% Cu, and 8% Ag. The colour resembles that of silver, and the alloy is not tarnished by exposure to acid foods or by the air.

A. R. POWELL.

(A) **Manufacture of metallic [copper] sheet, strip, or wire.** (B, c) **Electrodeposition of metals.** S. O. COWPER-COLES (B.P. 338,173, 338,487, and 338,540, [A] 16.5.29 and 17.1.30, [B] 15.5., 28.11., 27.12., and 31.12.29, and 7. and 17.1.30, [C] 15.5.29).—(A) The metal is deposited on a slowly rotating disc-cathode and is automatically stripped therefrom in the form of a sheet or strip which is thickened by a further electroplating process using the same or one or more other metals. During the thickening operation the original sheet or strip is supported on a glass plate and subjected to the smoothing action of rollers which are free to move to a limited extent in the direction of travel of the sheet. (B) The metal is deposited on a mandrel which is revolved at such a speed that fresh electrolyte is continuously brought into contact with it. By means of loosely mounted rotating discs the deposit is kept smooth, and means are provided for stripping the mandrel of the deposit in the form of a cylinder or as a helical spiral strip. (C) The mandrel consists of brass or copper the surface of which has been oxidised or sulphidised and then coated with a film of graphite to allow of ready removal of the deposit.

A. R. POWELL.

**Electrolytic production of alloys of calcium or strontium with lead.** METALLGES. A.-G. (B.P. 318,599, 6.9.29. Ger., 6.9.28).—Calcium or strontium chloride

is fused with 20—40% of its weight of sodium chloride or, preferably, with 60—80% of its weight of potassium chloride, and the bath is electrolysed at 650—700°, using a lead cathode and a carbon anode with a current density not exceeding 4 amp./cm.<sup>2</sup> of anode area.

A. R. POWELL.

**Apparatus for high-temperature treatment of ores etc.** M. FORTMENT (U.S.P. 1,786,202, 23.12.30. Appl., 22.12.26. Fr., 2.2.26).—See B.P. 265,548; B., 1927, 753.

**Reduction of ores.** H. WITTEK (U.S.P. 1,787,348, 30.12.30. Appl., 1.12.26. Ger., 26.7.26).—See B.P. 274,803; B., 1928, 269.

**Reduction of metals from ores.** J. W. HORNSEY, ASST. to GRANULAR IRON Co. (U.S.P. 1,786,999, 30.12.30. Appl., 27.9.28).—See B.P. 306,561; B., 1929, 360.

**Reduction of ore and conversion of hydrocarbons.** W. H. SMITH (U.S.P. 1,785,427—8, 16.12.30. Appl., [A] 27.12.26, [B] 4.4.27).—See B.P. 288,193; B., 1929, 398.

**Extraction of tin from ores or materials containing tin.** E. A. ASHCROFT (U.S.P. 1,786,386, 23.12.30. Appl., 4.5.28. U.K., 24.6.27).—See B.P. 297,784; B., 1928, 899.

**Recovery of fluorspar from ores thereof.** J. C. WILLIAMS and O. W. GREEMAN, ASSTS. to ALUMINUM Co. OF AMERICA (U.S.P. 1,785,992; 23.12.30. Appl., 26.9.28).—See B.P. 319,685; B., 1931, 68.

**Production of lamelliform metal powders.** E. PODSZUS, ASST. to HARTSTOFF-METALL A.-G. (HAMETAG) (U.S.P. 1,785,283, 16.12.30. Appl., 6.9.23. Ger., 12.9.22).—See B.P. 204,055; B., 1924, 985.

**Protectively treating metals.** A. ARENT, ASST. to A. ARENT LABS., INC. (U.S.P. 1,770,828, 15.7.30. Appl., 8.12.25).—See B.P. 338,221; B., 1931, 119.

**Removal of foreign substances such as grease and rust from metal surfaces.** C. F. DINLEY (B.P. 340,323, 28.9.29).—See U.S.P. 1,729,767; B., 1930, 18.

**Concentration of minerals** (B.P. 337,458).—See II.

## XI.—ELECTROTECHNICS.

**Industrial development of the electrical cleaning of gas.** LÉVÊQUE (Rev. Mét., 1930, 27, 513—521).—Several electrostatic gas-cleaning plants in various French metallurgical works are illustrated and brief details of the volume of gas treated, the recovery of suspended particles, and the power consumption are given.

A. R. POWELL.

**[Tar cokes from] torbanites.** NIEMANN. **Reacting methane-carbon dioxide mixture.** PETERS and PRANSCHKE.—See II. **Copper-steel.** CARIUS. **Conductivity of magnesium-aluminium alloys.** WILSON. **Protecting aluminium alloys.** BRENNER. **Tantalum carbide filaments.** BECKER and EWEST. **Treatment of metallurgical products.** MAHOUX; GUILLET. **Electrodeposited metals.** BARKLIE and DAVIES. **Contamination of plating solutions.** LISCOMB. **Chromium plating.** MACCHIA. **Plating of zinc.** ANDERSON and REINHARD. **Deposition of lead on metals.** ISGARISCHEV and IVANOV.—See X. **p<sub>H</sub>** of

**soils.** FROGNIER. **Calomel electrodes for soils.** SALMINEN.—See XVI. **Cinchona assay.** KRANTZ.—See XX.

## PATENTS.

**Conducting chemical reactions in gases and vapours with the aid of electrical discharges.** I. G. FARBENIND. A.-G., ASSEES. of (A) O. EISENHUT and (A, B) W. FRANKENBURGER (G.P. 457,563 and Addn. G.P. 458,756, [A] 25.9.24, [B] 30.9.24).—(A) The gas mixture is subjected to an interrupted glow discharge produced by allowing mercury to fall in a fine stream of drops from a mercury reservoir, which forms one electrode, into a small trough which forms the other electrode. The overflow from the lower electrode is automatically pumped back to the upper reservoir. Alternatively, the gas mixture may be passed down a tube containing perforations between metal electrodes on to which mercury is allowed to drop so as to produce a glow discharge in the neighbourhood of the perforations. (B) The gas mixture is passed through a reaction chamber filled with a metal-vapour catalyst at a low temperature and surrounding a transparent vessel in which the electrical discharge takes place. In this apparatus a mixture of methane and nitrogen yields hydrogen cyanide and methylcarbylamine.

A. R. POWELL.

**[Closing device for] doors of electric [annealing] furnaces.** HIRSCH, KUPFER- u. MESSINGWERKE A.-G. (B.P. 340,730, 3.12.29. Ger., 11.3.29).

**Manufacture of [indirectly heated] cathode for thermionic devices.** W. R. BULLIMORE (B.P. 340,212, 18.9.29).

**[Starting device for] electric-discharge lamps with rare gas filling.** A. LEDERER (B.P. 340,213, 19.9.29. Austr., 4.10.28).

**X-Ray tubes [for dental purposes].** T. H. FORDE and N. W. MELLARS (B.P. 317,451, 16.8.29).

**Light-sensitive apparatus [selenium cell].** BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of A. W. HULL (B.P. 319,286, 19.9.29. U.S., 19.9.28).

**Graphite** (B.P. 337,738). **Carbonaceous material** (U.S.P. 1,763,063). **Recovering naphthalene from gases** (B.P. 337,723). **Cracking of petroleum oil** (U.S.P. 1,766,987).—See II. **Rare-metal oxides** (B.P. 339,608).—See VII. **Reduction of steelworks' slag** (U.S.P. 1,754,845). **Magnetic materials** (U.S.P. 1,765,436). **Copper sheet etc.** (B.P. 338,173, 338,487, and 338,540). **Calcium- or strontium-lead alloys** (B.P. 318,599).—See X. **Indicator of atmospheric pollution** (B.P. 340,195).—See XXIII.

## XII.—FATS; OILS; WAXES.

**Examination of fats.** A. S. KOSS (Przemysl Chem., 1926, 10, 33—35; Chem. Zentr., 1930, ii, 491).—Values of  $n_D$  and viscosity of various oils and of their mixtures with solar oil are recorded. In certain cases the values may be employed analytically. A. A. ELDRIDGE.

**Use of ultra-violet light for detection of solvent-extracted cacao butter.** W. T. FIELD (Analyst, 1930, 55, 744—745).—As a means of differentiating extracted and expressed butters, examination of a 1% solution

of cacao butter in light petroleum in ultra-violet light has been found not entirely satisfactory. The conclusions reached are that (i) a non-fluorescent butter is almost certainly not solvent-extracted and is from low-roast beans, (ii) a highly fluorescent butter is probably solvent-extracted or is contaminated with mineral oil, (iii) slight fluorescence affords inconclusive evidence which must be supported from other sources.

H. J. DOWDEN.

[Olive] oils under ultra-violet light. E. R. BOLTON (Analyst, 1930, 55, 746).—The examination of olive oils in ultra-violet light (cf. Glantz, B., 1930, 916) has no diagnostic value since certain adulterants (e.g., tea-seed oil) will reproduce the effects of a virgin oil, whilst certain methods of refining will entirely remove the characteristic fluorescence.

H. J. DOWDEN.

Oxygen absorption and skin formation of oils and varnish. ANON. (Farben-Ztg., 1931, 36, 646—647, 691—693).—The changes in weight and in physical condition on storage of raw, varnish, boiled, and "stand" linseed oils, cobalt rosinate solution, and oil varnishes were observed, the samples being kept in closed press-capped tins about one fourth filled, the lids being removed at each weighing to renew the air. From the results, which are discussed on the light of current views on colloidal processes involved in the drying of oil films, it is concluded that oxygen absorption is the first and essential stage, and that the subsequent colloidal changes are produced thereby. The significance of oxygen absorption in varnish manufacture is indicated and the prevention of "skinning" by the use of non-drying oils and inhibitors and by exclusion of air is briefly discussed.

S. S. WOOLF.

Purification of castor oil. A. TIKHONOV (Masloboino Zhir.-Delo, 1929, No. 7, 9—14).—Purification (a) by heating with water and treatment with sodium chloride solution, and (b) by treatment with sodium hydroxide solution together with sodium chloride, is described.

CHEMICAL ABSTRACTS.

Causes of the increase in acidity of castor oil. V. JONES and M. BAUMAN (Masloboino Zhir.-Delo, 1929, No. 4, 28—39).—The lipolytic enzyme passes during pressing of the seed into the oil, where its activity is stimulated by the water-soluble acids of low mol. wt. arising from the fermentation of the foots in the unfiltered oil. The acids of high mol. wt. formed by hydrolysis of the oil do not exert any appreciable influence on the activity of the enzyme. CHEMICAL ABSTRACTS.

Darkening of cod-liver oil in the presence of iron. A. E. BRID and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1930, 19, 1308—1309).—Darkening and increase in the iron content of a commercial sample of poultry cod-liver oil stored in contact with iron is retarded by dehydration without removing the fatty acid, and, to a smaller degree, by using the alkali-refined oil.

E. H. SHARPLES.

Hydrogenation of polymerised and oxidised oils. A. BAG (Masloboino Zhir.-Delo, 1929, No. 7, 40—44).—Sunflower oil, (a) untreated, (b) polymerised by heating in carbon dioxide, and (c) oxidised by air, was hydrogenated with nickel at 230—240°. Poly-

merised oil was not depolymerised; oxidation products are reduced. When air is blown through the oil oxidation and polymerisation take place simultaneously. The hydrogenation products of the three oils are not identical.

CHEMICAL ABSTRACTS.

Formation of isooleic acid in the hydrogenation of sunflower oil. B. TRUTUNNIKOV and R. KHOLODOVSKAYA (Masloboino Zhir.-Delo, 1929, No. 5, 53—61).—Unsaturated solid acids cannot be produced by the dehydrogenation of stearic acid. In the earlier stage of hydrogenation 66% of the linoleic acid is reduced to  $\Delta^{\omega}$  oleic acid; in the latter stage this isomerises to  $\Delta^{\omega}$ -oleic acid, or isomerides with the double linking nearer to the carboxyl group. CHEMICAL ABSTRACTS.

Drying of palm oil in a drying oven and extraction in a Soxhlet apparatus. G. DE BELSUNCE (Bull. Mat. grasses, 1929, 289—290; Chem. Zentr., 1930, i, 3116).—Samples of palm oil dehydrated for 19 hrs. at 100° showed losses in weight which were almost inversely proportional to the weight taken. On account of their strong solvent powers for resinous and non-fatty substances carbon disulphide and trichloroethylene are unsuitable for extraction in a Soxhlet apparatus. Extracts with the latter must be purified by treatment with light petroleum, but even then the error is about 1%. Light petroleum is the most suitable extraction medium for dried substances. Drying of the fleshy fruit at 100° is unreliable, but the use of a vacuum desiccator is satisfactory.

L. S. THEOBALD.

Oil palms of Angola. C. DE M. GERALDES (Bull. Mat. grasses, 1930, 105—112; Chem. Zentr., 1930, ii, 492).—The oil content of the fruit and of the kernels of several varieties of *Elaeis guineensis* and *E. Poissoni* is recorded.

A. A. ELDRIDGE.

Oil-palm fruit and its oil. C. DE M. GERALDES [with C. DUARTE and F. GOUVEIA] (Bull. Mat. Grasses, 1930, 97—104; Chem. Zentr., 1930, ii, 492).—A study was made of varieties of *Elaeis guineensis* and *E. Poissoni* in respect of the characteristics of the oil, its yield, and the composition of the pericarp and kernel.

A. A. ELDRIDGE.

Pumpkin-seed oil. N. BELIAIEV (Masloboino Zhir.-Delo, 1929, No. 7, 38—40).—The whole seeds, kernels, and husks, respectively, contained: oil 24.73, 51.76, 10.0; crude protein 18.36, 35.03, 5.13; crude fibre 54.16, —, 61.15; ash 2.44, 4.16, 1.22%. The oils from the whole seeds and the kernels, respectively, had  $d_{20}^{20}$  0.9266, 0.9246; saponif. value 190.05, 188.46; iodine value 129.14, 126.50. The fatty acids, m.p. 27°, f.p. 17°, have iodine value 106.01, saponif. value 215.66. The oil resembles sunflower-seed oil.

CHEMICAL ABSTRACTS.

Rye oil. J. W. CROXFORD (Analyst, 1930, 55, 735—738; cf. Paul, B., 1921, 518 A).—Rye oils obtained from the grain, from "Ryvita crispbread," and from rye flour, by extraction with light petroleum followed by drying at 70° in an atmosphere of carbon dioxide, have been examined. The oil (semi-solid at 15°) may be classed as a semi-drying oil having an abnormally high proportion (9.3%) of unsaponifiable matter. Vitamins-A and -B are apparently absent. Rye oil

(average of 4 samples) had:  $d_{4}^{15}$  0.9353, acid value 20.6, iodine value (Wijs) 121.4, bromine value (vapour method) 75.6, saponif. value 181.3. The fatty acids derived from the oil had (aver. values): m.p. 36.8°, iodine value 126.2, bromine value (vapour method) 79.3, mean mol. wt. 299.6, and consisted of linoleic (60.9%), oleic (17.7%), and solid (21.4%) acids.

H. J. DOWDEN.

**Sulphonated oils.** VIII. Solubilities of ricinoleic acid sulphuric ester and its alkali salts in hydrochloric acid and salt solutions. K. WINOKUTI.

IX. Properties of commercial sulphonated oils. K. NISHIZAWA, K. WINOKUTI, and S. IGARAS (J. Soc. Chem. Ind. Japan, 1930, 33, 342–344 B, 344–349 B).—VIII. The solubilities are tabulated. A modification of the ether-brine method for the determination of inorganic sulphate, suitable for samples which are not salted out by brine, is mentioned. The mechanism of Kern's process (B., 1920, 632 A) is explained as the conversion, by the mild acidity of monosodium phosphate, of the sulphuric esters into acid salts, and the soaps into fatty acids, which are then salted out completely. It is shown (a) that losses of the esters would be negligible if sodium sulphate solution were used for washing sulphonated oils during the manufacture, and (b) that in separating sulphuricinoleic ester from aqueous solution, quantitative results are obtained when alkali chlorides, sulphates, etc. (cf. A., 1930, 321), or acetates followed by strong acids, are used as reagents for double decomposition and salting out.

IX. A systematic study has been made of seven commercial oils from German and Japanese sources. As in the case of pure sodium sulphuricinoleate (cf. A., 1930, 321), the properties of the oils are determined by the degree of neutralisation. The influence of concentration on the surface tension and viscosity is, in general, of the same type, but the effect on the stability of the solutions (towards acids and salts) differs markedly for the various samples.

E. LEWKOWITSCH.

**Separation of the acids of arachis oil by application of a new principle in fractional distillation.** E. JANTZEN and C. TIEDCKE (J. pr. Chem., 1930, [ii], 127, 277–291).—An apparatus is described for fractional distillation in a high vacuum, and measurement of the m.p. of the distillate while the operation is in progress. A pear-shaped flask, with a tubulure carrying a thermometer which reaches to the bottom, is affixed to an effectively lagged column, packed with aluminium rings, and maintained by electrical heating at a suitable temperature. A capillary is unnecessary. Condensed liquid from the analyser flows out of the column through a side-arm terminating in a capillary; but on account of the slow flow of liquid through this capillary, most of it overflows and is returned through the column. The distillate may be made to pass through another capillary surrounded by a water-jacket carrying a thermometer and stirrer, where, by cooling, it may be solidified, and by reheating its m.p. may be determined. The methyl esters of the acids of high m.p. from arachis oil give, when distilled in this apparatus, three main fractions of practically constant m.p. By recrystallisation to constant m.p., hydrolysis, and direct comparison of the

acids with the synthetic products, these are identified as the methyl esters of *n*-eicosic, m.p. 44.4–44.7°, *n*-docosoic, m.p. 52.4–52.6°, and *n*-tetracosic, m.p. 57.8–58°, acids. This is the first definite proof of the existence of *n*-tetracosic acid in arachis oil. No indication of the presence of *isodocosic* or *isotetracosic* acids could be obtained. H. A. PIGGOTT.

**Mechanism of the action of siccatives and of anti-oxidants.** G. DUPONT (Bull. Inst. Pin, 1930, 285–288).—In the air-oxidation of linoleic acid one of the oxidation products is an accelerator, i.e., the action is autocatalytic. In the case of abietic acid the catalyst is an oxygenated abietic acid, AO, capable of combining with oxygen to form a peroxide, (AO)O<sub>2</sub>, which oxidises 2 mols. of abietic acid. Siccatives (of which the most active is cobalt abietate or resinate) act by associating with the oxide, AO, and thus stabilising it in its active form. On the other hand, the association of AO with an anti-oxidant, such as quinol, leads to its stabilisation in an inactive form, and the amount of anti-oxidant necessary is that required to combine with and inactivate the AO present.

C. HOLLINS.

**Manganese soaps.** H. WAGNER and G. HOFFMANN (Farben-Ztg., 1931, 36, 689–691).—The behaviour of umbers (2–30% Mn), pyrolusites, and laboratory-prepared hydroxides of manganese of different degrees of oxidation, when mixed with linoleic acid and subjected to the action of ultra-violet light, was studied microscopically. Photomicrographs show that crystals of manganese soaps and of linoleic acid may separate out. Umbers of relatively high manganese content yield soaps, but pyrolusite does not so react. The experiments with the prepared hydroxides show that crystallisation is induced by the presence of free oxygen or other suitable catalyst.

S. S. WOOLF.

**Lubricating oils.** TAUSZ and STAAB.—See II. **Effect of soap on aluminium.** BOHNER. **Resistance of aluminium to fatty acids.** CLAUS.—See X. **Prevention of foaming.** MEYER.—See XV. **Iodometry of food products.** FUCHS and others. **Milk fat.** ANDERSEN.—See XIX. **Spiritus saponatus etc.** MEYER. **Emulsions.** SMITH and others.—See XX. **Germicidal soaps.** SCHAFER and TILLEY.—See XXIII.

PATENTS.

**Preservation of bones [for fat and glue extraction].** BRIT. GLUES & CHEMICALS, LTD., and R. B. DREW (B.P. 340,010, 7.9.29).—Fresh bones are comminuted (preferably in the form of shavings), washed, and dried by heating *in vacuo* or in a current of dry air until the moisture content is reduced to about 10%. Suitable plant is described. The product may be stored without decomposition. E. LEWKOWITSCH.

**Heat treatment of plants and fruits containing oil.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 340,107, 25.11.29. Ger., 23.11.28. Addn. to B.P. 339,048; B., 1931, 125).—The oily fruits are heated in a stream of hydrogen; catalysts may be introduced by mixing the fruits with oxides of iron, copper, manganese, etc., or by steeping them in solutions of salts of these metals prior to heating, or the vapours produced may be led over the catalyst. E. LEWKOWITSCH.

**Apparatus for recovery of fats, oils, or oleaginous substances from materials yielding them.** W. T. POWLING, Assr. to J. W. PITTOCK (U.S.P. 1,785,361, 16.12.30. Appl., 13.1.27. U.K., 29.1.26).—See B.P. 269,985; B., 1927, 495.

**Production of neutral fats and oils.** O. JORDAN and G. KRAEMER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,786,248, 23.12.30. Appl., 18.3.29. Ger., 5.4.28).—See B.P. 312,523; B., 1929, 608.

**Water-soluble product from fatty acids in wool fat.** G. MAUTHE and A. THAUSS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,784,941, 16.12.30. Appl., 31.1.29. Ger., 7.2.28).—See B.P. 305,597; B., 1930, 725.

**Shredded soap manufacture.** J. A. SCHWANTES, Assr. to COLGATE-PALMOLIVE-PEET Co. (U.S.P. 1,785,532—3, 16.12.30. Appl., 28.6.28).—See B.P. 314,482; B., 1930, 725.

**Extraction of vitamin-A from fish oils and the like.** K. TAKAHASHI, Assr. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,786,095, 23.12.30. Appl., 2.5.23. Jap., 10.2.23).—See B.P. 220,697; B., 1924, 886.

**Fatty acid derivatives (B.P. 337,368).**—See III. **Washing processes (B.P. 338,121).**—See VI. **Resinous products (B.P. 339,958).**—See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**"Setting" of red lead [paint].** W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1931, 36, 644—645).—The viscosity-time relationships were determined for a number of red lead paints comprising non-setting and "normal" red leads with a range of different proportions of oil, various mixtures of litharge and lead peroxide with the same proportion of oil, and paints in which the amounts of oil for grinding and for thinning out and the period between these two operations were varied. The results, which are tabulated and graphed, indicate that the amount of oil used should be kept as high as is compatible with general utility, opacity, etc. Increase in peroxide content diminishes settling on storage, other things being equal. Allowance of a period of 3—4 days between grinding and thinning out is recommended.

S. S. WOOLF.

**Possibilities of inorganic paint vehicles.** N. HEATON (J. Oil and Col. Chem. Assoc., 1930, 13, 330—340).—Organic media generally employed in the paint industry have decided limitations, chiefly with respect to temperature, moisture, and chemical activity. The author reviews the classical methods of mural decoration, and shows that the recent work of Laurie, Wilson, and of King on the uses of silicon ester as a paint vehicle offers a great advance on the older methods (cf. King, B., 1930, 622). Purely inorganic vehicles such as sodium silicate are not of general application, nor is the silicon ester method suitable where the surface to be painted is non-porous, owing to the limited elasticity of the silica film.

F. C. HARWOOD.

**Pigment primaries and mixtures.** F. F. RUPERT (J. Opt. Soc. Amer., 1930, 20, 661—684).—An account is given of an attempt to establish a method of predicting

quantitatively the results of mixing of coloured paints, especially of water colours, by the application of certain general principles to the spectrophotometric curves of the constituents of the paint mixture. Owing to the complexity of mathematical relations obtained from theoretical considerations and ignorance of some necessary factors, a satisfactory method was not discovered. W. GOOD.

**Inks and ultra-violet light.** C. A. MITCHELL (Analyst, 1930, 55, 746—747).—Whilst erasures of pencil markings are readily distinguishable under ultra-violet light, the differentiation of writing inks or of writings in the same ink of different ages is less satisfactory. Tannin solutions of different origin have characteristic fluorescent appearances, but when made into inks they are indistinguishable even when the inks on paper are obviously different. H. J. DOWDEN.

**Removal of printer's ink [from paper].** G. SCHWALBE (Z. angew. Chem., 1931, 44, 24).—Small quantities of gum or gutta-percha in trichloroethylene solution remove the ink by emulsification.

E. S. HEDGES.

**"Gemmes" [crude resin-terebenthene].** (MLLE.) M. BARRAUD (Bull. Inst. Pin, 1930, 217—223).—In Austrian pine "gemmes" (excluding the "barras" or sixth "gemme") the terebenthene varies from 19.8 to 20.8%, resin 64—72%, ash 2—4.5%, and water 3—14%; the Sourgen process gives a "gemme" with 4.5—23% of water. The proportion of terebenthene is largest in the early part of the year; the effects of the size of the incision, the mode of collection, the orientation and age of the incision, the size of the tree, and its distance from the sea are described. The rotatory power of the terebenthene obtained from Austrian and maritime pines varies considerably, especially in the latter, which is found to contain *d*-pinene. A Bordeaux pine has given a *l*-pinene of  $[\alpha]_D -51.13^\circ$ , the highest yet recorded. C. HOLLINS.

**Constitution of phenol-formaldehyde resins.** F. POLLAK and F. RIESENFELD (Z. angew. Chem., 1930, 43, 1129—1132).—The acid-condensation product ("Novolak") obtained from pure phenol and  $\beta$ -polyoxymethylene was isolated and its structure examined by determination of ultimate composition, mol. wt., hydroxyl number, behaviour with alkali, water, etc., procedure and results being detailed. It is deduced that the "Novolak" consists of a chain of 7 mols. of phenol and 6 mols. of formaldehyde, 6 mols. of water being eliminated. The two end phenol residues differ from the remaining five, in that the former are disubstituted and the latter trisubstituted. This evinces itself in the relatively loose attachment of two phenol groups per mol. etc. A possible ring structure for the product is rejected from similar reasoning, and the resinous character of the "Novolak" is attributed to existence of a number of isomeric products rather than to molecular magnitude. S. S. WOOLF.

**Pine wood.** SOUM. **Nitrocellulose.** BECK and others. **Nitrocellulose from jute.** BAGCHI.—See V. **Behaviour of aluminium etc. with moist lithopone.** ANON. **Protecting aluminium alloys.** BRENNER.—



See X. Skin formation on varnishes. ANON. Rye oil. CROXFORD.—See XII.

## PATENTS.

**Production of a composition for coating or impregnating fabric.** L. D'ANTAL (B.P. 338,538, 20.8.29. Hung., 19.10.28).—A cheap, soft, rubber-like product, suitable for impregnating paper or fabric bags to render them resistant to chemicals, such as caustic alkali, carbide, and quicklime, is prepared by oxidising above 180° a vegetable drying oil (*e.g.*, linseed or sunflower oil) with the acid sludge obtained as a residue in mineral oil refining; resin or asphalt-like substances may be added to the vegetable oil before oxidation.

A. J. HALL.

**Reflecting material.** H. H. HIGBIE (U.S.P. 1,767,285, 24.6.30. Appl., 28.7.28).—The material comprises a layer of a highly reflecting white substance, *e.g.*, magnesium carbonate, zinc oxide, titania, or lead carbonate, covered with a layer of a transparent or translucent substance, *e.g.*, cellophane or thin glass. Alternatively the white substance may be incorporated into a plastic and the mixture made into sheets which are dried in such a way that a highly reflecting surface is produced. Application to wall coverings, lamp-shade linings, etc. is indicated.

A. R. POWELL.

**Production of red lead.** H. ERZINGER, and CHEM. FABR. SCHONENWERD H. ERZINGER A.-G. (B.P. 340,082, 30.10.29).—A suspension of commercial litharge in water is treated with formic acid, carbon dioxide being passed through until a basic lead carbonate of very low density is produced. This is separated, dried, and oxidised at a temperature which is 100° lower than that employed in making crystal red lead, a finely-divided product ("disperse" red lead) being thus obtained.

W. J. WRIGHT.

**Condensation of urea or its derivatives with formaldehyde or its polymerides.** FABR. DE PROD. DE CHIM. ORGANIQUE DE LAIRE, J. MALET, and R. ARMEN-AULT (B.P. 340,114, 6.12.29. Fr., 6.12.28).—The components are condensed together in the presence of carbonate or oxide of zinc or lead, bismuth carbonate, a coloured metal carbonate, titanium oxide, etc. as condensing agent.

S. S. WOOLF.

**Manufacture of [resinous] condensation products.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,958, 12.8.29).—Almost neutral, resinous, oily products are obtained by heating (*e.g.*, at 260°) long-chain aliphatic hydroxy-acids (*e.g.*, ricinoleic, dihydroxystearic acids) or their esters or glycerides with colophony etc., *in vacuo*, or in a stream of inert gas until the acid value has practically disappeared. Acid or alkaline condensing agents may be used. E. LEWKOWITSCH.

**Synthetic resins and their uses.** BRIT. CELANESE, LTD. (B.P. 340,101—2, 18.11.29. U.S., 17.11.28).—An aromatic sulphonamide is condensed with (A) benzaldehyde, (B) furfuraldehyde, in the presence or absence of acid, neutral, or alkaline catalysts, and, if desired, of one or more reactants capable of forming other synthetic resins, *e.g.*, formaldehyde, acetone, urea, phenol, aniline. Solvents, resins, plasticisers, pigments, and/or dyes may be incorporated.

S. S. WOOLF.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Water absorption of rubber compounds.** H. A. WINKELMANN and E. G. CROAKMAN (Ind. Eng. Chem., 1930, 22, 1367—1370).—Using a mixture of rubber (93 pts.), zinc oxide (3 pts.), sulphur (5 pts.), and diphenylguanidine (0.75 pt.), with 5, 10, and 15 vols. of various compounding ingredients at various degrees of vulcanisation, increased volume-loading with any ingredient has little effect on the water absorption. With increasing time of vulcanisation the water absorption at first decreases, reaching a minimum near the "optimum cure," and then increases. Whiting and barytes increase the water absorption of over-vulcanised rubber, but clay and zinc oxide have a marked adverse effect at all degrees of vulcanisation; carbon black has little effect and thermatomic black decreases the water absorption. The presence of salts such as metallic acetates inflates the water absorption, as also does subjecting the rubber to stretch. A vulcanite mixture absorbs water in much the same manner as a soft rubber mixture, but the absorption decreases rapidly as optimum vulcanisation is approached. Reclaimed rubbers vary in water absorption, the effect increasing with the proportion of residual alkali. Immersion of a vulcanised tread-mixing in water for 30 days with subsequent drying does not affect the tear-resistance, but decreases the elongation and increases the tensile strength and resistance to abrasion.

D. F. TWISS.

## PATENTS.

**Vulcanising rubber to leather.** L. B. CONANT, ASSR. to STANDARD PATENT PROCESS CORP. (U.S.P. 1,787,145, 30.1.30. Appl., 22.7.27).—See B.P. 275,194; B., 1928, 494.

**Polymerised  $\alpha\gamma$ -butadiene** (B.P. 337,460).—See III.

## XV.—LEATHER; GLUE.

**Extraction of fir bark.** M. RAUCHWERGER (Gerber, 1930, 56, 81—83; Chem. Zentr., 1930, ii, 1025).—In order to avoid high losses of tannin, the material was extracted for 2 days with cold water and finally with water at 85°. Bark (1 kg.) containing 135 g. of tanning substances gave an extract containing 59.4 g., 48 g. remaining in the bark and 27.6 g. being lost by hydrolysis. Extraction at 15° yielded 69.4 g., 50 g. remaining and 15.6 g. being lost.

A. A. ELDRIDGE.

**Determination of insoluble matter in tanning extracts.** E. BĚLAVSKY and G. WANĚK (Gerber, 1930, 56, 83—84, 89—90; Chem. Zentr., 1930, ii, 1027).—The official method 'or the determination of insoluble material does not measure the insoluble matter in the actual tanning liquor, which is preferably determined by the sedimentation method. Factors on which the determination depends have been examined.

A. A. ELDRIDGE.

**Detection of mineral-tanned leather in smallest quantities of leather.** K. KLANFER (Mikrochem., 1931, 9, 34—37).—The tanning agent used in mineral tanning can best be recognised by microchemical methods. Chromium may be detected by igniting a small quantity of leather and treating the ash with sodium peroxide to form sodium chromate. An intense violet coloration is

obtained on the addition of a drop of sulphuric acid and 1 or 2 drops of 2% diphenylcarbazine. Iron may be recognised by performing the thiocyanate test on the ash, and aluminium by dissolving the leather ash in fused potassium hydrogen sulphate, cooling, dissolving in hot water, and testing with alizarin or with morin.

E. S. HEDGES.

**Prevention of foaming in aqueous binding and thickening media.** J. B. MEYER (Farben-Ztg., 1930, 36, 481—482, 573—574, 619—620).—Methods available for reducing the troublesome foaming in technical operations involving colloidal solutions are reviewed. In most cases the substances used in practice prevent or diminish foaming in a particular operation, but are not universally applicable. The effect of adding numerous organic liquids to a foam made with a glue solution was investigated; fatty acids had more effect in destroying the foam than had the corresponding alcohols. No relation could be found between the surface tension of the liquid and its power for destroying foam, but in a homologous series the preventive effect increases with the mol. wt. of the compound. With increasing concentration of glue the foaming power passes through a maximum. A mixture of linseed oil and alkali is very effective in preventing foaming.

E. S. HEDGES.

#### PATENTS.

**Working up cellulosic materials** (B.P. 340,164).—See V. **Glue from bones** (B.P. 340,010).—See XII.

### XVI.—AGRICULTURE.

**Soil colloids.** F. N. ANTIPOV-KARATAIEV and A. J. RABINERSON (Proc. Leningrad Lab. Agro-Soil Inst. U.S.S.R., 1930, No. 10 : Proc. Internat. Soc. Soil Sci., 1930, 5, 218—220).—A critical survey of recent work in the physical and physico-chemical properties of soil colloids.

A. G. POLLARD.

**Colloidal nature of soil humus.** L. WOLF and H. SCHLATTER (Cellulosechem., 1930, 11, 254—257).—A stable humus sol is obtained by shaking sandy soils with water for 8 days. The production of this sol depends on the presence of coarse sand acting as a colloid mill, since no humus is dissolved if the sand is initially removed. The humus obtained by electrodialysis can, after drying, no longer be peptised on shaking with water. The dry material contains 47—53% of ash, essentially ferric aluminium silicate; the ash content is diminished to about 30% by prolonged treatment with hydrochloric acid. A solution of humus in *N*-potassium chloride is somewhat more acid than a similar one in water.

T. H. MORTON.

**Influence of irrigation on the chemical and physical properties of saline alluvial soils.** S. J. SUSHKO (Proc. Lenin. Acad. Agric. Sci., U.S.S.R., 1930, No. 9; Proc. Internat. Soc. Soil Sci., 1930, 5, 238).—The effects of repeated leaching of soils are examined. In bog soils and alkaline bog soils the alkalinity decreased with the earlier leachings, but increased as the process continued. The soluble calcium content of the soil varied similarly. The reappearance of calcium in the later leachates is ascribed to the decomposition of calcium carbonate, to the displacement of adsorbed

calcium, or to the effect of both factors combined. In a "swelling" saline soil the increased calcium content of later washings resulted from the slow and irregular dissolution of gypsum. Each soil exhibited characteristic properties during prolonged extraction with water.

A. G. POLLARD.

**Soil acidity.** A. TERÄSVUORI (Valtion Maatalouskoetöiminnan Julkaisuja, 1930, No. 29 : Proc. Internat. Soc. Soil Sci., 1930, 5, 206).—The effect of electrolytes on the observed  $p_H$  values of soils is examined.

A. G. POLLARD.

**Determination of the  $p_H$  of soils by means of the quinhydrone electrode.** R. FROGNIER (Ann. Soc. Sci. Brux., 1930, 50, B, 135—144).—The  $p_H$  of soil suspensions is generally different from that of the liquid after the solid particles have been removed by centrifuging. An investigation of the causes of this phenomenon has shown that the principal factor may be traced to diffusion of the potassium chloride used in making the measurements; this reacts with the soil particles by exchange adsorption, liberating hydrogen ions therefrom. The diffusion can be greatly reduced by using a plug of filter paper at the end of the potassium chloride bridge. The purity of the quinhydrone is also of more importance than has hitherto been recognised, acid preparations affecting the results considerably, especially in suspensions which are not efficiently buffered. The form of the platinum electrode has received attention, and it is recommended to use a platinum wire 5 or 6 cm. long with only the tip immersed in the suspension; the tip should be passed through a flame before each measurement is made, and the wire should be kept in chromic acid mixture when not in use.

E. S. HEDGES.

**Calomel electrodes [for soils].** A. SALMINEN (J. Agric. Soc. Finland, 1930, 60—62; Proc. Internat. Soc. Soil Sci., 1930, 5, 206).—A form of calomel electrode suited to the field examination of soils is built in a test tube and connected by an agar-agar-potassium chloride bridge with the test solution.

A. G. POLLARD.

**Effects of manuring and cropping on soil dispersion.** V. S. DENISIEVSKI (Nat. Agric. Exp. Sta. Kiev, 1930, No. 40; Proc. Internat. Soc. Soil Sci., 1930, 5, 214).—Existing methods of defining soil dispersion are unsatisfactory. Dispersion decreases with the soil moisture content, which is the principal controlling factor. Cropping affects dispersion mainly by its influence on the moisture content of the soil. Application of lime, straw, and mineral fertilisers decreases soil dispersion, the effects decreasing in the order named. Farmyard manure may exert a reverse effect in spring and autumn. Dispersion decreases with increasing electrolyte content of the soil. A higher degree of dispersion is associated with increased  $p_H$  value and with higher ammonia absorption.

A. G. POLLARD.

**Nitrogen fixation in some Iowa soils.** R. H. WALKER and P. E. BROWN (Iowa State Coll. J. Sci., 1930, 4, 425—429).—Tests with two soils are described.

CHEMICAL ABSTRACTS.

**Mobilisation and immobilisation of calcium in soil.** A. T. KALATSCHIKOV (Nat. Agric. Exp. Sta.

Kiev, 1929, 38, No. 28; Proc. Internat. Soc. Soil Sci., 1930, 5, 209—210).—In moist soils (60% of water capacity) there occurs simultaneously a physico-chemical adsorption of calcium and a biological process of mobilisation. Addition to soil of organic material (*e.g.*, starch) causes the biological immobilisation of calcium. Nitrification and mobilisation of calcium are parallel processes. In water-saturated soils mobilisation proceeds in the presence or absence of starch. In soils of optimum moisture content the mobilisation of calcium and the immobilisation of phosphates are antagonistic processes. In the presence of starch the  $pH$  of the soil controls the mobilisation of calcium. A. G. POLLARD.

**Culture of *Aspergillus niger* for the biochemical determination of potassium and phosphoric acid fertiliser needs of soils.** H. NIKLAS, H. POSCHENRIEDER, and J. TRISCHLER (Ernähr. der Pflanze, 1930, 26, 97—103; Chem. Zentr., 1930, i, 3097—3098).—A method for determining potassium and phosphorus in soils by means of culture of *Aspergillus niger* is now described. L. S. THEOBALD.

**Orthophosphoric acid as a fertiliser constituent and an active soil component.** M. GRAČANIN (Jahresber. Univ. Zagreb, 1930; Proc. Internat. Soc. Soil Sci., 1930, 5, 224—226).—Orthophosphoric acid is produced in soil during the mineralisation of plant and animal residues, by the decomposition of phosphatides, nucleoproteins, and phytin. Superphosphate contains up to 5% of orthophosphate. In water cultures, orthophosphates are typical activators of germinative energy. Crop increases due to orthophosphate in pot cultures vary with the nature of the plant and of the soil. The injurious concentration of phosphoric acid is much higher than that of other fertiliser materials. No increase of soil acidity results from fertilisation with phosphate even in excessive amounts. Small dressings of orthophosphate reduce the acidity of acid soils and the alkalinity of alkaline soils. A. G. POLLARD.

**Effect of "nitrophoska" complete fertiliser compared with that of other manurial combinations.** T. POLLINGER (Fortschr. Landw., 1930, 5, 320—321; Chem. Zentr., 1930, ii, 446).—The composition of "nitrophoska" is variable; the conditions under which the 1928 field tests were carried out are criticised. A. A. ELDRIDGE.

**Fertilisation with phosphorites.** G. TOMMASI and S. D. DI DELUPIS (Ann. R. Staz. Chim.-Agr. Sperim. di Roma, 1930, 13, Publ. No. 271, 41 pp.).—Excepting in soil with an acid reaction or free from lime, the direct application of natural phosphates is not advisable to cereal crops, which are able to utilise the phosphoric acid of slightly soluble compounds in only slight degree. Indirect application of natural phosphorite—to the preceding crop—may, however, be of advantage, even in neutral soils, but the best results are then obtainable by the supplementary use of small amounts of soluble fertilisers to supply sufficient readily-assimilable phosphoric acid during the initial periods of growth. Tests with maize show that, after flowering, this is well able to utilise slightly soluble phosphates. With leguminous crops, especially when grown for several years on the same plot, direct application of finely-powdered natural

phosphates is profitable, not only to acid, but also to neutral soils, especially if these are poor in lime. Kosseir phosphorite is especially adapted to direct application, owing to the readiness with which it may be powdered to a high degree of fineness. T. H. POPE.

**Value of rock phosphate and "bone and superphosphate" as fertilisers for maize production.** A. D. HUSBAND (Rhodesia Agric. J., 1929, 126, 1247—1252).—In soil of  $pH$  7 raw rock phosphate was slightly more effective than a mixture of bone meal and superphosphate (1 : 2) containing the same amount of citrate-soluble phosphate. CHEMICAL ABSTRACTS.

**Plant growth experiments with ammonia solution [as fertiliser].** BIMSCHAS (Gas- u. Wasserfach, 1931, 74, 13—16).—Experiments have been made with 2% and 20% ammonia solutions as fertilisers for oats and mustard, in both sandy and heavy soils in conjunction with acid and alkaline fertilisers containing potash and phosphate. The results indicate that especially when used with an alkaline fertiliser gasworks by-product ammonia may be satisfactorily used as the source of nitrogen. H. F. GILLBE.

**Free-ammonia injury with concentrated fertilisers.** L. G. WILLIS and W. H. RANKIN (Ind. Eng. Chem., 1930, 22, 1405—1407).—Seedlings in light, sandy soil sustain root injury from organic ammoniates in mixed fertilisers, unless the latter contain calcium salts to combine with the free ammonia. In pot-culture tests with cottonseed meal on cotton seedlings, the effect of various amounts of nitrogen and gypsum per acre, and of various intervals between application and planting, was investigated on the basis of the percentage of seedlings injured. Root injury occurs with a proportion of 16 lb. of nitrogen per acre and no calcium salt, if the interval is less than 8 days, but if calcium is present planting may be done immediately. When the proportion of nitrogen is 32 lb. per acre (without gypsum), injury was severe until 16 days had elapsed; with an 8-day interval the gypsum has no protective action until the third planting. W. J. WRIGHT.

**Unsuitability of sodium chlorate as a weedicide in banana plantations.** H. W. EASTWOOD (Agric. Gaz. N. S. Wales, 1930, 41, 851—852).—The spraying of sodium chlorate for weed control injured banana plants, not only by contact with aerial portions, but also by root injury when applied 1 ft. from the stools. After several weeks leaves became discoloured and fell. New leaves produced subsequently were healthy. A. G. POLLARD.

**Abnormalities in the composition of oranges.** P. R. V. D. R. COPEMAN (S. Afr. J. Sci., 1930, 27, 310—316).—Fruit from two among six trees examined had abnormally low contents of soluble solids, sugar, and acids. Statistical examination of analytical data obtained at intervals during the growth season shows the abnormalities to be inherent characteristics of the trees. The difficulty of obtaining average "normal" data for fruit grown under apparently uniform conditions is emphasised. A. G. POLLARD.

**Species of wood on different types of soil.** H. BURGER (Mitt. schweiz. Centralanst. forstl. Vers., 1930, 16,

[i]; Proc. Internat. Soc. Soil Sci., 1930, 5, 231—234).—Ash constituents of a number of woods varied with the soils in which they grew, but, in general, were characteristic of species. Wood grown on light calcareous soils was richer in ash constituents and higher in calcium content than that from soils deficient in lime. The magnesia content of the wood varied with the amounts available in the soil. The CaO : MgO ratios of individual species of woods grown on different soils were within fairly well-defined limits. No direct relationship was observed between the phosphorus contents of woods and the soils in which they grew, although, in general, high phosphate contents in wood were associated with lime-deficient soils and vice versa. Wood grown on humus soils had a relatively low potash content, without, however, exhibiting potash deficiency. The CaO : SiO<sub>2</sub> ratios of various types of woods lie in fairly definite ranges. Relatively high nitrogen contents of woods are associated with lime-deficient soils. Relationships between the nature of soils, their lime and nutrient contents, and the growth and nutrient content of individual species of trees growing on them are discussed. A. G. POLLARD.

**Factors influencing yield and quality of peas.** V. R. BOSWELL (Maryland Agric. Exp. Sta. Bull., 1929, No. 306, 341—382).—The effect of soil conditions on the growth, nodulation, and quality of peas was examined. Acidity within the range  $p_H$  5.8—6.7 had little effect on nodulation. Of soil factors examined the organic content was the most closely related to growth, yield, and nodulation. Fertiliser trials indicate that larger applications of nitrogen than are customary are advantageous. The growth and maturing of peas is characterised by a rapid decrease in the proportions of sugar and soluble nitrogenous matter and by an increase in starch, acid-hydrolysable matter, and insoluble nitrogen. Good quality in peas is associated with high sugar and low starch contents. Late-sown, late-maturing peas were not markedly different in sugar or starch content from earlier varieties. A. G. POLLARD.

**Selection of potatoes and the effect of external conditions, especially manuring, on the result of selection.** H. M. QUANJER (Ernähr. der Pflanze, 1931, 27, 1—8).—Nitrogenous fertilisers increased the severity of pseudonecrosis in potatoes from infected seed and favoured its spread to adjacent plants from clean seed. Potash fertilisers produced the reverse effect. A. G. POLLARD.

**Sweet potato (*Ipomœa batatas*).** S. D. TIMSON (Rhodesia Agric. J., 1929, 26, 890—903).—Analyses of silages made from maize and from sweet potato tops are recorded. CHEMICAL ABSTRACTS.

**Incidence and control of apple scab and apple mildew at East Malling.** II—III. M. H. MOORE (J. Pomology, 1930, 8, 283—304).—The application of either lime-sulphur mixtures or Bordeaux mixture at the "pink bud" stage is important in the control of apple scab. Bordeaux mixture with lead arsenate, applied three times, once at the 8-8-100 concentration at the "pink bud" stage, and twice at 8-25-100 (i.e., with excess of quicklime) after blossoming, gave the best results. Trees of the variety Cox's Orange Pippin

suffer severely from such sprays. The results obtained suggest that post-blossom applications of Bordeaux mixture are cumulative and lead, in subsequent years, to a reduction in the number of blossom trusses. Lime-sulphur with lead arsenate, applied at the same three stages, gave good control and did not damage Cox's Orange Pippin trees. Colloidal sulphur gave approximately as good control as 1-150 lime-sulphur, both applied after blossoming; preliminary trials with sulphur dusts are recorded. There were indications in further experiments that trees receiving a fertiliser are less susceptible than untreated trees to scab infection. No treatment tried was completely successful against apple mildew, but three applications of lime-sulphur, without arsenate, were the most effective. E. HOLMES.

**Raspberry and loganberry beetle and its control.** Further experiments with pyrethrum emulsion sprays and a dust. C. L. WALTON (J. Pomology, 1930, 8, 309—315).—Experiments in which logan infestations were reduced from 35.8 and 9.6% to 7.4 and 1.0%, respectively, support earlier work proving the utility of pyrethrum emulsion sprays. Pyrethrum dusts are much inferior. E. HOLMES.

**Composition of soils of Hungarian lowlands.** I. Lime. S. ARANY (Mezőg.-Kutat., 1929, 2, 557—569; Chem. Zentr., 1930, ii, 447).

[Tar from] torbanites. NIEMANN.—See II. Pine wood. SOUM.—See V. Florida phosphates. HILL and others. Citrate-insoluble residues from superphosphates. JACOB and others. Ammoniation of superphosphate. KEENEN.—See VII.

#### PATENTS.

**Production of dicalcium fertilisers.** A. HOLZ (B.P. 316,583, 30.7.29. U.S., 31.7.28).—Phosphate rock is decomposed with sulphuric acid, one third of the precipitated calcium sulphate is removed, and ammonia is added, the remaining calcium sulphate being kept in suspension by stirring and the pressure increased. The dicalcium phosphate, which contains 2½ times as much available phosphoric acid as commercial acid phosphate fertiliser, is dried at a temperature insufficient to remove water of crystallisation, and the ammonium sulphate in the mother-liquor is crystallised and mixed with it. W. J. WRIGHT.

**Manufacture of a phosphate for fertilising.** E. HAMMEL, Assr. to M. RAUCH (U.S.P. 1,786,651, 30.12.30. Appl., 23.12.26. Ger., 19.8.26).—See B.P. 284,741; B., 1928, 280.

**Manufacture of ammonium phosphate fertiliser.** G. H. BUCHANAN, Assr. to AMER. CYANAMID CO. (U.S.P. 1,785,375, 16.12.30. Appl., 28.1.27).—See B.P. 284,322; B., 1928, 724.

**Fertilisers** (B.P. 339,340, 339,500, and 339,562, and U.S.P. 1,768,075).—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Volumetric determination of reducing sugars.** IV. Invert sugar. A. R. LONG and W. A. CARTER (Analyst, 1930, 55, 730—734; cf. B., 1908, 581, 582).—In the titration of reducing sugars with Fehling's solu-

tion, using methylene-blue as an internal indicator (cf. Lane and Eynon, J.S.C.I., 1923, 42, 32 r), the same volume of indicator must be used for standardising the Fehling's solution as for the assay, and it should be added just before titration is complete. The greatest accuracy is secured when (i) the liquid is kept boiling throughout the titration, (ii) the titration occupies more than 10 min., (iii) air is excluded from the surface. These conditions are conveniently fulfilled by using a closed flask and by adding to the boiling Fehling's solution small volumes of the assay liquid (0.5 c.c.) at short intervals of time (15 sec.). H. J. DOWDEN.

**Determination of starch in potatoes.** B. LAMPE (Z. Spiritusind., 1930, 52, 357).—Errors ranging from 0.2 to 0.3% in the value of the starch content of potatoes may occur if the measurement of the under-water weight of the potatoes is made before the potatoes have reached the normal temperature (17.5°). The potatoes, which may have been chilled by a washing water of low temperature, should be kept for at least  $\frac{1}{2}$  hr. in the water maintained at 17.5° prior to the determination of the under-water weight, on which is based the calculation of the starch value. C. RANKEN.

**Determination of the tenacity of starch.** A. PARLOW and G. DÜLL (Z. Spiritusind., 1930, 53, 356—357).—The viscosity of a solution of starch paste kept at 100° gradually decreases after the first 2 or 3 min. This decrease varies very much with different starches, and whilst the initial and final viscosities of one starch solution kept at 100° for 3 hrs. were 139 and 134%, respectively, the corresponding values for a second were 256 and 96%. Starches with large grains have a lower initial viscosity, but are not affected much by temperature, whereas the viscosity of those with small grains rapidly decreases and may eventually reach a value smaller than that given by a starch with a lower initial viscosity. It is accordingly suggested that it is misleading to take the initial viscosity of a starch solution as the criterion of its tenacity, and the viscosity changes and the final viscosity after retention at a high temperature should be taken as the governing factors. Owing to the extreme sensitiveness of the viscosity to the condition of the starch solution, full details are given of the methods for its preparation. C. RANKEN.

**Bakers' yeast.** ROSENBAUM. **Xylose for citric acid fermentation.** FREY. **Alcohol from molasses.** SCHINDLER. **Sugars in port wine.** MUTTELET.—See XVIII. **Photoactivity of honey.** STITZ. **Starch in vegetable preserves etc.** VON FELLEBERG.—See XIX. **Vanilla sugar.** DINGEMANS.—See XX.

#### PATENTS.

**Curing of masecuite.** J. BERGE, Assr. to RAFFINERIE TIRLEMONTTOISE Soc. ANON. (U.S.P. 1,784,982, 16.12.30. Appl., 11.2.27. Ger., 24.7.26).—See B.P. 274,799; B., 1928, 31.

### XVIII.—FERMENTATION INDUSTRIES.

**Bakers' yeast.** I. **Influence of age on the fermentation time of the yeast.** E. ROSENBAUM (Z. Unters. Lebensm., 1930, 59, 607—612).—The manufacture of yeast cake from beet-sugar molasses is described and the results of examination of a number of

samples of different origin are recorded. The water content as determined by heating at 105—108° for 4 hrs. is generally 72—73%; a high value is associated with poor keeping qualities. This property was gauged by the time required for the cake to soften when heated at 35°; the times ranged from 55 to 105 hrs. The nitrogen content (Kjeldahl) varied from 1.81 to 2.52% of the dry material, corresponding to protein contents of 41.38—57.25%. The "fermentation time" was determined by the time taken by a dough of standard composition pressed into a mould of fixed dimensions to rise 5 times in succession to a height of 7 cm. above the mould. The sum of the five periods or total fermentation time showed great variation (176—271 min.). The fermentative power of the yeast decreases rapidly on storage, but the deterioration is reduced by storage at low temperatures. II. J. DOWDEN.

**Diastatic power of malt and malt extract.** C. T. BENNETT and F. C. L. BATEMAN (Quart. J. Pharm., 1930, 3, 349—353).—The Lintner test (A., 1886, 386), even with later modifications (Harrison and Gair, B., 1906, 830; Ling, B., 1910, 834; Lane and Eynon, A., 1923, ii, 193; Baker and Hulton, B., 1921, 272), is stated to be unsatisfactory; variability of the soluble starch used is given as the chief cause of inconsistent results. It is recommended that attention should be paid to the freedom of the distilled water and the laboratory atmosphere from ammonia and nitrites, to careful temperature regulation (the use of a thermostat is desirable), and to frequent agitation during digestion and starch conversion. It is considered that the present official test (Ministry of Agriculture's Marketing Leaflet No. 14, Appendix 3) based on a report of the Institute of Brewing, as modified by Hind and others (B., 1926, 170), needs amendment before it can be considered suitable for inclusion in the British Pharmacopoeia. R. CHILD.

**Xylose as a nutritive substrate for citric acid fermentation.** A. FREY (Z. angew. Chem., 1931, 44, 16—21).—The value of xylose as a substrate for the production of citric acid by means of *Aspergillus niger* and *Citromyces* has been investigated. Preliminary purification of the xylose is necessary, and, in particular, furfuraldehyde and other aldehydes must be removed. This may be effected by steam-distillation or by adsorption on active charcoal. Under favourable conditions the yield of citric acid may be 20%. but is generally lower. E. S. HEDGES.

**Alcohol from cane-sugar molasses.** H. K. SCHINDLER (Chem.-Ztg., 1930, 54, 993—994).—As a result of the general and increasing use of mixtures of benzine, benzol, etc. with 99% alcohol for power fuel, the problem arises as to whether it is more remunerative to convert cane-sugar molasses into alcohol than to manufacture sugar from it. The loss of residues from the manufacture of sugar, which can be used as fodder, is more than counterbalanced by the value of the fermentation carbon dioxide. C. RANKEN.

**Barley husks.** H. LÜERS (Woch. Brau., 1930, 47, 557—561, 571—575).—The amounts of nitrogen, ash, and pentoses extracted by water, 0.25% caustic potash, 0.25% hydrochloric acid, saturated lime water, and lactic buffer solution ( $p_H$  5.0) at various temperatures were

determined. The amounts of nitrogen and ash dissolved varied widely with the conditions of extraction, that of the pentoses much less. There were indications that enzyme action is a factor in aqueous extracts. The nitrogenous constituents were extracted in greatest quantity by dilute caustic potash, and by hydrochloric acid especially at high temperatures. The solubility in water was much less, but was increased by heating under 1 atm. pressure. The increased nitrogenous material extracted by alkali consisted of relatively complex proteins, the formol nitrogen of this extract being the same as that of the aqueous extract. Total ash was most soluble in alkali, less in acid, and least in water, but the silicic acid was extracted in greatest amounts by alkali and by hot water. Phosphoric acid, lime, and magnesia were extracted in greatest quantity by acid media. Water extracted the least colouring matter and the extracts developed least turbidity; the alkaline extracts were extremely dark. "Testinic" acid was prepared according to Moufang's directions and found to be a complex of protein, tannin, and ash, the ash containing about 50% of silicic acid; reference is made to a similar complex noted in beer by Hartong (B., 1930, 836). Preliminary separation of the tannins was effected. Comparative brewings were made with a barley steeped normally and with a preliminary steep for 3 hrs. in 0.2% caustic soda at 45°. In the latter case the wort contained slightly less non-coagulable nitrogen, and yielded a paler, better coloured beer, of superior flavour.

F. E. DAY.

[Wine] must in 1929 from the wine-growing districts Pillnitz, Lössnitz, Meissen, and Seussnitz. A. HEIDUSCHKA and C. PYRIKI (Z. Unters. Lebensm., 1930, 59, 613—615).—The grape harvest for 1929 was small in quantity, but of good quality. The range of density of the must was 60.7—88.4° Oechsle, that of the titratable acids 4.4—12.8 g./litre, and of the mineral constituents 2.64—5.04 g./litre. As compared with the preceding year, the average density was 4.2° higher, the acid 2.7 g./litre lower, and the mineral content 0.42 g./litre higher for the year 1929. H. J. DOWDEN.

[Wine] must in 1929 from the wine-growing districts of Nahe and Glan, Rheintal, the Rhine district, Lahn, Rhine, and Main. A. RÖHLING (Z. Unters. Lebensm., 1930, 59, 629—631).—The must densities and acidities for some 360 varieties are tabulated. The influences of the severe winter are shown chiefly in a reduced crop, although the quality was excellent. The wine was the best since 1921. In the Wiesbaden district the musts showed a high sugar or alcohol content with correspondingly lower acid content.

H. J. DOWDEN.

[Wine] must in Baden for the year 1929. F. MACH and M. FISCHLER (Z. Unters. Lebensm., 1930, 59, 617—628).—The influences of the severe winter and the ensuing seasonal vagaries on the grape harvest in 1929 are described and analyses of a large number of varieties of wine are statistically examined. The most noticeable features are the high density and the low acidity of the must.

H. J. DOWDEN.

Sugars in port wines. C. F. MUTTELET (Ann. Falsif., 1930, 23, 205—207).—In view of the statement that port wine contains sucrose, three genuine samples

were examined. No sucrose could be detected, but there were found about 4% of dextrose and 5% of levulose.

E. B. HUGHES.

Lactic acid in Hérault wines. E. HUGHES and R. CHEVALIER (Ann. Falsif., 1930, 23, 214—216).—The lactic acid content of 80 normal samples of wine varied from 0.64% to 2.88% (average 1.5%). Wines from the lees were a little richer in lactic acid, whilst in the case of spoiled samples the amounts found were 0.5% higher than in normal samples. Whereas the lactic acid content is considered important in oenology, no limit could be fixed beyond which it would be necessary to take account of the amount present. E. B. HUGHES.

Determination of hydrogen cyanide in kirsch. T. VON FELLEBERG (Mitt. Lebensm. Hyg., 1930, 21, 43—52; Chem. Zentr., 1930, i, 3111).—The spirit (40 c.c.) is vigorously shaken with 5 c.c. of silver nitrate solution (1 c.c.  $\equiv$  0.5 mg. HCN), then with 2.5 c.c. of *N*-ammonia solution, followed by 3.5 c.c. of *N*-nitric acid. After filtering and washing the precipitate with 0.1% nitric acid the filter and precipitate are evaporated with concentrated ammonia solution (1—2 c.c.) and boiled with water until all the ammonia is removed. The silver cyanide is dissolved in concentrated nitric acid, and the silver determined by titration with ammonium thiocyanate in presence of iron alum.

A. A. ELDRIDGE.

Detection of isopropyl alcohol in spirituous liquors. K. BODENDORF (Z. Unters. Lebensm., 1930, 59, 616—617).—The method of Böhm and Bodendorf (B., 1930, 531) has been applied in the case of a large number of commercial spirits, wines, and liqueurs. The sample (5 c.c.), diluted with an equal vol. of water, is distilled, and, after about 5 c.c. have been collected, 1—2 c.c. of the distillate are diluted with a double volume of water, shaken with about 0.2 g. of charcoal, and filtered. A portion of the filtrate is floated on a few c.c. of a freshly prepared solution of *m*-nitrobenzaldehyde in concentrated sulphuric acid (1 g. in 50 c.c.), when, if isopropyl alcohol is present, an intense red coloration develops at the junction on warming for a short time. Concentrations of 0.1% may be detected and positive results were obtained with all adulterated samples, whilst with pure liquors the test was completely negative.

H. J. DOWDEN.

Measurement of the colour of red wines. I. Roos (Ann. Falsif., 1930, 23, 207—211).—Two colorimeters are described for measuring tint and quantity of colour. A range of standards is made from varying proportions of potassium permanganate and dichromate, and the tint is found by matching the wine against these. For intensity of colour, 1 c.c. of wine is diluted with water until it matches the standard. E. B. HUGHES.

Polarographic examination of vinegar with the dropping mercury cathode. J. HEYROVSKÝ, I. SMOLÉR, and J. STASTNÝ (Vestn. Českoslov. Akad. Zem., 1930, 6, 490—500; Chem. Zentr., 1930, ii, 641).—Reproducible results are automatically recorded. Fermentation vinegar contains acetaldehyde, as intermediate product of bacterial alcoholic fermentation, and surface-active substances.

A. A. ELDRIDGE.

"Elmocid" disinfection. STOCKHAUSEN.—See XXIII.

## PATENTS.

**Filter plugs [for wine casks].** K. PRILLINGER (B.P. 339,761, 18.11.29).—A plug for allowing purified air to enter the casks comprises strata containing various combinations of salicylic acid, potassium dichromate, alcohol, potassium permanganate, charcoal, and calcium cyanamide. Finally a portion of the air is bubbled through sulphurous acid. B. M. VENABLES.

**Production of wort.** F. O. LANDTBLOM, Assr. to LAVAL SEPARATOR Co. (U.S.P. 1,770,411, 15.7.30. Appl., 30.4.25. Swed., 24.6.24).—See B.P. 233,321; B., 1926, 251.

## XIX.—FOODS.

**Chemical composition of authentic samples of whole-wheat flours and modified whole-wheat flours.** L. H. BAILEY and S. C. ROWE (J. Assoc. Off. Agric. Chem., 1930, 13, 503—506).—Six varieties of wheat were milled and portions of the flour from each variety were modified (a) by sifting to effect a 10% (by wt.) removal of bran, and (b) by a 10% (by wt.) addition of bran. Analysis of the series of whole-wheat and the modified flours showed that with each variety of wheat the greatest differentiation between the grades of flour was shown by the crude fibre content, the pentosan determination being next in importance. The ratios of starch to crude fibre, starch to ash, and starch to pentosans also are significant. The maximum and minimum values for the fibre content of the whole-wheat flours were 2.56 and 2.23%, respectively, and those for whole flour less 10% of bran were 1.62 and 1.21%, respectively, whilst the corresponding pentosan values were: max. (min.) 7.46 (6.36)% and 5.70% (4.77)%, respectively. There is thus a dividing line between the two grades of flour. H. J. DOWDEN.

**Detection of corncockle in flour and bread.** R. FISCHER and E. RIEDL (Z. Unters. Lebensm., 1930, 59, 595—598).—Two methods are described for the detection of corncockle. An aqueous suspension of flour (5 g. in 20 c.c.) is prepared, and after keeping for 6—20 hrs. it is filtered and the filtrate centrifuged. The clear liquid is examined for saponin by the method of Kofler and co-workers (cf. B., 1930, 166), in which the liquid is capillarised by strips of filter paper having a band of cholesterol with which the saponin combines. After washing, the saponin-cholesterol is dried, decomposed by boiling with xylene, and tested with blood-gelatin, the hæmolytic zone indicating the presence of *Agrostemma* saponin. Concentrations as low as 1:17,000 may be detected by this method. In the examination of bread the sample is extracted with alcohol-chloroform, the extract being evaporated to dryness and, after dissolving the residue in hot water, the solution is examined as described. Concentrations of 1 in 2000 may be detected. When the content is 0.05—0.1% a second method enables macroscopic fragments to be identified. The sample of flour (0.01—0.02 g.) is mixed with 1.5—2.0 c.c. of blood-gelatin and examined between glass plates. After  $\frac{1}{2}$ —1 hr. circular hæmolytic areas are visible to the naked eye and fragments of seed are visible under the microscope. H. J. DOWDEN.

**Iodometry of milled [food] products.** II. K. FUCHS, W. RUZICZKA, and E. KOHN (Z. Unters. Lebensm.,

1930, 59, 573—585; cf. B., 1930, 790).—Further series of flours, brans, and fats have been examined, and the observation that the iodine value increases with degree of grinding has been confirmed. In a series of wheat and rye flours the iodine and periodine values followed closely the degree of grinding and with greater sensitivity than the fat content. The values for flours of the same grade from different mills agreed fairly well; those for brans were, however, less consistent. Bleaching has but little influence on the iodine or periodine values, so that the degree of grinding can still be followed in bleached flours. Meals from oats, barley, maize, and leguminous materials were examined by the same method with satisfactory results. In a series of wheat flours the  $p_H$  was found to decrease with increased fineness of grinding. H. J. DOWDEN.

**Detection of rice hulls and sawdust in bran and sharps.** C. RAUNIER and II. PAU (Ann. Falsif., 1930, 23, 229—233).—Rice hulls and sawdust give different colours from those given by bran and sharps when treated with iodine or with dimethyl-*p*-phenylenediamine sulphate or with phloroglucinol and phosphoric acid. The amount of adulterant is determined by calculation of the ratio of ash to silica, the figures obtained being for bran 7.78, sawdust 2.25, rice hulls 1.05. To characterise the adulterant, parallel colorimetric tests are made on the sample and on mixtures made according to the results calculated from ash and silica content. E. B. HUGHES.

**New procedure in the chemical investigation of fodder and foodstuffs.** J. KÖNIG [with F. BARTSCHAT and B. STEMPPEL] (Z. Unters. Lebensm., 1930, 59, 564—572).—The deficiencies in the normal methods of recording the constituents of foodstuffs are enumerated and a systematic scheme is presented for analysis and separation into the following groups:—water content; fat (ether extract); crude protein (amide + pure protein, both water-soluble and soluble or insoluble in 2% hydrochloric acid); water-soluble carbohydrates (acid as lactic acid + sugar, reducing powers before and after inversion + dextrin + residue); starch; proto- and hemi-celluloses (including hemipentosans); orthocellulose and ortholignin (including orthopentosans); insoluble lignin, cutin, and suberin; mineral matter (water-soluble and soluble or insoluble in 2% hydrochloric acid). The results of analyses of wheat, flour, bran, etc. are tabulated. H. J. DOWDEN.

**Rapid determination of dry gluten.** E. BERLINER and R. RÜTER (Z. ges. Mühlenw., 1930, 7, 1—4; Chem. Zentr., 1930, ii, 487).—The dry gluten of 14 wheat flours had  $d$  1.325, whence the weight of dry gluten is equal to the under-water weight multiplied by 4.08. A. A. ELDRIDGE.

**Significance of the coloration of flours by hydrochloric acid.** H. KÜHL (Z. ges. Getreidew., 1930, 17, 101—105; Chem. Zentr., 1930, ii, 1007).—The colour developed by the action of hydrochloric acid on flour is due to protein degradation products containing aldehyde or keto-groups. A. A. ELDRIDGE.

**Ferric chloride reaction of aqueous extracts of cereals. Detection of rye flour in wheat flour.** H. KÜHL (Z. ges. Getreidew., 1930, 17, 122—128; Chem. Zentr., 1930, ii, 1299).—Soluble carbohydrates



affect the reaction between ferric chloride and proteins. Characteristic colour reactions are obtainable only when definite quantity ratios are employed. Rye flour in wheat flour can be determined by observing the degree of turbidity produced when an extract is treated with ferric chloride and kept for 2 min. in a water-bath.

A. A. ELDRIDGE.

**Abnormal sweetened condensed milk.** S. DIXON and J. H. SUGDEN (Analyst, 1930, 55, 749—751).—Two samples of sweetened condensed milk were found to have a gelatinous consistency and to contain "buttons" of a reddish-brown colour. These characteristics coupled with a high content of invert sugar (3.9%) were ascribed to the presence of micro-organisms of the *Aspergillus* genus, the development of which had been favoured by storage of the milk at too high a temperature.

H. J. DOWDEN.

**Determination of milk fat.** A. C. ANDERSEN (Z. Unters. Lebensm., 1930, 59, 600—602).—In the determination of butter fat in milk which is beginning to decompose, separation of the fat under alkaline conditions, as in the Röse-Gottlieb method, is liable to lead to low results owing to saponification even when potassium dichromate has been added as preservative. It is preferable to use the Schmid-Bondzynski-Ratzlaff method, 10 g. of the milk being mixed with 10 c.c. of hydrochloric acid ( $d$  1.19) and boiled for 4—5 min., the heating being as gentle as possible in order to avoid discoloration. After cooling, the mixture is extracted with 10 c.c. of alcohol, 25 c.c. of ether, and 25 c.c. of light petroleum according to the normal Röse-Gottlieb method.

H. J. DOWDEN.

**Detection of viscogen [calcium saccharate] in cream.** G. T. PYNE (Analyst, 1930, 55, 747—749; cf. B., 1929, 735).—The detection of viscogen in cream by the high calcium content of the ash is troublesome, whilst if souring has commenced  $p_H$  determinations are of little value. The thickening effect of viscogen may be detected, however, by the decrease in viscosity produced by the addition of a soluble oxalate, which converts the gelatinous calcium phosphate into crystalline calcium oxalate with redissolution of the precipitated casein.

H. J. DOWDEN.

**Factors influencing the growth of moulds in butter.** H. MACY (Minn. Agric. Exp. Sta. Tech. Bull., 1929, No. 64, 86 pp.).—The growth of moulds isolated from butter in media containing butter constituents or their decomposition products is examined. The partial removal of carbon dioxide from the atmosphere does not prevent the growth of moulds, but none developed in the absence of oxygen. The growth of moulds in butter is largely controlled by atmospheric humidity and temperature, the oxygen supply, and the concentration of salt present.

A. G. POLLARD.

**Detection of the onset of decomposition in meat by the content of ammonia in the form of salts.** F. M. LITTERSCHEID (Z. Unters. Lebensm., 1930, 59, 599—600).—The influence of ammonia gas (from a defective cold-store) on the appearance and quality of meat is described. Such contamination may be detected by testing the rinsing water with Nessler's solution. In conducting the Glassmann-Rochwarger test (cf. B., 1930, 530) it is recommended that the outer and inner

portions of the meat be examined separately in order to determine whether the meat has been exposed to ammonia gas or has been dressed with ammonium salts as preservatives.

H. J. DOWDEN.

**Red colour in sardines in oil.** G. HINARD and M. BOURY (Ann. Falsif., 1930, 23, 216—218).—It was found that towards the end of the packing season in a few of the boxes the colour of the oil has a reddish tint and in such boxes is generally found at least one sardine having red-brown spots. It is considered that the red colour is probably due to a particular condition of the natural oil of the fish and not to any fault in, or effect of, the packing.

E. B. HUGHES.

**Photoactivity of honey.** J. STITZ (Z. Unters. Lebensm., 1930, 59, 606—607).—The possibility that the radium reported to be present in the ash of honey may be accounted for by the photoactivity of potassium compounds has been investigated. Samples of natural honey and of the ash were placed in glass or quartz containers and stored for 1 month in contact with photographic plates. Other samples were irradiated with ultra-violet light and Röntgen rays and similarly exposed. In all cases the results were negative, and no evidence of radioactivity was manifested.

H. J. DOWDEN.

**Fruit jellies. VI. Rôle of pectin. Extraction of pectin from pectic materials.** P. B. MYERS and G. L. BAKER (Delaware Agric. Exp. Sta. Bull., 1929, No. 160, 64 pp.; cf. B., 1928, 138).—The jellying power of pectin reaches an optimum when extracted at  $p_H$  2.4 (approx.), the optimum point being independent of the total titratable acidity of the extracting solution and of the nature of the acid used. Under conditions of greater acidity the rapid decline of jellying power is indicative of pectin hydrolysis. The decline of jellying power with increasing time of boiling during extraction is irregular. Jellying power is not related to the methoxyl content of the pectin. No relationship exists between the yield of pectic acid from pectin and its jellying power, since pectic acid is also produced by alkaline hydrolysis of the decomposition products of pectin. Viscosity measurements can only be accepted as criteria of the jellying power of pectins extracted by the same method. The yield of pectin depends on the  $p_H$  of the extracting solution (maxima  $p_H$  2.0 for tartaric acid and  $p_H$  1.45 for hydrochloric acid) and on the period of boiling (maximum at 120 min.), but the optimum jelly units is reached after 30 min. A method for the extraction of pectin at  $p_H$  2.15 after boiling for  $\frac{1}{2}$  hr. is described.

A. G. POLLARD.

**Jelly test for jellying fruit juices and pectin preparations.** L. GERET (Mitt. Lebensm. Hyg., 1930, 21, 116—117; Chem. Zentr., 1930, ii, 834).—The fruit juice or pectin solution (15 c.c.), mixed if necessary with the corresponding quantity of fruit acid, is mixed with 5 c.c. of 95% alcohol and then kept for 1 hr. at 0°, when a good sample may be removed as a single rod.

A. A. ELDRIDGE.

**Behaviour of pure natural and artificial fruit essences for lemonades towards the sodium salt of *p*-toluenesulphonchloroamide (chloramine-Heyden).** A. MIERMEISTER (Z. Unters. Lebensm., 1930, 59, 585—594).—The method of Tillmans and Hollatz (cf. B., 1929, 926) for differentiating artificial and natural

fruit extracts has been applied to a number of essences used in the manufacture of still and aerated beverages. For the direct titration, 1 c.c. of essence was mixed with 20 c.c. of water, 50 c.c. of 0.01*N*-chloramine solution, and 0.5 c.c. of 10% acetic acid, and, after keeping for  $\frac{1}{2}$  hr., the excess of chlorine was determined iodometrically. Whilst, in general, artificial essences were characterised by a high chloramine value, there were certain exceptions. Further evidence was afforded by steam-distillation followed by ether-extraction of the residue. With the exception of citrus fruit essences the natural products contain but small amounts of volatile aromatic substances and the ethereal extracts have a high chloramine value, whilst in the artificial essences some are completely volatile and others only partly so. The aqueous residue after ether-extraction possessed an aroma in the case of certain artificials due to the addition of vanilla extracts as a "fixative." The commonly used citric, malic, and lactic acids do not affect the chloramine titrations, but colouring matters do, and these have to be removed by absorption with wool. The chloramine values of some 22 natural and artificial products are given, with notes on their identification by means of the behaviour after distillation or extraction with ether.

H. J. DOWDEN.

**Spinach for canning.** F. W. GEISE (Maryland Agric. Exp. Sta. Bull., 1930, No. 320, 303—326).—Seasonal effects on the quality and chemical composition of spinach are examined. As the age of the leaves increased there was a decrease in free reducing substances, an increase in total and invert sugars, and a decrease in acid-hydrolysable matter. Variations in crude fibre content could not be correlated with seasonal conditions or period of harvesting. The total nitrogen content was greatest in young leaves. Storage of leaves at 40° and 60° led to losses of total and invert sugars. In leaves stored at above 0° there was a loss of acid-hydrolysable matter accompanied by an increase in crude fibre. A minimum period of storage prior to canning is recommended.

A. G. POLLARD.

**Determination of starch in vegetable preserves and other pasty products.** T. VON FELLEBERG (Mitt. Lebensm. Hyg., 1930, 21, 78—79; Chem. Zentr., 1930, i, 3114).—The preserve (10 g.) is boiled with water (5 c.c.), filtered off through cloth, and gently pressed; the procedure is repeated twice, using 20—30 c.c. of water each time, until the residue gives no blue colour with iodine and potassium iodide. The turbid filtrate is treated with 0.1 vol. of 50% calcium chloride solution and excess of 0.1*N*-iodine in potassium iodide solution, the starch iodide being then centrifuged. Sodium hydrogen sulphite is added, the acid is neutralised with calcium carbonate, and the mixture is boiled with calcium chloride and filtered through cellulose pulp, an aliquot part of the filtrate being treated with iodine and calcium chloride solution. Further procedure is as previously described (B., 1928, 833). A. A. ELDRIDGE.

**Effect of soda and soap on aluminium.** BOHNER. **Resistance of aluminium to fatty acids.** CLAUS. **Aluminium alloys.** STERNER-RAINER.—See X. **Cacao butter.** FIELD.—See XII. **Banana plantations.** EASTWOOD. **Oranges.** COPEMAN.—See XVI. **Determination of starch in potatoes.** LAMPE.—See XVII.

**Bakers' yeast.** ROSENBAUM.—See XVIII. **Emulsions.** SMITH and others.—See XX.

#### PATENTS.

[Machine for] preparation of eggs. A. BEATTIE and A. A. LYON (B.P. 340,543, 2.7.29).

**Silver-copper-nickel alloy** (U.S.P. 1,757,507).—See X.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Iron and ammonium citrate, B.P.** A. J. JONES and N. GLASS (Quart. J. Pharm., 1930, 3, 488—498).—This preparation should be neutral ( $p_H$  7.5), and should contain 7—8%  $NH_3$  and 53—57%  $C_6H_5O_7$ . A series of ten commercial samples has been examined: in 7 cases the sample contained <30%  $Fe_2O_3$ , and in the others below the B.P. minimum (31%). Iron was determined iodometrically. The scales (0.5 g.) are dissolved in water, hydrochloric acid ( $d$  1.16) is added, and the mixture brought to boiling and cooled. Potassium iodide is added and, after 3 min., the solution is diluted and titrated with thiosulphate. Ferrous iron, if present, is originally oxidised by permanganate. Samples may be satisfactory according to B.P. requirements, and yet may give precipitates in solution with magnesium sulphate. The cause of such precipitation remains obscure and requires further elucidation. R. CHILD.

**Determination of alkalinity and silver content of protargol.** (MIES.) A. L. DRAGENESCO and E. WEINBERG-SACHETTI (J. Pharm. Chim., 1930, [viii], 12, 536—539).—Samples of protargol and silver proteinate, although corresponding to Pharmacopœia requirements, caused irritation when used as eyedrops, due, it is thought, to excessive alkalinity. The dark colour of their solutions prevents a direct determination of their reaction, but pretreatment with neutral sodium thiosulphate enables the alkalinity to be titrated in the ordinary way with 0.1*N*-sulphuric acid, using phenolphthalein as indicator. Samples of protargol were found to be less alkaline than those of silver proteinate. In the determination of the silver content, oxidation of the organic matter by potassium permanganate and sulphuric acid was found to give higher and more consistent results than precalcination. T. McLACHLAN.

**Variation of sp. gr. and refractive index with concentration of camphor and alcohol in Spiritus camphoræ, B.P.** F. H. MILNER (Quart. J. Pharm., 1930, 3, 338—341).—Owing to natural variations in the optical rotation of camphor, the determination of camphor in spirit of camphor by this means is inaccurate unless the rotation of the camphor used be known. From a number of determinations of sp. gr. and refractive index of spirit of camphor, prepared to contain different percentages of camphor and with alcohol of varying concentration, it is shown that the former factor is the better criterion. Allowing for a variation in camphor content of from 9.5 to 10.5% wt./vol., and in the alcohol concentration from 89 to 91% wt./vol., the possible variations are:  $d_{15.5}^{20}$  0.8438—0.8503,  $n_D^{20}$  1.3746—1.3759. Since increase of water content increases  $d$  but reduces  $n$ , whilst loss of camphor decreases both values, a combination of the two determinations affords a useful means of testing the preparation.

R. CHILD.

**Spiritus saponatus and Spiritus saponis kalini, D.A.B. VI.** W. MEYER (Arch. Pharm., 1930, 268, 572—585).—Analyses of samples of (a) Spiritus saponato-camphoratus, (b) Spiritus saponatus, and (c) Spiritus saponis kalini, show that, although these conform to the D.A.B. VI requirements and have been produced by official methods, they may exhibit considerable variations in their content of alcohol, free alkali, unsaponified oil, and fatty acid, according as an olive oil or a linseed oil soap has been employed. The following additional standards are proposed for these preparations:  $d^{20}_4$ : (a) 0.912—0.920; (c) 0.930—0.940; minimum fatty acid content: (a) 6.5%, (b) 9.3%, (c) 19.5%; iodine value of fatty acids: (a) and (b) 93—104, (c) 190—210; reaction: (a) and (b) neutral to phenolphthalein, (c) neutral after addition of 1 c.c. of 0.1N-hydrochloric acid; minimum spirit content: (c) 42.4%; minimum alcohol value: (c) 5.7. Methods for the determination of these values are described. H. E. F. NOTTON.

**Assay of stramonium leaves and tincture of stramonium.** C. M. CAINES (Quart. J. Pharm., 1930, 3, 342—348).—The following method (which avoids the emulsification occurring in the B.P. process) is recommended. Stramonium leaves (10 g.) in No. 60 powder, after moistening first with ether-chloroform mixture and then with dilute ammonia, are percolated with 4:1 ether-chloroform to remove total alkaloids. The percolate is concentrated and transferred to a separator, together with chloroform washings; 90% alcohol is added and the mixture extracted with 0.2N-sulphuric acid until the alkaloids are removed. The combined acid extracts are washed with chloroform, basified with ammonia, and alkaloids are extracted with successive quantities of chloroform. The chloroform extracts after washing with water are evaporated, and the residue is dissolved successively in ether and absolute alcohol, the solvent being evaporated each time. The residue, dried for 30 min. at 100°, is dissolved in absolute alcohol, 0.02N-sulphuric acid added, and excess of acid titrated against methyl-red with 0.02N-sodium hydroxide (1 c.c. of 0.02N-acid  $\equiv$  0.005785 g. of alkaloids as hyoscyamine). A similar method is used for the assay of the tincture as for the percolate above. After shaking, the chloroform is separated and the extraction repeated with two lots of chloroform (10 c.c. each); alcohol (15 c.c. of 90%) is added to the combined chloroform extracts, which are then treated with 0.2N-sulphuric acid and the determination completed as described above. R. CHILD.

**Determination of moisture in tobacco by the Steich-hygrometer.** N. M. MILOSLAVSKI and A. I. PALANT (Ukraine Chem. J., 1930, 5, 117—125).—The Steich-hygrometer, for the determination of moisture in stored goods, consists of a hair-hygrometer through which air is drawn, relative humidity being read from the scale. Results given by it do not agree with those obtained analytically. There is no simple relationship between moisture content of tobacco and the R.H. of air. The moisture content depends on the biological state, absorptive capacity, and chemical constitution of the leaf. The hygrometer is therefore inapplicable in the case of tobacco. E. B. UVAROV.

**[Tobacco]-smoking, nicotine limits, and the determination of nicotine by Pfyl and Schmitt's method.** F. BOLM (Z. Unters. Lebensm., 1930, 59, 602—606).—Apart from minor modifications the method of Pfyl and Schmitt (cf. B., 1927, 955) has been found entirely satisfactory and the observations on the influence of rate, intermittency, etc. of the smoking on the nicotine content of the smoke have been confirmed. The results of analysis of so-called "nicotine-free" tobacco and cigarettes are given, both as regards the total nicotine and the amount evolved in the smoke. None of the treatments was found to fix the nicotine in the tobacco and give a nicotine-free smoke, and hence determination of total nicotine in the tobacco is alone sufficient in judging the tobacco. H. J. DOWDEN.

**Potentiometric assay of cinchona.** J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1930, 19, 1299—1303).—A potentiometric method for the evaluation of cinchona alkaloid residues is described. Either the quinhydrone or the hydrogen electrode may be used and the shaking-out process is unnecessary. E. H. SHARPLES.

**Mandelin's test for strychnine.** C. F. POE and D. W. O'DAY (J. Amer. Pharm. Assoc., 1930, 19, 1292—1299).—About 500 organic compounds have been tested with Mandelin's reagent (ammonium vanadate-sulphuric acid) under varying conditions. A number give colour reactions similar to strychnine, but very few completely mask the test when present in small amounts. None of the aliphatic compounds tested gave the reaction, but no special group seems to be responsible for the test, and the reaction should be checked by other characteristics. E. H. SHARPLES.

**Determination of alkaloid in Extractum belladonnae siccum cum rad. liquirit. paratum 1 + 1.** G. BÜMMING (Arch. Pharm., 1930, 268, 590—592).—Application of the D.A.B. VI and B.P. 1914 methods for determining the alkaloid in Extractum belladonnae to dry extracts containing liquorice powder or extract gives consistently low results, although a mixture of atropine and lactose gives a correct result. Commercial samples which give correct results must therefore be assumed to contain an excess of alkaloid.

H. E. F. NOTTON.

**Transmission spectra of vanilla extracts.** E. H. HARVEY (Amer. J. Pharm., 1930, 102, 659—661).—The visual transmission spectra of extracts of vanilla beans from different sources and of the adulterants, caramel and prune juice, have been measured. All show general absorption of varying intensity in the violet and green regions, the differences being insufficient to allow the source of the bean to be distinguished.

H. E. F. NOTTON.

**Vanilla sugar.** J. J. J. DINGEMANS (Chem. Weekblad, 1930, 27, 694).—None of the usual colour reactions for vanillin serves to differentiate between this substance and "vanirom" (protocatechualdehyde ethyl ether). If to the solution in hot water of the residue from the ether extract of 10 g. of vanilla sugar a solution of hydrazine sulphate, followed by 4N-hydrochloric acid, be added, "vanirom" yields a citron-yellow precipitate, whereas vanillin yields an orange-red precipitate. The two compounds may be readily distinguished even at high dilutions. H. F. GILLBE.

**Evaluation of chlorophyll and chlorophyll preparations.** U. DREHMANN (Arch. Pharm., 1930, 268, 585—589).—Chlorophyllin and commercial samples of chlorophyll containing copper do not show the red fluorescence spectrum excited by ultra-violet light in solutions of pure chlorophyll. Examination of the absorption and fluorescence spectra of extracts shows that among nineteen pharmaceutical preparations stated to contain chlorophyll, only three contain it in notable quantity. H. E. F. NOTTON.

**Emulsification. I. Examination and analysis of pharmaceutical emulsions. II. Manufacture of pharmaceutical emulsions. III. A factor inhibiting the emulsification of cod-liver oil.** E. L. SMITH (Quart. J. Pharm., 1930, 3, 354—361, 362—372, 373—374).—I. [With G. N. GRINLING.] The most satisfactory criterion of the degree of subdivision of the disperse phase of an emulsion is considered to be the number of globules into which 1 c.c. of the oil is subdivided; a convenient method is described, employing the Thoma haemocytometer, by which this may be measured. Simple analytical methods are given by means of which the nature and amount of the oil and emulsification agents may be determined.

II. [With (Miss) V. HAZLEY.] Gum acacia is preferred as an emulsifying agent for edible emulsions, with gum tragacanth as thickener. Factors influencing the "creaming" of emulsions are discussed, and the methods of combating this are considered; details are given for the large-scale preparations of a typical emulsion, based on the foregoing results.

III. The oxidation or "drying" of cod-liver oil produces a substance capable of stabilising water-in-oil emulsions and of inverting or reducing the stability of oil-in-water emulsions. The suitability of a cod-liver oil for the preparation of emulsions may be estimated by measurement of the interfacial tension between the oil and water by the drop-pipette method. R. CHILD.

**Diatomite, its analysis and use in pharmacy.** N. I. HENDEY (Quart. J. Pharm., 1930, 3, 390—407).—It is suggested that fossil deposits of diatomaceous character be termed "diatomite" or "diatomaceous earth," and that local names (kieselguhr, tripolite, etc.) be avoided. Methods are given for the chemical, physical (colour, moisture, grit determination, porosity, and apparent  $d$ ), and microscopical examination of the product, and recommendations are made of the types of diatomite (as classified by such examination) best adapted for certain purposes. R. CHILD.

**Extraction of odoriferous substances from flowers.** I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst., Moscow, 1930, No. 22, 130—139).—Light petroleum, b.p. 60—70°, is preferred, and prolonged extraction is avoided. The odour is influenced by the hour of collection of the flowers. CHEMICAL ABSTRACTS.

**Testing ether for aldehyde.** GREEN and SCHOETZOW.—See III. **Arsenic in barium and bismuth salts.** GREEN and SCHOETZOW. **Arsenious iodide.** HUSA.—See VII.

#### PATENTS.

**Manufacture of physiologically active hormone preparations.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,943, 10.9.29).—The activity of thymus

gland preparations is measured by observing the occurrence or failure of rutting phenomena on administering the extract to immature female animals; e.g., a given extract in Ringer's fluid may be injected and from the result the extract can be adjusted to any desired activity (rat units/c.c.) by dilution or concentration *in vacuo*.

E. H. SHARPLES.

**Treatment of physiological or pathological liquids containing albumin, or of used bandages etc.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,473, 1.5.29. Cf. B.P. 334,876; B., 1930, 1167).—The condensation products (diarylmethanes, diarylbenzenes, etc.) from aldehydes or ketones and chlorinated or brominated phenols or cresols are added to the liquids or to the material (except wool) to prevent attack by bacteria, moulds, etc. C. HOLLINS.

**Preparation of thiosemicarbazones of hydroxy-arsenobenzenes.** K. STREITWOLF and A. FEHRLE, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,785,660, 16.12.30. Appl., 11.7.28. Ger., 21.7.27).—See B.P. 294,263; B., 1929, 339.

**Generation of chlorine** (U.S.P. 1,767,676).—See VII.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Determination of so-called primary silver in photographic gels.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 28, 293—297).—The amounts of free silver present in unwashed and washed shredded silver chloride emulsions fixed in sodium thiosulphate or sodium sulphite solution have been determined gravimetrically. Contrary to the results of Weigert and Lühr (B., 1930, 303), the silver values were the same as in blank determinations (gelatin shreds washed in used fixing solution) within experimental error. The very small amounts of free silver found are due to hydrolysis of silver halide (or complex formed in the fixing solution) during washing (cf. following abstracts).

J. LEWKOWITSCH.

**Silver values and fixing processes. II.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 28, 297—302).—Sodium thiosulphate solution cannot be used as a fixer in the determination of free silver in emulsions, since it dissolves appreciable amounts of colloidal silver. Sodium sulphite, the solvent power of which is negligible, forms a satisfactory fixer. Gravimetric determinations of the free silver in unfixed "synthetic" emulsions gave values of 0 and 0.0684 g. (per 50 g. of dry material) for unwashed and washed emulsions, respectively. Values of 0.0012 g. (a) and 0.0010 g. (b) were found for the corresponding fixed emulsions. The value (a) must be due to silver derived from the used fixing solution. Since the silver value (b) is smaller than that of the unfixed washed emulsion, some of the silver in the latter must have been present as silver oxide and dissolved by the sodium sulphite. The residual silver in the fixed emulsion is probably present as sulphide. (Cf. preceding and following abstracts.)

J. LEWKOWITSCH.

**Origin and chemical nature of silver values [free silver in emulsion]. II.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 28, 302—311).—Washed and unwashed synthetic silver chloride emulsions

were centrifuged and the fractions analysed gravimetrically. The results confirm previous conclusions (cf. preceding abstracts) that free silver is not present in unwashed emulsions, but is formed during the washing process by hydrolysis of silver halides; owing to differential diffusion of the ions in gelatin (comparable with their mobilities in water) halogen is lost; the silver is converted into oxide which forms a gelatin complex sparingly soluble in water but soluble in sodium sulphite solution. Passing from chloride to bromide and iodide emulsions, the apparent silver value decreases, as is to be expected from a consideration of the relative solubilities of the halides. J. LEWKOWITSCH.

**Ripening processes in silver bromide-gelatin emulsions, "primary silver," and lysidine as sensitiser.** J. M. EDER (Z. wiss. Phot., Schaum-Festschr., 1930, 29, 20—22).—Early theories suggesting the formation of primary or reduction silver nuclei during the ripening process receive support from recent analytical investigations on photographic emulsions. Lysidine added to a washed and melted emulsion causes a definite increase in sensitivity as shown by a lowering of the threshold value and an increase in gamma. The optimum quantity must be determined for each emulsion, and varies between 0.02% and 0.05% of the weight of emulsion. The sensitising effect is most marked with under-ripened emulsions and is almost negligible with those fully ripened by the ammonia process. The formation of silver bromide-lysidine complexes may play a part in the sensitisation.

J. W. GLASSETT.

**Amyl acetate in sensitometry.** K. KIESER (Z. wiss. Phot., Schaum-Festschr., 1930, 29, 87—90).—Commercial amyl acetate was fractionated and the suitability of the various fractions for sensitometric purposes was tested in a Hefner lamp, using a sample of amyl acetate recommended for this purpose as a standard of comparison. The fractions obtained at 120—133° and 138—144° gave a photographic intensity equal to that of the standard, whilst the 133—138° fraction gave a slightly higher value. For ordinary work the 120—145° fraction may be used. Measurements of the refractive index of each fraction indicated that this property could be used as a criterion of suitability for sensitometric work. The value at 15° should be 1.4000—1.4050.

J. W. GLASSETT.

**Early uses of light filters in practical photography.** E. STENGER (Z. wiss. Phot., Schaum-Festschr., 1930, 29, 43—46).—Historical. J. W. GLASSETT.

PATENT.

**Pretreatment of metal plates for production of [photozincograph] printing plates.** F. ULLMANN (U.S.P. 1,785,606, 16.12.30. Appl., 7.3.27. Ger., 10.3.26).—See B.P. 288,023; B., 1928, 503.

## XXII.—EXPLOSIVES; MATCHES.

**Black powder.** K. A. HOFMANN (Sitzungsber. preuss. Akad. Wiss., Berlin, 1930, 25, 432—436).—The ignition of mixtures of sulphur, charcoal, and saltpetre at 290—300°, as compared with 320° for charcoal-saltpetre, and 470° for sulphur-saltpetre mixtures, is due to formation at 150° of hydrogen sulphide. This gas decomposes the saltpetre at 285—290° to form sul-

phate and give a rise in temperature, so that the saltpetre fuses, and in this condition reacts with the charcoal and the melted sulphur. Ignition by sparks runs parallel with ignition by external heating. Under the fall-hammer, mixtures of sulphur, charcoal, and saltpetre are less sensitive than sulphur-saltpetre mixtures, the former igniting at 70—85 cm. and the latter at 45—50 cm., whilst charcoal-saltpetre mixtures are insensitive. Incorporation of graphite with the mixtures lowers their sensitiveness to shock, but superficial coating has no effect, the inference being that ignition is caused by electrically-produced sparks from friction of sulphur particles and crystals of saltpetre. Sulphur increases the amount of the explosion gases, but in absence of sulphur more carbon monoxide is formed. Mixtures containing charcoal give rise to hydrocyanic acid; to prevent this, powder used in mines should contain not less than 10% of sulphur. As regards the relative advantages of brown and black charcoal, it is found that with the former larger amounts of water vapours are developed, thus increasing the gas volume and lowering the temperature.

W. J. WRIGHT.

**Instability of colloidal powders.** H. MURAOUR (Bull. Soc. chim., 1930, [iv], 47, 1259—1264).—Experiments are described in support of the view that the instability of nitric esters is due to hydrolysis, catalytically accelerated by free nitric acid, derived from incomplete washing of the initial product or through the presence of a trace of an unstable nitric ester. Further confirmation of this view is drawn from a consideration of the results of Marquoyrol (B., 1929, 873) on the thermal decomposition of nitric esters. This is regarded as due to hydrolysis in addition to the normal thermal effect.

J. R. I. HEPBURN.

**Detonation of solid explosives.** P. LAFFITTE and M. PATRY (Compt. rend., 1930, 191, 1335—1337).—The author's method (A., 1924, ii, 472, 623) has been extended to the determination of explosion velocity by measurement of the angle produced by the front of a compression wave propagated in an explosion tube illuminated by a powerful arc lamp, in such a way as to render visible the change in refractive index. In the case of dynamite or mercury fulminate detonated in a glass tube 15 mm. in diam., a partial or complete opacity of the glass was observed (7000 m./sec.). In the air of the tube a non-luminous wave of compression or shock and a luminous column of gas are propagated simultaneously, at the same initial speed (about 7000 m./sec.), the latter, however, weakening more rapidly so that after 60 cm. the speeds are 2300 and 1750 m./sec., respectively.

J. GRANT.

**Testing of detonators.** A. HAD and H. KOENEN (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 393—398, 433—439, 463—468).—In applying the Esop test, the authors used charges of compressed trinitrotoluene phlegmatised with talcum. Detonators loaded with various compositions were tested with these charges on lead plates, 10 × 10 × 3 cm., in lead blocks, and in Kast's brisance apparatus, the detonators alone being also tested on 7-mm. lead plates. The results are tabulated. It was found that the initiating effect of detonators could be determined by any of the methods

described, and that it depends on the brisance. The percentages of talcum with which, on lead plates, the critical point was reached were 65, 50, and 25 for tetryl, trinitrotoluene, and fulminate detonators, respectively. Methods of measuring the dimensions of detonators, removing the composition, and determining the effects of wet and dry storage are briefly described.

W. J. WRIGHT.

**High brisance [of explosives] in relation to blasting efficiency and chemical constitution. II. Mannitol hexanitrate and pentaerythritol tetranitrate.** A. STETTBACHER (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 461—462).—The brisance, determined in iron-plate tests, of pentaerythritol tetranitrate ("Penthrite") mixtures is superior to that of mixtures containing mannitol hexanitrate ("Nitromannite"). This superiority is ascribed to the central carbon linking of the former, as compared with the open-chain constitution of the latter, this theory being confirmed by the high brisance of tetranitromethane-trinitrotoluene mixtures.

W. J. WRIGHT.

**Nitrocellulose.** BECK and others.—See V.

#### PATENTS.

**Blasting cartridges.** D. HODGE (B.P. 339,983, 14.9.29).—In blasting cartridges, in which gaseous carbon dioxide is generated by means of a heating element, solid, instead of liquid, carbon dioxide is employed, and the release of the gas is effected by means of a valve instead of a rupturable disc. W. J. WRIGHT.

**Electric blasting fuses and detonators.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,041, 24.9.29).—A non-inflammable sealing composition for electric detonators consists of chloronaphthalene (45—60% Cl) mixed with about 26% of sulphur, a non-inflammable substance (e.g., kieselguhr or talcum), which does not react with sulphur, being added if desired.

W. J. WRIGHT.

### XXIII.—SANITATION; WATER PURIFICATION.

**Germicidal efficiency of soaps and of mixtures of soaps with sodium hydroxide or with phenols.** J. M. SCHAEFFER and F. W. TILLEY (J. Agric. Res., 1930, 41, 737—747).—The bactericidal efficiencies of neutral soaps prepared from coconut oil, linseed oil, and castor oil were examined. Addition of sodium hydroxide increased the efficiency of coconut oil soaps in most cases. The toxicity of sodium laurate to *Pasteurella avicida* was decreased by the presence of excess of soda, but increased by excess of lauric acid. The bactericidal effect of phenol was increased by admixture with 50% of linseed, castor, or coconut soaps in the absence of other organic matter, but in the presence of milk or blood-serum differences were small. Germicidal soaps containing cresol or *o*-hydroxydiphenyl were efficient against *Staph. aureus* even where organic matter was present.

A. G. POLLARD.

**The "Elmocid" treatment; a new method in disinfection technique.** F. STOCKHAUSEN (Woch. Brau., 1930, 47, 533—538, 545—549, 561—565, 575—579).—The antiseptic action of solutions of inorganic bases and acids is first increased, then decreased, by the addition of successive amounts of salts of the alkali metals. Based on this fact, disinfecting solutions mar-

keted by the Elektro-Osmose A.-G. under the names of "Elmocid-alkalisch" and "Elmocid-sauer," consisting, respectively, of equimolecular proportions of sodium hydroxide and chloride, and of nitric acid and potassium nitrate, to be used at 0.1*N*-concentration, have been examined regarding their suitability for brewery practice. The alkaline preparation is for general use, the acid for aluminium and lacquered vessels. Laboratory and technical experiments show that they have a satisfactory cleansing action and antiseptic power. F. E. DAY.

**Water softening and purification methods.** J. A. MCGARIGLE (J. Amer. Water Works' Assoc., 1930, 22, 1609—1624).—The improved floc obtained by the use of mechanical agitators during the coagulation process enables the size of the settling basin to be reduced. The addition of clay prior to flocculation is often of value in facilitating colour removal. The advantages of the zeolite softening process in obtaining water suitable for use in modern high-pressure boilers are emphasised. The cost of this treatment is quoted at 5 cents per thousand gals. as against 55 cents for evaporators, and since calcium salts are absent it requires a smaller addition of phosphate to prevent corrosion fatigue than water softened by the hot lime-soda process. With the latter process the correction of the sulphate-carbonate ratio is apt to result in the formation of a hard calcium sulphate scale with consequent local overheating.

C. JEPSON.

**Purification of highly turbid waters.** J. D. FLEMING (J. Amer. Water Works' Assoc., 1930, 22, 1559—1576).—The usual methods adopted in dealing with highly turbid waters, e.g., those of the rivers Mississippi and Missouri, are described and compared with those in use at the 50,000,000-gal./day plant at St. Louis. When the raw water contains upwards of 1200 p.p.m. of suspended solids, presedimentation in specially designed tanks is recommended, as the reduced variation in turbidity will enable economies to be made in the use of chemicals. Presedimentation may be assisted by the introduction of sludge from the coagulation basins, and the control of the conditions promoting satisfactory coagulation is rendered more flexible by the use of variable-speed mechanical agitators.

C. JEPSON.

**Comparison of the Dominick-Lauter presumptive test with "Standard Methods" test for *B. coli* in water. II.** W. LEAHY (J. Amer. Water Works' Assoc., 1930, 22, 1490—1494).—Three hundred samples of water from 91 different sources were tested by these two methods. It is shown that the same number of positive results could be obtained by either method, but the percentage confirmation was much higher with the Dominick-Lauter medium (cf. B., 1929, 836) than with standard lactose broth, as of the total samples showing a positive presumptive test 100% were confirmed for the new test and only 26.9% for the standard method.

C. JEPSON.

**Chemical and mechanical utilisation of activated carbon in water purification.** A. S. BEHRMAN and H. B. CRANE (J. Amer. Water Works' Assoc., 1930, 22, 1399—1413).—Experience with many plants of all sizes suggests that the most economical function of activated carbon is in the removal of colour, odours, and tastes, e.g., excess of chlorine, from previously clarified and



filtered water, and particularly as a safeguard in small plants where adequate supervision is not always available. The material recommended is Hydrodarco, obtained from certain Texas lignites and containing 70% of pure carbon. Its density renders it suitable for use in upward-flow filters operating at rates up to 15 gals./sq. ft./min. Designs of plants utilising activated carbon or water treatment are described and discussed.

C. JEPSON.

**Activated carbon for removal of taste and odour [from river water].** G. D. NORCOM and R. I. DODD (J. Amer. Water Works' Assoc., 1930, 22, 1414—1437).—Small-scale experiments on Delaware River water show that from a raw water containing a wide variety of polluting materials, a drinking water can be obtained free at all times from appreciable odour or taste. The filters used were constructed from 4-in. diam. glass tubes and 8-in. diam. cast-iron pipes, and three types of activated carbon (Darco, Nuchar, and Minchar) were tested. When dealing with previously coagulated and filtered water Darco proved to be the most efficient and Minchar the least, although the latter, being the harder, would be the most suitable for use in place of filter sand. Odour is best removed by downward-flow units, but when possible the upward-flow method is preferred because of the decreased loss of head in operating and the tendency of the material to be self-cleansing. A rate of 2 gals./sq. ft./min. is recommended, and an effective life of about a year may be expected under conditions similar to those in the experiment. By passing steam through beds of Darco or Nuchar so as to maintain boiling heat for  $\frac{1}{2}$  hr. much of the substances previously absorbed by the carbon will be removed, but it is considered that revivification may be incomplete and the material may eventually need replacement.

C. JEPSON.

**Use of activated carbon in removing objectionable taste and odours from water.** J. R. BAYLIS (J. Amer. Water Works' Assoc., 1930, 22, 1438—1461).—A continuation of earlier work (cf. B., 1929, 624). An experimental carbon adsorption unit with a Hydrodarco carbon bed 24 in. thick and 10 ft.<sup>2</sup> in area continues to remove successfully over 1 p.p.m. of chlorine from filtered water at a rate of 2 gals./ft.<sup>2</sup>/min. after 17 months' operation. The water is applied in an upward direction and the bed has been washed twice in a similar manner to that used for rapid sand filters. Experiments carried out in glass tubes  $1\frac{1}{2}$  in. diam. indicate that (a) deep beds are more efficient than shallow ones; (b) the maximum rate of upward flow for 4—12-mesh Hydrodarco is about 4 gals./ft.<sup>2</sup>/min.; (c) the lower the rate of application, the larger is the volume of water dealt with per lb. of material; and (d) 1 lb. of Hydrodarco will reduce the residual chlorine in 30,000 gals. of water from 1.0 to less than 0.1 p.p.m. when operating at the 2 gals./min. rate without revivification, or 200,000—500,000 gals. if revivified when necessary.

C. JEPSON.

**Determination of silicic acid in water.** W. STEFFENS (Chem. Ztg., 1930, 54, 996—997).—The addition of ammonium molybdate to the acidified water yields a solution of silicomolybdate the yellow colour of which is compared with that of a standard solution

of either potassium chromate or of picric acid; the latter is considered to give a colour which permits the more accurate matching. A solution containing 50 mg. of silicic acid per litre is equivalent to 25.6 mg. per litre of picric acid dried *in vacuo*. C. RANKEN.

**Dust filters.** ALBRECHT.—See I.

PATENTS.

**Innocuous destruction of pests and germs.** T. GOLDSCHMIDT A.-G. (B.P. 318,898, 11.9.29. Ger., 11.9.28).—The inflammability of ethylene oxide or similar alkylene oxides used for this purpose may be reduced by dilution with inert substances, e.g., carbon dioxide or nitrogen, which are gases under normal temperature and pressure. Thus a solution of 1 pt. of ethylene oxide in 20 pts. of liquid carbon dioxide, contained under pressure in a cylinder, is claimed. [Stat. ref.]

C. JEPSON.

**Indicator of atmospheric pollution by illuminating and similar gases.** F. FREITAG (B.P. 340,195, 22.4.30. Ger., 24.4.29).—A contact layer of platinised asbestos is placed inside a U-shaped bow of two superposed metals of different coefficients of expansion. One end of the bow is fixed; the other, when stretched, e.g., by heat, makes contact in an electric circuit containing an alarm device. The heat produced by the oxidation of small quantities of coal gas, carbon monoxide, etc. in the presence of this layer is sufficient to give the warning signal. Greater sensitivity is attained if the catalyst be heated to 70°, by inserting it between the bow and a similar shaped silundum heating element directly connected to the electrical mains.

C. JEPSON.

**Softening of water [by base exchange].** A. ROSENHEIM (B.P. 314,067, 11.6.29. Ger., 23.6.28).—The de-solution of silicic acid from artificial base exchangers prepared from silicic acid sols (cf. B.P. 286,307; B., 1929, 962) may be prevented and the removal of silicic acid from water accomplished by increasing the proportion of the metal oxide present. The ordinary water-softening powers of the base exchanger when exhausted may be regenerated in the usual way, but the regeneration of the silicic acid-removing powers requires treatment with silicic acid-dissolving agents, e.g., aqueous solutions of alkalis, hot water, etc., under pressure, if necessary.

C. JEPSON.

**Carbonation [of water etc.].** P. W. SHIELDS (U.S.P. 1,768,158, 24.6.30. Appl., 20.10.23).—The water is sprayed into a vacuum chamber, whereby the dissolved air is removed, and is then pumped through a type of Venturi valve where a stream of carbon dioxide under pressure is introduced at the point at which the water is under maximum pressure. The reduction of pressure of the gas cools the water and thus aids absorption. The carbonated water passes to an agitation chamber, where the gas is stirred into the water by means of rotating paddles and finally the saturated water is drawn into reservoirs.

A. R. POWELL.

**Charcoal for dechlorinating water** (B.P. 316,269).—See II. **Acid waste liquors** (U.S.P. 1,765,424). **Silicious materials** (U.S.P. 1,755,496). **Base-exchange material** (U.S.P. 1,766,350). **Generation of chlorine** (U.S.P. 1,767,676).—See VII.



# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAR. 13 and 20, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Vapour pressure and heat of vaporisation.** P. G. NUTTING (Ind. Eng. Chem., 1930, 22, 771).—From the thermodynamical relation between internal thermal energy, mechanical work, and vapour pressure and the Clapeyron equation,  $dW/dm = RT/(C_v/C_l - 1)$ ,  $C_v$  and  $C_l$  being the vapour and liquid concentrations, there is obtained  $L = RT/M(d \log p/d \log T)(C_v/C_l - 1)$ , which checks with experimental data. The line obtained by plotting  $\log p$  against  $\log T$  corresponds well to the relation  $(\log T + a)(\log p + b) = C$ . The constants  $a$ ,  $b$ , and  $C$  for water, toluene, and carbon tetrachloride are given. Pressures calculated from these are in good agreement with those observed. Another new formula with a rational basis is  $L = A(T_c - T)^n$ , where  $T_c$  is the critical temperature;  $n$  is about 1/3 for latent heats. Several exact values of  $n$  are given, and the linear relation indicated holds well in all cases tried.

C. IRWIN.

**Fundamentals of three-roll grinding.** G. A. VASEL (Chem. and Met. Eng., 1930, 37, 759—761).—When possible the material should be used with a dispersing liquid having lubricating properties. The pressure along the length of the rolls should be uniform. The effects of an increase of pressure are: to increase the output to a maximum value, which then falls; to increase the size reduction, but if the pressure is too great separation of the solid from the liquid may occur; to increase the heat generation. Cooling water is used to prevent undue rise of temperature and keep it uniform along the length of the roll. The feed should be kept uniform; variation of the feed head between the centre and third rolls results in an uneven product. The minimum quantity of dispersing liquid should be used, as too much reduces the capacity of the machine. Increased relative and absolute speeds of the rolls increase fineness and uniformity. The output increases at a greater rate than the corresponding increase in roll diameter. Rolls should be driven from both ends to avoid distortion.

D. K. MOORE.

**Influence of insoluble powders on emulsion type.** J. F. CARRIÈRE (Chem. Weekblad, 1930, 27, 638—639).—An explanation of the empirical rule that the continuous phase is that which has the greater wetting power for the emulsifying powder.

S. I. LEVY.

**Dialysis, ultrafiltration, osmosis, and applications thereof.** A. BOUTARIC (Chim. et Ind., 1930, 24, 1295—1303).—The theory of filtration is discussed and the effect of pore size and of adsorption indicated. Every solvent requires its own membrane in dialysis. The

disadvantage of dialysis for the purification of colloids is the dilution of the colloidal solution. Ultrafiltration endeavours to remedy this by the use of pressure, whilst any kind of gel suitable to the liquid may be used as filtering medium; collodion is most often employed. The comparative significance of the capillarity and dissolution theories of dialysis are discussed. Osmosis in which crystalloids are also retained is common in nature. Its artificial production and the laws of osmotic pressure are summarised. Two theories of its mechanism can be propounded as with dialysis. The osmotic pressure of colloidal solutions relative to pure solvents cannot be determined in practice owing to the inevitable presence of crystalloid impurities in the filtering membrane. The osmotic pressure relative to such an intercellular liquid can, however, be measured. It is much less than with crystalloids and decreases with the size of the particles. The osmotic pressure of colloid solutions is not directly proportional to the concentration, varies in different directions with the temperature, and is greatly affected by  $p_H$ . Particle size can therefore only be determined very roughly.

C. IRWIN.

**Velocity ratio—chief factor in good mixing of gases.** T. H. CHILTON and R. P. GENÈREAU (Chem. and Met. Eng., 1930, 37, 755—758).—When a gas is introduced into the main gas stream through a T-inlet, and the ratio of the diameter of the main-stream tube to that of the inlet tube is 3.5, the lower limit of the ratio of inlet-gas momentum (mass velocity) to main-stream momentum for good mixing is 2; an increase to 3.5 still gives good mixing, with best about 2.7; ratios 3.5—6 give poor mixing because the inlet gas is carried to the farther side of the tube, whilst ratios above 6 give good mixing because of the rebound from the opposite wall. The lower limit of momentum ratio for good mixing increases from 1.0 for diam. ratio 1.16 to 2.5 for diam. ratio 7.0. The inlet tube tilted at 45° upstream affords good mixing with a slightly lower momentum ratio than when the inlet tube is perpendicular, but if the angle is downstream mixing is more difficult. Two perpendicular inlet tubes arranged diametrically opposite to one another have a wider range of momentum ratio for good mixing than has a single tube. Admission at the centre line of the tube by an injector shows no advantage over the T-connection.

D. K. MOORE.

**Moisture in technical gases.** II, III. F. LÜTH (Arch. Eisenhüttenw., 1930—1, 4, 185—189, 281—287; Stahl u. Eisen, 1931, 51, 14—16; cf. B., 1930, 305).—Examples are given of the use of the expressions

\* The remainder of this set of Abstracts will appear in next week's issue.

previously developed for the determination of the moisture content of gases from readings made with the wet- and dry-bulb thermometers. A. R. POWELL.

**Drying of salt.** LAGRANGE.—See VII. **CO<sub>2</sub> meter for kilns.** McCANN.—See VIII. **Dry granulation of slag.** BERGER. **Boiler plate.** POMP. **Copper firebars etc.** KÜHNEL. **Cylinder alloys.** VON SCHWARZ.—See X. **Micromanipulator.** HAUSER.—See XIV.

See also A., Feb., 190, **Optical pyrometry** (EGERTON and MILFORD). **Pyknometer** (HOFFMANN).

## PATENTS.

**Determining the limiting creep stress of materials.** D. COLVILLE & SONS, LTD., W. BARR, and W. E. BARDGETT (B.P. 339,890, 26.2.30).—A specimen is axially connected rigidly to a calibrated steel bar constituting a weigh bar and the assembly is mounted through a rectangular framework. The specimen bar is heated to a determined temperature after applying an initial stress rather greater than the estimated limiting creep stress at that temperature. As the test proceeds the specimen gradually stretches, and the stress to which the weigh bar is subjected becomes proportionately decreased, and eventually no change in length is noted. This final stress is the limiting stress of the material at the particular temperature employed; it is claimed that a value for this may be obtained within one week, and no elaborate extensometer within the furnace is needed, as measurements are made on the weigh bar.

C. A. KING.

**Porous masses for storage of compressed gases.** GAS ACCUMULATOR CO. (UNITED KINGDOM), LTD., Assees. of AUTOGEN GASACCUMULATOR A.-G. (B.P. 339,899, 8.3.30. Ger., 30.5.29).—Vessels for storing acetylene etc. are filled with pieces of pumice, trass, slag, etc., and the interstices are filled up with a mixture of kieselguhr and cement.

L. A. COLES.

**Apparatus [roasting furnace] for heat treatment of metalliferous materials.** R. A. STOKES and E. G. L. ROBERTS, Assrs. to E.M.S. INDUSTRIAL PROCESSES, LTD. (U.S.P. 1,788,351, 6.1.31. Appl., 5.3.30. U.K., 20.12.28).—See B.P. 330,930; B., 1930, 913.

**Grinding mill.** J. R. KUENEMAN (U.S.P. 1,787,788, 6.1.31. Appl., 21.8.28).—See B.P. 317,769; B., 1931, 94.

**Solids breaking and handling apparatus.** F. B. ALLEN (U.S.P. 1,772,452, 12.8.30. Appl., 15.4.29).—See B.P. 325,442; B., 1930, 352.

**Evaporation of liquids.** R. A. STOKES and E. G. L. ROBERTS, Assrs. to E.M.S. INDUSTRIAL PROCESSES, LTD. (U.S.P. 1,788,352, 6.1.31. Appl., 5.3.30. U.K., 20.12.28).—See B.P. 330,931; B., 1930, 845.

**Refrigerating machines.** J. SCHNEIDER (B.P. 341,276, 22.1.30). SULZER FRÈRES SOC. ANON. (B.P. 341,799, 10.4.30. Switz., 7.10.29. Addn. to B.P. 330,818).

**Refrigerating apparatus.** WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (B.P. 341,292, 31.1.30. U.S., 15.2.29). SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 341,552, 16.7.29. U.S., 18.8.28).

**[Absorption] refrigerating apparatus.** G. MAIURI and R. F. BOSSINI (B.P. 341,051, 6.7.29).

**Steel for pressure receptacles** (B.P. 340,819). [Non-corrosive] alcoholic solution (U.S.P. 1,771,985).—See X. [Acid-resisting] rubber compositions (B.P. 339,002).—See XIV.

## II.—FUEL; GAS; TAR; MINERAL OILS.

I. Living bacteria in coal, and their properties.  
II. Occurrence of bacteria in bituminous coal.  
III. Influence of thermophilic bacteria on coal.  
R. LIESKE (Ges. Abh. Kenntn. Kohle, 1930, 9, 27—29, 30—34, 35—39; cf. B., 1928, 774).—I. Bacteria have been found to occur in brown and bituminous coal seams, those in the former being related to the fluorescent group. None of the bacteria is pathogenic.

II. Bacteria occur in bituminous coal seams to a depth of at least 1100 m. and are mainly of the *subtilis* and *mesentericus* groups. The osmotic conditions in the coal are shown to be consistent with the occurrence of such bacteria. Their origin is discussed.

III. Anaerobic, thermophilic bacteria are shown to exist in brown and bituminous coals. Their development results in the production of carbon dioxide, but no combustible gases were formed. A. KEY.

**Testing of fuels.** O. NIEZOLDI (Chem.-Ztg., 1930, 54, 953—954).—The recommendations of the Standardisation Committee for Mines with reference to the determination of heat of combustion and calorific value are criticised (publication D.I.N., E.3716. cf. Arch. f. Wärme-wirts, 1930, No. 7, 257.) The use of briquettes in the determination of calorific value by the bomb method, as recommended, results in errors due to incomplete combustion and is harmful to the inner walls of the bomb. The use of powdered fuel leads to better results. The formula recommended for radiation correction (*i.e.*, that of Regnault and Pfaundler) is not suitable for technical purposes, the simpler formula due to Langbein being the more suitable, viz.,  $c = (m - 1)n + \frac{1}{2}(n - v)$ , where  $m$  = no. of min. from ignition to attainment of maximum temperature,  $n$  = temperature fall per min. on cooling, and  $v$  = initial temperature rise per min. Objection is raised to the excessive time (7 min.) during which temperature rise occurs in the standard method. Efficient heat distribution in the bomb reduces the time to 4 min. and minimises errors. It is considered that correction of the calorific value to allow for formation of sulphuric and nitric acids and partial combustion of the wire used for ignition is often unnecessary for technical purposes on account of the small differences involved. The heat developed by combustion of the wire may be reduced by using nickel-alloy wire (Nickelin: heat of combustion 0.775 kg.-cal. per g.) in place of the iron wire (1.6 kg.-cal. per g.) usually used. The values adopted in correcting for heat of vaporisation of water are criticised. H. E. BLAYDEN.

**Organic and other forms of sulphur in [Canadian] coals containing large amounts of sulphur.** J. H. H. NICOLLS and E. SWARTZMAN (Canada Dept. Mines, Invest. Fuels, 1930, No. 712, 28—35; cf. B., 1925, 485).—Sulphate sulphur was determined by treating 5 g. of coal with 3% hydrochloric acid for 40 hrs. at 60°, and pyritic sulphur by digesting 1 g. with nitric acid (*d* 1.12) for 96 hrs. at room temperature and subtracting from

the amount thus obtained the amount of sulphate sulphur. Organic sulphur was not determined, it being assumed that it corresponded to the difference between the total sulphur, as determined by the Eschka (or sodium peroxide) method, and the sum of the sulphate and pyritic sulphur. Resinic sulphur was determined by digesting 0.5 g. of coal with phenol for 20 hrs. at 140°, filtering off the residue, and determining the sulphur in this by fusion with sodium peroxide; the difference between this sulphur and total organic sulphur is resinic sulphur. Humus sulphur was determined by digesting 1 g. of coal with concentrated nitric acid for 30 min. at room temperature, treating the washed residue with strong ammonia water for several hours at room temperature, filtering off the residue, evaporating the filtrate to dryness, and then determining its sulphur content by fusion with sodium peroxide. Treatment of coal with 3% hydrochloric acid as described above or with water under similar conditions showed that (i) oxidation of pyritic sulphur occurred in regular increments with successive treatments of coal with hydrochloric acid and water, especially with the latter medium; (ii) inorganic sulphide sulphur was evolved from certain coals following the addition of dilute hydrochloric acid, and dilute nitric acid dissolved some of the organic sulphur, hence it follows that sulphur, described previously as "pyritic," is better described as "sulphide" sulphur. Treatment of coal with nitric acid of  $d$  1.12 showed that organic sulphur was often lost during the digestion, hence the most suitable method for its estimation is by subtracting the amount of inorganic sulphur from the total sulphur content; the method originally proposed for the determination of humus sulphur is unreliable owing to the liability of loss of organic sulphur.

C. B. MARSON.

**Low-temperature distillation of coal; its technical and economic significance.** F. FISCHER (Ges. Abh. Kenntn. Kohle, 1930, 9, 420—443).—The author reviews the low-temperature process to the end of 1928, stating differences between coke, gas, tar, and liquor formed from different German coals carbonised at different temperatures. The uses of the different carbonisation products are outlined and a survey is made of many low-temperature plants.

C. B. BOLLAND.

**Production of suitable oven cokes by mixing coals of different type.** E. KOCH (Ges. Abh. Kenntn. Kohle, 1930, 9, 367—375).—A laboratory method is described for producing cokes with properties of coke-oven cokes. Varying mixtures of different types of coals were coked in order to find the maximum percentage of lean coals which could be used. The abrasion coefficient of the cokes was the percentage remaining on a standard sieve after a standard treatment in a ball mill.

C. B. BOLLAND.

**Humic acid as the raw material of ulmin coals.** W. FUCHS (Ges. Abh. Kenntn. Kohle, 1930, 9, 171—176).—The method by which the ulmin content of coals was determined depended on the following facts, viz., that ulmins decompose acetates, this being a measure of the carboxyl groups present, and that ulmin salts can change their contained metal when in contact with other metal salts. The carboxyl content decreased much

more rapidly than the hydroxyl groups when passing from the brown coals to ordinary coals. All coals were found to reduce silver salts to metal under suitable conditions and to take up bromine. There was found to be a connexion between the amount of silver salt decomposed and that of bromine taken up by coals. These properties were found not to be proportional to the age of the coal. (Cf. B., 1928, 555.) C. B. BOLLAND.

**Oxidation of Cassel humic acid.** W. STENGEL (Ges. Abh. Kenntn. Kohle, 1930, 9, 125—129).—By the action of a solution of sodium hypochlorite saturated with chlorine, 70% of Cassel humic acid can be converted into a bright yellow humic acid containing chlorine, most of which is soluble in water. The remainder is highly polymerised and only further oxidised with difficulty. The oxidation was carried out at 0° because of the rapidity of the action. Some of the oxidised humic acid soluble in water was also soluble in ether and some was volatile in steam. The ether-soluble portion is mainly a hard resinous substance, from which a small amount of oxalic acid crystallises. Benzene-pentacarboxylic acid could not be detected in the water-soluble portion. Methylation of the portion soluble in ether produced viscous esters. (Cf. B., 1929, 771; A., 1930, 601.)

C. B. BOLLAND.

**Electrolysis of montanic acid.** F. FISCHER and O. HORN (Ges. Abh. Kenntn. Kohle, 1930, 9, 305—308).—As salts of the lower fatty acids on electrolysis yield hydrocarbons, the production of higher paraffins from montanic acid by electrolysis was attempted. From potassium palmitate, triacontane was obtained. Similar attempts to decompose crude montanic acid failed owing either to the insolubility of the salts used or to some constitutional peculiarity.

H. J. HODSMAN.

**Effect of illium crucibles on volatile determinations in coal.** J. A. ADDLESTONE (Ind. Eng. Chem. [Anal.], 1930, 2, 422).—Determination of volatile matter in coal in illium crucibles yields results which are consistently lower by about 0.25—0.5% than when platinum crucibles are used. Although when placed in the furnace the illium crucible produces a more prolonged lowering of temperature, the time of heating is without influence on the discrepancy.

H. F. GILLBE.

**M.p. of [fuel] ash.** C. STAEMMLER (Chem.-Ztg., 1931, 55, 59—60).—The m.p. of synthetic mixtures of lime with ferric oxide, silica, and mixtures of these in various molecular proportions has been determined. In the lime-ferric oxide series the m.p. of mixtures with 0.5—2 mols. CaO to 1 mol. Fe<sub>2</sub>O<sub>3</sub> is higher by 60—70° when the lime is added as calcium sulphate than when added as carbonate. Mixtures of lime and silica in the ratio 0.5—1 : 1 melt at 30—40° higher when the sulphate is used, whereas mixtures in the ratio 1.5—2 : 1 melt at 80—90° lower with sulphate than with carbonate. In the ternary system silica-ferric oxide-lime, higher m.p. are observed with sulphate mixtures only when the lime content exceeds 25% of the fritted mass. Ignition at 1200° of lignite ash with a high sulphate content results in only a slow loss of sulphur; thus an ash with 34% SO<sub>3</sub> lost 12—9% after 90 min. at 1200°.

A. R. POWELL.

**Regeneration of activated carbons.** A. S. SIPYAGIN and E. S. SERKIN (Zhur. Sakh. Prom., 1930, 4, 176—181).—The material is boiled for 2 hrs. in 2% sodium hydroxide solution and then in 2% hydrochloric acid: it is washed to  $pH$  7.0—7.2, dried, and heated at 600—650° for 10 min. CHEMICAL ABSTRACTS.

**Natural asphalt from Strečno in Slovakia.** M. LANDOVA (Chem. Listy, 1930, 24, 461—462).—Asphalt extracted from Strečno dolomite has m.p. 37°, 1.37% of asphaltic acids, 2.20% of lactones and anhydrides, 41.29% of asphaltic resins, and 51.25% of oily constituents; on analysis it yields: C 81.77, H 10.85, S 1.29, N 0.94, O 4.07, and ash 1.08%.

R. TRUSZKOWSKI.

**Partial combustion of methane under various pressures with especial reference to the formation of acetylene thereby.** F. FISCHER and H. PICHLER (Brennstoff-Chem., 1930, 11, 501—507).—By passing mixtures of methane and air or oxygen through a narrow tube at 1000—1400°, the time of contact being about 0.005 sec., the methane was converted more or less completely into acetylene, hydrogen, and carbon monoxide. The yield of acetylene increased with an increase in the proportion of oxygen in the mixture up to a maximum, and thereafter decreased; the yield also increased with rise of temperature. The maximum yield of acetylene obtained amounted to 50—60% of the methane supplied. Lowering the pressure had little influence on the yield of acetylene. Under increased pressure in a closed vessel (initial pressure 120 atm.) the methane was more or less completely converted into carbon and oxides of carbon, and little or no acetylene was formed. Similar results were obtained by starting with mixtures of coke-oven gas (23%  $CH_4$ ) and oxygen. At any particular temperature the yield of acetylene passed through a maximum as the time of contact was varied. At 1400° and with about 0.006 sec. contact 30% of the methane was converted into acetylene; at 1600° and 0.001 sec. the conversion was 50—60%. By passing a mixture of coke-oven gas and oxygen through a tube packed with pieces of pot at 1200°, the gas could be completely converted into carbon monoxide and hydrogen. The acetylene may be separated from the gaseous mixtures obtained, or the latter may be utilised directly for the synthesis of liquid hydrocarbons.

A. B. MANNING.

**Determination of carbon monoxide.** E. DITTRICH (Z. angew. Chem., 1930, 43, 979—980).—The method of determination of carbon monoxide by absorption in iodine pentoxide and oleum offers no advantages over the cuprous chloride method in point of speed or accuracy, and has certain disadvantages in the analysis of a mixture of gases. Although hydrogen and methane are left unattacked by iodine pentoxide, ethane is absorbed slightly and propane and butane almost completely under the conditions of analysis. The absorption of the higher paraffins by ammoniacal cuprous chloride is only slight.

E. S. HEDGES.

**Behaviour of benzene in hydrogen at high temperatures.** F. FISCHER and A. HINTERMAIER (Ges. Abh. Kenntn. Kohle, 1930, 9, 678—679).—After heating a stream of methane in a tube at 1090°, some

benzene was detected in the products. Under similar conditions benzene itself rapidly decomposed; this limits the yields obtainable by heating methane.

A. KEY.

**Influence of sulphur in the water-gas and in the catalyst on the action of the contact material in the synthesis of petroleum.** H. BAHR (Ges. Abh. Kenntn. Kohle, 1930, 9, 514—516).—In the synthesis of paraffins from water-gas at 270° over an iron-copper catalyst, the same result was obtained whether a "synthetic" water-gas made from pure hydrogen and carbon monoxide or a normally purified water-gas was used. The sulphur content of the latter had no appreciable effect. Of two copper-cobalt catalysts differing in sulphur content, the one with the lower sulphur content was the more readily deactivated.

H. J. HODSMAN.

**Influence of organic sulphur compounds in the water-gas on the synthesis of petroleum.** F. FISCHER and P. DILTHEY (Ges. Abh. Kenntn. Kohle, 1930, 9, 512—513).—In the synthesis of paraffins from water-gas at 270°, using a contact mass prepared from lead carbonate and iron oxide, the catalyst takes up sulphur from unpurified gas. The contact material can be regenerated by oxidation with air. The water-gas used for the synthesis must be completely freed from sulphur compounds.

H. J. HODSMAN.

**Catalytic conversion of the organic sulphur compounds of water-gas into hydrogen sulphide.** F. FISCHER and P. DILTHEY (Ges. Abh. Kenntn. Kohle, 1930, 9, 494—500; cf. B., 1927, 385).—A contact mass obtained by the reduction of a mixture of lead chromate and copper oxide with water-gas proved an active catalyst for the conversion at 500° of the organic sulphur in the gas into hydrogen sulphide. It is considered that the mechanism consists in the intermediate formation of metal sulphides, which are reduced by the hydrogen present to hydrogen sulphide. The carbon of the carbon disulphide is probably converted into methane, deposition of carbon never being observed, and therefore little falling off of activity occurs. Other metals can be used instead of copper. A contact mass of silver on an asbestos-fibre carrier showed even greater activity.

H. J. HODSMAN.

**Disintegration of the catalyst in the synthesis of benzene.** O. ROELEN and A. HINTERMAIER (Ges. Abh. Kenntn. Kohle, 1930, 9, 517—518).—An alkaline copper-iron catalyst exposed to water-gas at 250° showed disintegration with increase in bulk volume. After 500 hrs. its bulk density had fallen to about one fourth the original value. Thereafter it remained constant.

H. J. HODSMAN.

**Composition of the products obtained in the synthesis of petroleum.** H. TROPSCH (Ges. Abh. Kenntn. Kohle, 1930, 9, 561—570).—The benzene fraction (b.p. 60—185°) of the synthetic petroleum obtained by the catalytic reduction of carbon monoxide at ordinary pressures (cf. B., 1926, 475) has been examined. After fractionating at every 10°, the sp. gr., refractive index, and iodine value of each fraction were determined. The olefines were determined by separation as the double compound with mercury acetate, being

subsequently regenerated by dilute hydrochloric acid. The benzene was found to consist almost entirely of a mixture of paraffins (*n*-pentane to *n*-nonane, also *iso*-nonane) with the corresponding olefines, the latter predominating, especially in the lower-boiling fractions. It was quite stable to air and light for at least two years.

A. KEY.

**Re-utilisation of the exit gas from petroleum catalysts and its conversion into methane.** H. BAHR (Ges. Abh. Kenntn. Kohle, 1930, 9, 519—521).—The exit gas from catalysts used for petroleum syntheses was passed over iron at 850°, whereby the carbon dioxide was reduced by hydrogen and hydrocarbons to carbon monoxide. This gas could be re-used for petroleum synthesis. The gas can also be converted into methane by passing over nickel at 240°. The calorific value of the gas after extraction of carbon dioxide was 6400 kg.-cal. per m.<sup>3</sup>

C. B. BOLLAND.

**Development of the synthesis of benzene from carbon monoxide and hydrogen at the ordinary pressure.** F. FISCHER (Brennstoff-Chem., 1930, 11, 489—500. Cf. B., 1926, 475; 1927, 748; 1928, 324; 1929, 1003).—Laboratory experiments on this synthesis have been carried out in a gas-heated thermostatically-controlled aluminium block furnace, which can accommodate four glass tubes and so permits the exact comparison of the activity of four catalysts simultaneously. The gas used was a mixture of water-gas and "spaltgas," in which CO : H<sub>2</sub> = 1 : 2; the "spaltgas" was obtained by heating a mixture of coke-oven gas and steam at about 1000°. The gas was freed from sulphur by passing it over a gas-purifying mass, converting the organic sulphur into hydrogen sulphide catalytically, and removing this by washing with a solution of potassium ferricyanide, the latter subsequently being regenerated electrolytically. The purified gas contained not more than 0.2 g. of sulphur per 100 cub. m. The catalysts used were cobalt-copper (9 : 1) or manganese-iron-copper (4 : 1); the addition of a small amount of alkali improved the latter, but adversely affected the activity of the former. The catalysts were prepared in the form of the mixed oxides, and no preliminary reduction of these was necessary. The yields of liquid products per cub. m. of gas in one passage (4 litres/hr.) over 5 g. of catalyst were 100 c.c. with the cobalt catalyst and 50 c.c. with the iron catalyst, i.e., about 50% and 25%, respectively, of the theoretical. About 80% of the liquid products consisted of benzene boiling below 200°. The activity of the catalyst decreased in time due to the absorption of high-boiling products, but was readily restored by passing air over the material at 200° for 1—2 hrs. After some months the activity fell, due to sulphur poisoning; it then became necessary to regenerate the catalyst chemically. It is suggested that the reaction proceeds through the intermediate formation of a carbide. The products consist almost exclusively of saturated and unsaturated aliphatic hydrocarbons, presumably of the straight-chain type. To obtain high yields of the more valuable products the reaction temperature must be maintained within narrow limits. The reaction is exothermic, and the greatest difficulty in designing a large-scale unit arose in providing for the necessary temperature control. Various designs were

tried; in the most successful the contact mass was packed between perforated metal plates attached to the walls of the vessel wherein were arranged the vertical tubes of a boiler; the temperature was controlled by the rate of evaporation of the water, i.e., by the pressure maintained in the boiler. With this apparatus, designed to treat 10 cub. m. of gas per hr., 80% of the laboratory yield of liquid products has been obtained. In considering the economics of the process account must be taken of the exothermic nature of the reaction, the heat thereby lost amounting to about 25% of the heating value of the gas used. Some progress has been made in producing a cheaper catalyst by replacing the cobalt by nickel.

A. B. MANNING.

**Effect of different carriers on the synthesis of benzene.** F. FISCHER and P. DILTHEY (Ges. Abh. Kenntn. Kohle, 1930, 9, 501—511).—A satisfactory catalyst was prepared by depositing iron and copper (4 : 1) in the form of nitrates upon a suitable support. Iron alone was unsatisfactory, as also were iron and silver catalysts. The reaction was carried out in iron tubes, but unless these were treated with 5*N*-nitric acid the catalyst lost its activity. The contact materials lose activity by the accumulation of high-boiling substances, but neither extraction with solvents nor hydrogenation sufficed to restore activity, which could only be achieved by combustion in a current of air. Platinum and ruthenium on an asbestos support proved an unsatisfactory catalyst. The formation of unsaturated hydrocarbons was considered doubtful.

H. J. HODSMAN.

**Occurrence of hydroaromatic constituents in synthetic benzene.** H. BAHR (Ges. Abh. Kenntn. Kohle, 9, 521—523).—The action of nickel-alumina catalysts on aliphatic and hydroaromatic compounds in presence of hydrogen depends on the composition of the catalyst. One containing 75% Ni was found to be without action on *n*-hexane, but decomposed the corresponding *cyclo*-compound, thus providing a means for separating the two. The treatment of synthetic benzene with such a catalyst gave a small condensate of a characteristic aromatic odour, indicative of the presence of *cyclo*-aromatic compounds.

A. KEY.

**Mechanism of the cracking process.** N. MAYER (Petroleum, 1930, 26, 1248—1250).—It is suggested that the primary reaction in the cracking of oils is a reversible thermal dissociation, similar in character to that undergone by hexaphenylethane at lower temperatures, C<sub>2</sub>Ph<sub>6</sub> ⇌ 2CPh<sub>3</sub>, and that this dissociation is followed by the relatively slow conversion, by addition, condensation, or polymerisation, of the unsaturated radicals so formed into more stable compounds. This hypothesis seems to conform better with the observed phenomena than that of Gurwitsch, who assumed a practically instantaneous transformation of the primary dissociation products.

A. B. MANNING.

**Hydrogenation adapted to the [mineral] oil refinery.** M. W. BOYER (Chem. and Met. Eng., 1930, 37, 741—745).—Hydrogenation of petroleum is carried out at 3000 lb./in.<sup>2</sup> pressure, and as the catalysts are not poisoned by carbon monoxide or sulphur compounds hydrogen of a high degree of purity is not required.

The process may be used for the conversion of high-sulphur, heavy, asphaltic crude oils and refinery residues into motor spirit and distillates low in sulphur and free from asphalt without coke formation; conversion of low-grade into high-grade lubricating oils; improvement of the quality of inferior burning oils; desulphurisation and gum stabilisation of high-sulphur, badly gumming, cracked naphthas; and for the conversion of paraffin oils into good anti-knock motor spirit. D. K. MOORE.

**Physico-chemical properties of [Japanese] acid clay.** V. Decolorisation of petroleum by adsorption (1—3). K. KOBAYASHI and K. YAMAMOTO (J. Soc. Chem. Ind., Japan, 1930, 33, 428—430 B).—Freundlich's adsorption formula has been found to apply to the treatment of petroleum distillates by acid clay. The decolorisation is therefore presumed to be mainly due to adsorption. This is also the case with decolorisation with fuller's earth, norit, silica gel, etc. Decolorising powers are compared by the use of this formula, taking the final or equilibrium concentration of the solution. Solutions obtained by extracting used acid clay with benzol or kerosene similarly follow the formula.

C. IRWIN.

**Production of motor fuels containing alcohol.** K. R. DIETRICH (Z. Spiritusind., 1930, 53, 349).—For the production of such fuels, benzine or benzol is usually mixed with alcohol of at least 99·6% concentration. A mixture of 20% of alcohol with benzine separates into layers if the concentration of the alcohol is less than 96%, whereas with a mixture containing 30% of alcohol 95% spirit can be used. With benzol lower strengths of alcohol are possible, and 87% spirit may be used in a mixture of 20% of alcohol without subsequent separation. It is officially decreed that the fuels should contain at least 20% of alcohol, and the mixtures which are most prevalent contain 20% of absolute alcohol with 80% of benzine, 30% of absolute alcohol with 70% of benzol, and 20% of absolute alcohol with 30% of benzol and 50% of benzine. In all cases the percentages are by weight. When the benzine is distilled, 25% must volatilise below 100° and 95% below 200°. whilst its content of paraffin hydrocarbons should not exceed 60%. A table is given of "water values" which indicate the amount (c.c.) of water which induces a haze when it is added to 100 c.c. of the motor fuel.

C. RANKEN.

**Motor spirits containing alcohol.** X, XI. W. SWIENTOSLAWSKI (Przemysl Chem., 1930, 14, 513—517, 518—529; cf. B., 1931, 101).—X. [With T. KALIŃSKI and B. KARPINSKI.] The phenomenon of premature spontaneous inflammation is observed only in those motor mixtures containing over 40% of benzine. The spontaneous inflammation point is not lowered by the addition of gasoline up to 20%, and the zone of premature spontaneous inflammation decreases as the alcohol content rises.

XI. [With S. BAKOWSKI and B. KARPINSKI.] The velocity of vaporisation of drops of various motor fuels containing alcohol, in contact with metal surfaces heated at 70—550°, has a minimum value at about 100° for aluminium and 200° for platinum surfaces;

above these temperatures it at first increases and then decreases slowly with rise in temperature. The curves obtained for mixtures differ little from those of the pure constituents. R. TRUSZKOWSKI.

**Fractional analysis of light [hydrocarbon] mixtures.** W. M. SCHNAUFELBERGER (Oil Gas J., 1930, 29, No. 16, 46, 163—164, 167—168).—An apparatus is described. CHEMICAL ABSTRACTS.

**Anhydrous aluminium chloride.** SIMON.—See VII. **Tanning materials.** ANON.—See XV. **Petroleum insecticides.** WOODWORTH.—See XVI.

See also A., Feb., 160, **Extraction of phenols from alkaline solution** (VAVON and PAIRA). 192, **American peat profiles** (FEUSTEL and BYERS). 194, **Pyrolysis of propylene** (HURD and MEINERT).

## PATENTS.

**Rendering fuel agglomerates formed with the aid of algine, which swell on contact with water, unaffected by the action of water.** P. GLOESS (B.P. 339,786, 14.2.30. Fr., 14.2.29).—The agglomerates are impregnated with coal tar, wood tar, or other organic coking material, which is then raised to the coking temperature. H. ROYAL-DAWSON.

**Coking of coal.** S. W. PARR and T. E. LAYNG, ASSRS. to URBANA COKE CORP. (U.S.P. 1,754,765, 15.4.30. Appl., 31.10.18. Renewed 21.9.23).—The coal is heated uniformly throughout its mass to a temperature slightly below that at which it becomes plastic, and is then charged into a chamber wherein it is heated while quiescent to bring the whole mass to a fused condition and finally to coke it. The coking temperature is preferably 750° or just below. The preheating may be effected by passing a hot inert gas, e.g., superheated steam, through the material. The temperature of the preheated material is raised to such a point that the additional heat required to fuse the mass is less than the exothermic heat liberated during the process. A. B. MANNING.

**Coking retort ovens.** KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 337,801, 9.7.29. U.S., 25.4.29).—Means are provided for supplying air to the nozzles at one level of the flame flues of a heating wall while gas is being supplied to those at another level in the flues, the air serving to decarbonise the nozzles and to form secondary air for combustion. A. B. MANNING.

**Vertical coking-retort oven.** J. VAN ACKEREN, ASSR. to KOPPERS Co. (U.S.P. 1,769,810, 1.7.30. Appl., 18.11.25).—The retorts are arranged in a battery, being separated from one another by heating walls. The latter have flues through which the combustion gases pass, the path being down one set of walls and up another, before going to regenerators which are arranged alongside the battery of retorts. The path of the gases is periodically reversed. The retorts are wider at the bottom than at the top, to facilitate the passage of material through them, and are worked continuously. T. A. SMITH.

**Gas producer.** H. F. SMITH, ASSR. to GAS RESEARCH Co. (U.S.P. 1,769,192, 1.7.30. Appl., 24.7.20).—The gas producer has a rotatable top moving in a water-seal and fitted with a fuel-feeding device and poker. These

are water-cooled and steam-operated. The speed of operation of the steam cylinder is governed by the escape of oil through a valve. A cylinder for this oil forms part of the steam-valve mechanism. Hot water from the water-seal is used to saturate the air which is admitted to the producer.

T. A. SMITH.

**Gas producer apparatus.** J. A. MACDONALD (B.P. 336,932, 29.7.29).—A producer-gas plant with a combustion chamber of substantial volume has a horizontal air-inlet pipe extending within the chamber and mounted as near the bottom as is practicable. The outlet is mounted horizontally on the opposite wall of the chamber at a slightly higher level; its depth of penetration into the chamber is adjustable. Means are provided for spraying water on to the incandescent zone through the air-inlet pipe, and sufficient water is supplied to maintain the base of the chamber flooded. The producer is designed to operate as a suction-gas plant in conjunction with an internal-combustion engine.

A. B. MANNING.

**Gas producer.** J. BECKER, Assr. to KOPPERS CO. (U.S.P. 1,757,232, 6.5.30. Appl., 24.10.23).—The cylindrical producer chamber is water-jacketed, whilst above the chamber and within the producer shell a high-pressure boiler is mounted. A low-pressure steam drum mounted outside the producer communicates with the water-jacket in such a way as to permit thermosiphonic circulation of water within the system; feed-water is supplied thereto at a point preferably just below the low-pressure steam drum. The pressure within the water-jacket is maintained sufficiently high to prevent steam forming therein. Part of the heated water is withdrawn from the upper part of the water-jacket and utilised as feed-water for the high-pressure boiler, being forced thereto by means of a high-pressure pump.

A. B. MANNING.

**Apparatus for the manufacture of gas.** HUMPHREY & GLASGOW, LTD., Assces. of H. G. TERZIAN (B.P. 336,902, 27.1.30. U.S., 20.4.29).—Apparatus for the complete gasification of bituminous fuel comprises a generator, a carburettor, a superheater, and a wash box. The generator contains an upper carbonising zone and a lower gasifying zone. An annular chamber surrounding the former communicates therewith through ports at different levels in the wall, the lower row of ports being approximately at the top of the water-gas zone. The gas offtake takes the form of a perforated hollow core suspended centrally in the carbonising zone. When the lower zone is air-blasted the blast gases pass through the lower row of ports into the annulus and thence to the carburettor and superheater, wherein they are burned. An up-run is then made with steam, and the resultant water-gas passes through the lower ports into the annulus and thence back through the other ports, through the fuel in the carbonising zone, into the gas offtake. By this arrangement a more uniform carbonisation of the fuel is ensured. The up-run may be followed by a down-run with steam preheated by passage through the superheater and carburettor.

A. B. MANNING.

**Gas producers.** W. B. CHAPMAN (B.P. 337,388, 29.7.29).—The producer is provided with an underfeed

mechanism, the fuel being fed from a hopper by a worm to a hollow central column, and thence by a vertical feed screw into the lower part of the fuel bed. An agitator member rotates round the fuel-feeding mechanism, sweeping through the lower portion of the fire bed below the point of entry of the fuel, and is adapted to impart an outward movement thereto. An annular ash-discharge opening is provided between the fire-bed support and the side walls. The producer is especially suitable for direct boiler-heating purposes, the combustion chamber of the boiler being disposed just above the fire-bed. Means are then provided for projecting secondary air tangentially into the chamber through openings in the side walls. The secondary air is preheated by the waste gases, the preheating device forming an annular chamber surrounding the combustion chamber.

A. B. MANNING.

**Manufacture of producer gas.** R. L. WILLIS (U.S.P. 1,758,991, 20.5.30. Appl., 17.9.24).—The fuel bed of a producer is blasted with mixed air and steam, the quantity of air admitted being automatically controlled by the temperature of the outgoing producer gas, and the quantity of steam admitted similarly controlled by the temperature of the ingoing mixed air and steam. For this purpose thermocouples are placed in the corresponding conduits and are arranged to control relays.

A. B. MANNING.

**Production of gas and aromatic hydrocarbons from heavy hydrocarbon oils.** A. S. RAMAGE, Assr. to OZONIDE CORP. (U.S.P. 1,752,692, 1.4.30. Appl., 23.12.25).—Hydrocarbon vapours are mixed with 1–2% of steam and passed in contact with ferric oxide at 470–540°. The resulting vapours and gases are mixed with 10–20% of steam and brought into contact with finely-divided metallic iron at about 620°. The resulting vapours and gases are further mixed with 20–40% of steam and brought into contact with finely-divided iron at 700° or over. The final hot gases are immediately cooled by passing them through a spray of incoming oil and are then delivered to a reflux tower from which the gaseous portion is passed to condensers in which the permanent gases are separated from the hydrocarbons suitable for motor fuel. The liquid product from the reflux tower is mixed with the fresh supply of oil and re-treated.

H. S. GARLICK.

**[Water-]gas manufacture.** F. W. STEERE, Assr. to SEMET-SOLVAY ENG. CORP. (U.S.P. 1,752,037, 25.3.30. Appl., 27.7.26).—The effluent waste liquor produced in the manufacture of water-gas in a plant comprising a generator and a heat regenerator is passed into a decanter, wherein the sediment is allowed to settle, and thence is pumped to the heat regenerator, wherein it is volatilised, the vapours being then passed through the fuel bed of the generator for the production of water-gas. The whole of the effluent liquor can thus be utilised in the plant.

A. B. MANNING.

**Manufacture of illuminating gas. Gas-making process.** D. J. YOUNG, Assr. to YOUNG-WHITEWELL GAS PROCESS CO. (U.S.P. 1,751,784 and 1,751,849, 25.3.30. Appl., [A] 20.8.23, [B] 22.6.22).—Carburetted water-gas plant comprising a generator, a carburettor, and a superheater connected in series is described. In



(A) a permanently free passage is left between the generator and carburettor, and means are provided for producing a reversal of steam flow through the entire plant and for introducing fuel to the generator during the reverse flow of the steam. In (B) the plant is operated in accordance with the following steps: the fuel bed is air-blasted and the blast gases are burned in the carburettor and superheater; steam is passed up through the fuel bed and the water-gas so formed is passed successively through the carburettor and superheater; steam is passed down through the fuel bed and the resultant water-gas is withdrawn directly from the generator and led to storage. The water-gas produced during the steam up-run may be carburetted. The steam for the down-run may be admitted either at the top of the generator or at a suitable point in the carburettor or superheater, whereby it is given any desired degree of preheating, and serves to interact with and remove any carbon deposited in the apparatus.

A. B. MANNING.

**Water-gas generators.** HUMPHREYS & GLASGOW, LTD., Assees. of J. S. HAUG (B.P. 339,743, 4.11.29. U.S.; 7.12.28).—A water-gas generator with annular water jacket has a rotary grate containing a central air and steam inlet which extends well into the fuel bed, and is arranged so that the hottest part of the generator is distant from the walls. The total area of the openings in the projecting part of the inlet is considerably larger than that of the horizontal portion. To the grate are fitted cutting blades which remove clinker from the bottom of the fire.

R. H. GRIFFITH.

**Heat treatment of gaseous mixtures.** SOC. D'ETUDES ET RÉALISATION DITE "ÉREAL" (B.P. 314,807, 1.7.29. Belg., 2.7.28).—Gaseous mixtures composed mainly of hydrocarbons, *e.g.*, coke-oven gases more or less deprived of their hydrogen content, are forced through an incandescent mass, whereby the hydrocarbons are completely cracked. The fuel mass is periodically blasted with air, and thereby maintained at 800–1200° substantially by the combustion of the separated carbon only. The apparatus comprises a fuel shaft and recuperators for preheating the air and the initial gas mixture. The cracked gas and the original gas may be mixed in suitable proportions to produce a gas adapted for heating and illuminating purposes.

A. B. MANNING.

**Apparatus for treating gases.** F. W. SPERR, JUN., ASSR. to KOPPERS Co. (U.S.P. 1,768,162, 24.6.30. Cf. U.S.P. 1,578,687; B., 1926, 430).—An apparatus is described for the removal of substances similar in nature to naphthalene from coal gas, coke-oven gas, etc. An absorbing tower is divided into two or more compartments vertically above each other, and filled with steel turnings spaced and supported upon wooden staves. The lower compartments are provided with a circulatory system by means of which the solvent (*e.g.*, anthracene oil or petroleum oil) is continuously circulated. The upper portion of the tower is provided with fixed or revolving sprays from which is intermittently delivered a quantity of fresh solvent by means of a suitable siphon fitted with a by-pass. The fresh solvent flows down into the lower portion of the tower, and mixes

with the circulating liquid. Means are provided for heating the liquid.

H. E. BLAYDEN.

**Measurement of the calorific value of combustible gases.** H. N. PACKARD, ASSR. to CUTLER-HAMMER, INC. (U.S.P. 1,767,771, 24.6.30. Appl., 21.1.21).—The apparatus described in the prior patent (U.S.P. 1,625,277) is modified in such a manner that the changes in heating value of the gas under examination are measured by the changes in the proportions of gas and air (under the same conditions of pressure, temperature, and humidity) necessary to maintain a substantially constant temperature. Changes of temperature due to combustion of the gas influence one of two electrical resistance thermometers, which are included in the two arms of a Wheatstone bridge. The resultant flow of current actuates a continuously variable gear device which alters the relative speeds of rotation of pumps which supply the gas and air in such a manner as to maintain the temperature of combustion constant. Alteration of the variable gear is indicated by a pointer, which serves for observation and recording of the changes in the volumetric ratio of the two gases supplied for combustion, and hence of the calorific value of the gas.

H. E. BLAYDEN.

**Purifying gas from hydrogen sulphide with production of zinc sulphide.** A. F. KUNBERGER, ASSR. to UNITED GAS IMPROVEMENT Co. (U.S.P. 1,759,408, 20.5.30. Appl., 8.3.28).—The gas is washed with a solution of zinc ammonium chloride in ammonium chloride. After separation of the precipitated zinc sulphide the spent ammonium chloride solution is revived by subjecting it to the galvanic action of zinc and a substance electronegative to zinc.

A. B. MANNING.

**Removal of benzol and its homologues from fuel gases.** J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,760,335, 27.5.30. Appl., 7.7.26).—The gases are oil-scrubbed in three successive stages. Debenzolised oil is introduced into the intermediate stage to remove the major portion of the benzol from the gas, and is thence passed to the first stage to reduce the initial benzol content of the gas. The major portion of the absorbed benzol is then recovered from the enriched oil and the debenzolised oil is returned to the intermediate stage. In the third stage the gas is scrubbed with fresh uncontaminated oil, which after removing the remaining traces of benzol, naphthalene, etc. is added to the debenzolised oil entering the intermediate stage.

A. B. MANNING.

**Distillation of tar or oil.** T. O. WILTON, and CHEM. ENG. & WILTON'S PATENT FURNACE Co., LTD. (B.P. 337,581, 13.6.29. Addn. to B.P. 307,577; B., 1929, 385).—In a plant of the type described in the prior patent, the incoming crude tar or oil and the hot soft pitch from the vapour box are led side by side into a constant-level tank in such a manner, *e.g.*, in sprayed or subdivided form, that they are at once mixed as they issue from their respective inlet pipes so as to ensure intimate contact. The hot pitch dehydrates the tar and causes predistillation of the most volatile constituents.

A. B. MANNING.

**Apparatus for distilling oil.** A. SCHWARZ (U.S.P. 1,765,886, 24.6.30. Appl., 16.6.28).—A heating element for stills consists of two concentric tubes, the outer one

being closed, and a stream of combustion gases or superheated steam passes down the central tube and out through the annular space. The element is suspended in the still and may be readily removed for cleaning purposes. A suitable still is constructed of a third tube enclosing the heating element so that only a thin layer, preferably about 3 in., of oil is present. A gauge is attached to the still and the level of oil is maintained constant, the vaporised oil being replaced by raw stock. The still may be used for cracking purposes.

T. A. SMITH.

**Apparatus for distilling oil.** S. SCHWARTZ, Assr. to DOHERTY RES. Co. (U.S.P. 1,768,965, 1.7.30. Appl., 18.2.22).—Heat economy is obtained in oil distillation by using fresh oil as a means of cooling hot residues and for the partial condensation of vapours in fractional condensers. After being preheated in the fractional condensers the oil is passed to a vapour separator in which it flows in thin layers over baffles, so that vapours may be readily disengaged, and the residue is heated with live steam to assist vaporisation. The unvolatilised oil is passed successively through a series of heating coils and vaporisers, and finally down a tower fitted with baffles, through the centre of which tower the flue of the heating furnace passes. The condensed vapours from the fractional condensers are run to a series of stills arranged in steps so that oil will flow through the series. These stills are fitted with dephlegmator columns packed with earthenware rings and with longitudinal baffles to lengthen the path of the oil through the still. The stills are heated by steam pipes, both closed and open. The cuts obtained from the apparatus do not require further distillation.

T. A. SMITH.

**Destructive distillation of petroleum oil.** A. D. DAVID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,766,983, 24.6.30. Appl., 14.2.23).—The horizontal expansion chamber of a cracking unit contains a pool of molten lead on the surface of which carbon is deposited. The molten lead with the deposited carbon is pumped to a chamber maintained under the same pressure as the cracking unit and into which air is forced so that the carbon is burned. The lead is then returned to the expansion chamber. Heat from the combustion of the carbon is recovered by passing the cracking stock through a coil situated in the upper part of the combustion chamber.

T. A. SMITH.

**Cracking of petroleum oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,766,987, 24.6.30. Appl., 18.10.23. Renewed 2.7.28).—The heavy residues from the digestion chamber of a cracking unit are passed through a precipitating chamber which contains a rotating electrode. Carbon particles collect at the bottom of the precipitation chamber and are removed, the cleaned oil being returned to the cracking cycle.

T. A. SMITH.

**Conversion of [petroleum] oil.** G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,765,663, 24.6.30. Appl., 1.9.20. Renewed 18.3.29).—The operation is carried out in a pair of superposed, horizontal, rotatable shells which are heated by rows of burners, the flames of which are arranged to impinge on the shells above the level of the contained

oil. The shells are closed at one end by a non-rotating portion, ground joints preventing the escape of oil or vapour. Oil is fed into the lower shell by means of a centrally disposed pipe and is vaporised by contact with the hot shell. Foaming is thus avoided with emulsified oils. The vapours evolved are passed by a vapour line through the fixed portion of the upper shell and are cracked by contact with the hot surface. A pressure of 60 lb. may be maintained in the apparatus.

T. A. SMITH.

**Cracking of petroleum oils.** R. A. HALLORAN and A. L. STROUT, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,765,976, 24.6.30. Appl., 4.9.23. Renewed 5.12.27).—The deposition of carbon in cracking plant is minimised by mixing the cracking stock with a lighter "solvent" oil which is not so readily cracked. After passing through the cracking tube the oil is delivered into a special vaporising chamber. The vapours evolved in the first part of the chamber are caused to bubble through a supply of residual oil maintained in the second part of the chamber on their way to the dephlegmator. The residual oil is thus well stripped of light products. Reflux from the dephlegmator is returned to the cracking coil.

T. A. SMITH.

**Cracking of [petroleum] oil.** J. DELATTRE-SEGUY, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,766,246, 24.6.30. Appl., 28.6.26).—Oil is heated at 1 atm. pressure to about 510° in a short heating coil and pumped into an expansion chamber maintained at 150 lb. pressure. The vapours are passed to a dephlegmator, the reflux from which is passed through a second (longer) heating coil and heated to about 455°, being finally delivered into the expansion chamber. The process is particularly applicable to oils containing solid matter (e.g., salt), the deposition of which in the expansion chamber is overcome owing to the high velocity of the oil through the primary heating coil.

T. A. SMITH.

**Cracking of petroleum oil.** J. PRIMROSE, Assr. to POWER SPECIALTY Co. (U.S.P. 1,769,459, 1.7.30. Appl., 11.1.23).—The tube furnace in which cracking tubes are heated is divided into two portions. In the first portion, where the tubes are exposed to radiant heat and in which the furnace walls are at high temperature, the tubes are enclosed in refractory material so that heat cannot be absorbed at too high a rate. In the other portion the tubes are enclosed in corrugated cast-iron cases so that a large surface is exposed to hot gases. To equalise the absorption of heat, the corrugations are increased in size on those tubes exposed to the cooler gases. A uniform heat absorption is aimed at, as the oil passes through the heating system.

T. A. SMITH.

**Continuous cracking still.** W. C. BAILEY (U.S.P. 1,766,209, 24.6.30. Appl., 20.9.26).—Oil is cracked in a horizontal still fitted with internal ribs and longitudinal rotating agitator. Oil and steel balls are fed into the still, the balls being caused to progress along the still by means of the agitator. Cracking is aided as a result of the oil being distributed in thin films over the balls to which the deposited carbon adheres until after traversing the length of the still it reaches a zone free from oil, deposited carbon together with the balls being then removed from the still. The carbon is then separated from the balls

by sieving and the latter are returned to the other end of the still. The balls are admitted to and removed from the still by means of piston valves, so that the still is never open to the atmosphere. T. A. SMITH

**Retort for distilling oil shale and the like.** M. R. KRAUL (U.S.P. 1,766,132, 24.6.30. Appl., 20.6.27).—A narrow rectangular retort has spaced shelves, extending inwards and sloping downwards, attached to the longer sides, the shale being fed in from the top and passing in a thin layer from shelf to shelf. The heat is applied through the sides of the retort. The gases are removed from the spaces under the shelves, and automatic gear for feeding the shale and removing spent material is provided. One end of the retort is removable and provides access to the shelves when these require to be repaired. T. A. SMITH.

**Transformation of hydrocarbons having a high b.p. into volatile hydrocarbons.** F. P. E. S. DUPLAN (B.P. 339,841, 20.1.30. Ger., 21.1.29).—Heavy hydrocarbons are heated in the liquid state under a pressure exceeding the vapour pressure in a furnace which comprises a series of straight parallel tubes, with removable ends to facilitate cleaning, embedded in a block of conducting material, *e.g.*, cast iron, which is subjected to the direct action of the flames of burners, and which has disposed at a certain distance above it a sheet of cast iron providing a motionless heat-insulating air cushion. The oil is circulated at a predetermined velocity (between 6 and 72 cm./sec. for a tube up to 10 mm. in diam.) and is maintained at about 360° for 1–1½ min. The heavy residual hydrocarbons are recycled and mixed with fresh stock to be treated.

H. S. GARLICK.

**Mixtures for addition to motor fuels to reduce "knock."** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,637, 10.7.29).—An anti-knock agent suitable for storage and transport consists of a mixture of at least 4%, and preferably 10–20%, of iron carbonyl with one or more arylamines (*N*-alkylated or -arylated and containing at least 3 carbon atoms); such amines should be soluble in the fuel or capable of being rendered soluble by an assistant such as alcohol. Lubricating oil and a colouring agent may be added if desired. Nineteen examples are given. H. S. GARLICK.

**Low-boiling hydrocarbon oils.** STANDARD OIL DEVELOPMENT Co., Asses. of W. K. LEWIS and B. MEAD (B.P. 318,521, 3.9.29. U.S., 4.9.28).—The formation of gum in relatively low-boiling, non-viscous hydrocarbons, *e.g.*, naphthas, particularly those produced by cracking, is reduced by the addition of pyrogallol in an amount less than 5 g. (normally about 0.1 g.) per gal. of oil. [Stat. ref.] H. S. GARLICK.

**Recovery of natural gasoline.** W. G. LAIRD, Assr. to HEAT TREATING Co. (U.S.P. 1,769,698, 1.7.30. Appl., 10.6.27).—Gas containing gasoline is treated with absorption oil under a pressure of 150–400 lb./in.<sup>2</sup> The oil is passed to a rectifier consisting of a bell-cap tower, where it is heated under pressure, heat-exchanging elements being incorporated in the tower for this purpose, and the lighter gases are removed. The oil is then treated in a similar tower for the recovery of gasoline at atmospheric pressure. T. A. SMITH.

**Reclamation of used lubricating oil.** AKTIEB. SEPARATOR (B.P. 318,626, 4.9.29. Swed., 8.9.28).—Used lubricating oil is freed from diluting light oil fractions, water, and solid impurities, and the viscosity is restored, by repeated centrifuging at 90–120° in a centrifugal separator provided with arrangements for producing a strong air current in the collecting vessel, any impurities remaining in the oil being then removed by filtration or by treatment with bleaching material and subsequent centrifuging and/or filtration. Means may be added for condensing and collecting the light oil fractions distilled off from the oil during centrifuging. H. S. GARLICK.

**Manufacture of oil gas.** P. HAHN (U.S.P. 1,788,055, 6.1.31. Appl., 21.3.24. Ger., 12.2.24).—See B.P. 235,625; B., 1925, 661.

**Bleaching of mineral oils and fats.** W. POSTH and F. BESEMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,788,204, 6.1.31. Appl., 19.8.27. Ger., 21.7.26).—See B.P. 274,828; B., 1928, 702.

**[Machine for] manufacture of coal balls and the like.** SAHUT, CONREUR & CIE. (B.P. 341,255, 8.1.30. Fr., 25.2.29).

**[Firing device for] furnaces fired with pulverised fuel.** H. GARREAU (B.P. 341,029, 5.7.29. Fr., 28.2.29).

**[Burner for] combustion of fuels.** R. ZANIROLI (B.P. 341,052, 6.8.29. Addn. to B.P. 278,374).

**Storage of compressed gases** (B.P. 339,899).—See I. **Modified mineral oils** (B.P. 337,750—1).—See III. **Peat for insulation** (B.P. 339,067).—See IX. **Detergent** (U.S.P. 1,767,345).—See XII. **Resins from petroleum distillates** (U.S.P. 1,766,927).—See XIII. **Curing of tobacco** (U.S.P. 1,768,142).—See XX.

### III.—ORGANIC INTERMEDIATES.

**Caution in working with ether.** NOLTE (Z. angew. Chem., 1930, 43, 979).—A serious explosion in working with ether was traced to the presence of ether peroxide. E. S. HEDGES.

**Separation of alkyl and aryl halogen; modification of the Stepanov method, with particular reference to the analysis of certain insecticides.** Q. LANDIS and H. J. WICHMANN (Ind. Eng. Chem. [Anal.], 1930, 2, 394–397).—Total halogen, in absence of volatile constituents, may be determined by refluxing the material with an excess of sodium in kerosene or xylene with the addition of a few c.c. of amyl alcohol (cf. Stefanov, B., 1907, 35). Alkyl halogen derivatives only are hydrolysed by heating at 110° in kerosene or xylene solution with a 15% butyl alcohol solution of potassium hydroxide; if the substance is appreciably volatile the mixture should be covered with a layer of the solvent, or else the hydrolysis should be conducted in a stoppered vessel. Total halogen, if volatile substances are present, may be determined by the second method by heating the mixture until the boundary between the layers begins to disappear, cooling, adding sodium, and refluxing for 2 hrs. The method is widely applicable, and is especially suitable for the separation of carbon tetrachloride and *p*-dichlorobenzene. H. F. GILLBE.

**Benzine synthesis.** FISCHER.—See II.

See also A., Feb., 160, Extraction of phenols from alkaline solution (VAVON and PAIRA). 193, Preparation of ethylene, using phosphoric acid (WEBER and WALTON). 195, Automatic distillation of ether, alcohol, chloroform, etc. (LAPP). 208, Preparation of 2-naphthalene derivatives (BARBOT). 211, Preparation of tetryl (DESERGNES). 212, Preparation of *p*-nitrophenol (POPOV). Preparation of picramic acid (CLAYTON). 213, Derivatives of *m*-xylenes (ROWE and others). 225, Anthracene derivatives (BARNETT and GOODWAY). 226, 5 : 6 : 5' : 6'-Dibenzo-*NN'*-dihydro-1 : 2 : 1' : 2'-anthraquinoneazine (SCHWENK and WALDMANN). 265, Bacterial oxidation of phenols and dimethyl-*p*-phenylenediamine (HAPPOLD). 266, Phosphoric esters produced during lactic fermentation (VIRTANEN and TIKKA). Biochemical preparation of optically active glyceraldehyde (NEUBERG).

## PATENTS.

**Polymerisation of diolefines.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 338,534, 17.8.29).—In the polymerisation of diolefines an alkali metal or alkaline-earth metal or alloy is used in the form of particles uniform in size within 10%; *e.g.*, the metal may be comminuted in a liquid and sieved. Spheres 0.1–1.5 mm. in diam. are suitable. C. HOLLINS.

**Manufacture of methylene ethers.** IMPERIAL CHEM. INDUSTRIES, LTD., T. BIRCHALL, and S. COFFEY (B.P. 338,624, 30.8.29).—A mono- or di-hydric aliphatic, alicyclic, or aralkyl alcohol is heated with hexamethylenetetramine and aqueous or anhydrous hydrochloric or sulphuric acid. Methylene diethyl (b.p. 88–90°) and di-*n*-butyl (b.p. 180–182°) ethers and 1 : 3-dioxan, b.p. 100–103° (from trimethylene glycol), are thus prepared. C. HOLLINS.

**Manufacture of 1 : 3[ $\alpha$ ]-butylene glycol by hydrogenising paraldol.** I. G. FARBERIND. A.-G. (B.P. 338,807, 30.1.30. Ger., 30.1.29).—Paraldol is hydrogenated under pressure (*e.g.*, 20–25 atm.) in presence of a hydrogenation catalyst (*e.g.*, nickel on pumice at 30–50° in water or an alcohol). C. HOLLINS.

**Manufacture of glycollic acid esters.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,609, 8.11.29).—Water-free alkali chloroacetates are heated with anhydrous mono- or poly-hydric alcohols in absence of esterifying agents; *e.g.*,  $\text{CH}_2\text{Cl} \cdot \text{CO}_2\text{Na} + \text{EtOH} = \text{OH} \cdot \text{CH}_2 \cdot \text{CO}_2\text{Et} + \text{NaCl}$ . The preparation of *n*-butyl glycolate (yield 90–95%), ethyl glycolate, b.p. 66–67°/10 mm., and  $\gamma$ -hydroxy-*n*-butyl glycolate is described. C. HOLLINS.

**Manufacture of anhydrides of organic acids [acetic anhydride].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 338,507, 9.8.29).—An aliphatic acid,  $\text{R} \cdot \text{CO}_2\text{H}$ , is passed with the corresponding ketone,  $\text{R}_2\text{CO}$ , in vapour form over broken porcelain, copper, yellow brass, or aluminium sulphate at 600–700°, whereby the acid anhydride and the hydrocarbon,  $\text{RH}$ , are produced. The ketone may be produced *in situ*, *e.g.*, acetone from isopropyl alcohol, methyl acetate, or propylene oxide. Acetic acid and acetone give a 48% conversion into acetic anhydride. C. HOLLINS.

**Treatment [concentration] of aliphatic [acetic] acids.** H. DREYFUS (B.P. 338,187, 12.8.29).—Dilute acetic acid is extracted with a solvent, and water is precipitated from the extract by addition of a hydrocarbon or halogenated hydrocarbon, or other liquid which diminishes the solubility of water in the extract. Benzene, *e.g.*, is added to an ether extract of dilute acetic acid, the separated water is removed, and concentrated acetic acid is recovered by known means.

C. HOLLINS.

**Manufacture of [aliphatic] ketones [acetone].** H. DREYFUS (B.P. 338,518–9, [A, B] 19.8.29 and 28.5.30).—Ethyl or a higher aliphatic alcohol vapour is passed with (A) air or oxygen, or (B) air or oxygen and steam and/or the aliphatic acid vapour, at 430–480° over a catalyst consisting of an alkaline-earth oxide, hydroxide, carbonate, or weak acid salt. Ethyl alcohol over lime, calcium silicate, or calcium magnesium silicate gives acetone. C. HOLLINS.

**Manufacture of alkyl halogen compounds.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 338,742, 29.11.29. Holl., 8.1.29).—A paraffin hydrocarbon and chlorine or bromine are mixed in the vapour phase in the dark and heated—for methane to 380–400°, ethane 360–380°, propane or butane 330–380°, pentane 250–330°—without access of light. The main product is the alkyl halide; all the halogen is used up, and no carbonisation or flame results. C. HOLLINS.

**Manufacture of 2- and 3-hydroxy[hydro]carbazoles.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,821, 8.8.29).—A 2- or 3-alkoxytetrahydrocarbazole is distilled with lead oxide, preferably in a current of inert gas, whereby dehydrogenation and dealylation occur. C. HOLLINS.

**Manufacture of wetting, cleansing, emulsifying, and dispersing agents and preparations containing the same.** (A) J. Y. JOHNSON. From I. G. FARBERIND. A.-G., (B) I. G. FARBERIND. A.-G. (B.P. 337,737 and 337,774, [A] 27.4. and 8.7.29, [B] 27.4.29. Addn. to B.P. 306,116; B., 1930, 809).—(A) Aliphatic carboxylic and/or sulphonic acids or esters or acid halides above  $\text{C}_8$  are heated (at 150–180° if free acids are used) with hydroxyalkylamines, with or without subsequent alkylation or esterification or conversion into salts; protective colloids and/or organic solvents may then be added. (B) The products of the parent patent are converted into salts with acids (with or without alkylation) or bases, or are esterified. Examples are: (A) stearic acid with  $\beta$ -hydroxyethylamine, hydroxypropylamine, or hydroxybutylamine at 180°, and the product treated with oleyl chloride;  $\alpha$ -hydroxystearyl chloride with hydroxybutylamine and zinc chloride, alkylated; oleic acid with dimethyl- $\beta$ -hydroxyethylamine at 150–170°; (B) oleic acid with tri- $\beta$ -hydroxyethylamine at 170°, and the product neutralised with butylated naphthalenesulphonic acid; oleic acid with di-( $\beta$ -hydroxyethyl)hydroxybutylamine, sulphated; oleic acid or train oil acids with tri-( $\beta'$ -hydroxyethoxyethyl)amine at 180°, with 30% aqueous magnesium sulphate solution. [(A) Stat. ref.] C. HOLLINS.

**Manufacture of a basic product derived from oleic acid.** I. G. FARBERIND. A.-G. (B.P. 317,325, 7.8.29.

Ger., 13.8.28).—Oleyl chloride reacts with *p*- $\beta$ -diethylaminoethoxyaniline, b.p. 170°, in acetone to give the corresponding oleanilide (hydrochloride, m.p. 85–87°); the free base yields benzoate, lactate, and phenol-*p*-sulphonate in gel form on concentrating the aqueous solutions. C. HOLLINS.

**Modification of the physical properties (c) of organic isocolloids (A, D) consisting of or containing high-molecular organic acids (E) or their derivatives, (B, F) of resins, (G) of mineral oils.** L. AUER (B.P. 337,732–6, 337,750–1, 24.4.29. [B] Addn. to B.P. 321,724; B., 1930, 652. Cf. also B.P. 287,943, 289,414–5, 318,562, 321–689–92, 321, 722–7; B., 1928, 398; 1929, 806; 1930, 8, 652, 67).—Oils such as linseed oil, tung oil, castor oil, thickened or not, are “modified” (A) by heating with primary aromatic diamines or with high-molecular aromatic amines above 200° (above 250° if unthickened), with or without other modifying agents and/or a second metal-free organic compound; e.g., linseed oil with 5% of benzidine or *p*-phenylenediamine at 280–300° gives a base for water-resistant varnishes; the products may be vulcanised for rubber substitutes; or (B) by incorporation of 5–20% of an alkali, alkaline-earth, zinc, or magnesium soap at 100–250°, followed by vulcanisation for rubber substitutes. (E) By using a modifying agent insoluble in varnish solvents and adding a soluble secondary agent to promote the solubility of the modified product, there are obtained (e.g., from castor oil with 2% of lithium carbonate and 5% of *p*-cresol at 260°) bases for varnishes, lacquers, or leather cloth. (c, G) Highly modified isocolloids can themselves be used as modifying agents; (c) dissolved in linseed oil etc. they give light-coloured varnish bases or, on vulcanisation, rubber substitutes; (G) with mineral oils they give jelly-like greases; for these preferably a metal-free modified isocolloid is used. Resins are converted into liquids or into solids by heating at 280–300°, (B) with aromatic sulphonic acids or chlorides, (F) with chlorosulphonic acid etc. at reduced pressure, with or without other modifying agents. These products are useful in varnishes or may be vulcanised. C. HOLLINS.

**Manufacture of styrol [styrene] and its homologues.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 338,262, 10.9.29).—Phenylmethylcarbinol or its ether, or a homologue, is led in vapour form through a hot tube which may contain a dehydration catalyst, e.g., alumina, thoria, or tungsten oxide, on pumice or clay at 250–400°. Styrene is obtained in 90% yield from phenylmethylcarbinol over alumina on pumice at 290–300°.  $\alpha$ -Methylstyrene and *p*-ethylstyrene are similarly prepared. C. HOLLINS.

**Manufacture of [azoxy-]reduction products of nitro-compounds.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 337,563, 10.10.29).—*m*- and *p*-Nitrobenzoic hydroxyarylamides having a free position *ortho* or *para* to the hydroxyl group are reduced in alkaline solution, e.g., with glucose or sodium arsenite, to give azoxy-compounds, which have affinity for cotton and can be coupled on the fibre. The reduction of 3-*m*-nitrobenzamidophenol, m.p. 219°, 3-*p*-nitrobenzamidophenol, m.p. 215°, 4-*m*-nitrobenzamidophenol, m.p. 235–

237°, 3-nitroanisic 5-hydroxy-*o*-toluidide, m.p. 213–214° (azoxy-compound, m.p. 233–245°), 6-hydroxy-*m*-toluidide, m.p. 215°, and 2-hydroxy-*p*-toluidide, m.p. 209–210°, *p*-nitrobenzoic 4-hydroxy-*o*-toluidide, m.p. 255° (azoxy-compound, m.p. 285–287°), is described. C. HOLLINS.

**Manufacture of hydroxy-di- or -tri-arylmethane compounds [sulphonation products].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,808 and 337,832 [A] 6.8.29, [B] 9.8.29. [A] Addn. to B.P. 316,900; B., 1929, 888).—(A) The condensation products from aldehydes etc. and *p*-halogenated phenols (cf. B.P. 316,900, 330,893–4, 333,561, 335,547; B., 1929, 888; 1930, 896, 1017; 1931, 13) are treated with sulphonating agents in an organic solvent if desired. The products are moth-proofing agents. (B) The products of B.P. 316,900 (*loc. cit.*) are also obtained when in place of the aldehyde there are used acetals, aldehyde disulphites, or dichlorides, or chlorosulphites. C. HOLLINS.

**Manufacture of [halogenated] hydroxy-di- and -tri-arylmethane compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 338,126, 9.8.29).—*o*-Hydroxylated di- and tri-arylmethanes are chlorinated or brominated. The product from formaldehyde and 2 mols. of *p*-chlorophenol gives a dibromo-compound, m.p. 188°. 3:5-Dibromo-2-hydroxydiphenylmethane, m.p. 94°, and the 3:5-dichloro-compound, m.p. 81°, are also described. C. HOLLINS.

**Production of high-boiling complex diaryl compounds.** J. N. CAROTHERS, T. J. SCOTT, and FEDERAL PHOSPHORUS Co. (B.P. 338,631, 5.9.29. Addn. to B.P. 312,902; B., 1930, 453).—“90% Commercial benzene,” containing toluene and xylene, are preheated as in the prior process, and then heated rapidly to 800–850°, the products being quickly cooled. Higher diaryls, b.p. above 270°, are produced. C. HOLLINS.

**Manufacture of aminodiphenylamine compounds.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 338,240, 27.8.29).—Nitrosobenzenes, *p*-X·C<sub>6</sub>H<sub>4</sub>·NO, and *o*-Y·C<sub>6</sub>H<sub>4</sub>·NO, in which at least one of the hydrogen atoms X and Y is replaced by chlorine, alkyl, or alkoxy, are condensed together, e.g., by adding their acetic acid solution to sulphuric acid at 0–5°. The precipitated product is then reduced, e.g., with sodium sulphide. The following 4-aminodiphenylamines are described: 4'-chloro-2-methyl-, m.p. 82–83°; 4'-chloro-2-methoxy-, m.p. 94–95°; 2:4-dichloro-, m.p. 60–61°; 2:4'-dimethyl-, m.p. 73–74°; 2-methoxy-4'-methyl-, m.p. 94–96°; 2-chloro-4'-methyl-, m.p. 71–72°; 4'-chloro-, m.p. 70–71°; 4'-methyl-, m.p. 116–118°; 2-methoxy-, m.p. 87°; 2-methyl-, m.p. 70–71°. C. HOLLINS.

**Manufacture of urea derivatives [diarylcarb-amidedicarboxylic hydroxyarylamides].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 338,314, 18.10.29).—A dicarboxydiarylcarbamide is condensed in presence of a dehydrating agent with an aminophenol or -naphthol containing no carboxylic or sulphonic acid groups and having at least one free *o*- or *p*-position; alternatively, the acid chlorides may be used, or an aminoaroylamino-phenol or -naphthol is phosphorylated. The products have affinity for cotton and

may be coupled on the fibre. Phosgenated *p*-aminobenzoic acid is heated at 70–120° with phosphorus trichloride and *p*-aminophenol in dimethylaniline to give a product, m.p. above 300°; with 2-amino-*p*-cresol a product of m.p. 215–217° is obtained. Phosgenated *m*-aminobenzoic acid, m.p. 290–293°, with *p*-aminophenol yields a product, m.p. 255° (decomp.), or with *m*-aminophenol a product, m.p. 255–257°, which is also produced by phosgenating the reduction of 3-*m*-nitrobenzamidophenol, m.p. 219°. C. HOLLINS.

**Manufacture of mono-oxamic acids of 4:4'-diaminodiarlylcyclohexane compounds and of [azo] dyes derived therefrom.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 337,860, 20.8.29).—The products of B.P. 313,421 (B., 1929, 746), from cyclohexanes and *o*-toluidine, *o*-alkoxy- or *o*-chloro-anilines, are heated with 2.2–2.6 mols. of oxalic acid in water or other solvent to give mono-oxamic acids. These are diazotised and coupled with azo components and the resulting dyes may be hydrolysed, diazotised, and coupled again. Examples are: oxamic acid (I), m.p. 218° (decomp.), from cyclohexanone-aniline product; (II), m.p. 216° (decomp.), from cyclohexanone-*o*-toluidine product; (III), decomp. above 200°, from cyclohexanone-*o*-anisidine product; (IV) from cyclohexanone-*o*-chloro-aniline product; (IV) → Schaffer acid (orange on wool); (III) → benzoyl-K-acid (red-violet on wool); (II) → 2:8-dihydroxynaphthalene-6-sulphonic acid (blue-red on wool, bluer on hydrolysis); (I) → 1-(2-chloro-5-sulphonyl)-3-methyl-5-pyrazolone (yellow on wool), hydrolysed, → H-acid ← *p*-nitroaniline (yellow-green on wool); (II) → Cleve acid → phenyl-peri acid (black on wool, bluer black on hydrolysis); (II) → H-acid ← *p*-nitroaniline (greenish-blue-black on wool); (II) → salicyclic acid, hydrolysed, 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow on wool, yellow after chroming). C. HOLLINS.

**Manufacture of 2-[4]-aryolbenzantrones.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 338,764, 16.12.29. Addn. to B.P. 319,593; B., 1930, 136).—The 4-*ω*-cyano-*ω*-arylmethylbenzantrones, obtained according to the prior patent from aralkyl cyanides and benzanthrone, are oxidised in presence of an organic diluent, e.g., with chromic acid or permanganic acid in acetic acid or acetone. Phenyl and *p*-chlorophenyl 4-benzanthronyl ketones are described. C. HOLLINS.

**Manufacture of *o*-aminoaryl mercaptans [o-aminothiophenols].** R. HERZ and M. SCHUBERT, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,788,297, 6.1.31. Appl. 6.7.28. Ger., 9.7.27).—See B.P. 306,590; B., 1929, 316.

**Benzol etc. from fuel gases** (U.S.P. 1,760,335).—See II. **Non-corrosive alcoholic solution** (U.S.P. 1,771,985).—See X. **Condensation products from diolefines** (B.P. 338,109 and 338,152).—See XIII. **Aldol condensation product** (U.S.P. 1,754,865).—See XIV.

#### IV.—DYE STUFFS.

**Polypodium occidentale.** FISCHER and GOODRICH. —See XX.

See also A., Feb., 192, **Infusorial earth from Caucasus** (WEINBERGER). 209, **Deeply-coloured radical**

**hydrocarbons** (CLAR and JOHN). 226, 5:6:5':6'-**Dibenzo-*NN'*-dihydro-1:2:1':2'-anthraquinone-azine** (SCHWENK and WALDMANN). 227, **Fungus dyes: xylindein** (KÖGL and others). 238, **Indolenine-yellow** (KUHN and others).

#### PATENTS.

**Manufacture of vat dyes [of the benzanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 337,741, 4.7.29. Cf. also B.P. 306,874; B., 1929, 351).—3-Anthraquinonylamino-benzantrones containing one or more further anthraquinonylamino-groups but having a free 4-position are converted into vat dyes by alkaline fusion, followed if desired by acid condensation. 3:9-Di-*α*-anthraquinonylbenzanthrone with alcoholic potassium hydroxide at 180–200° gives an olive-brown vat dye, and on further treatment with sulphuric acid at 80–100° a green vat dye. Other starting materials are: product from 3:9-dibromobenzanthrone and 2 mols. of 1:6-diaminoanthraquinone (alkaline condensation, olive-brown); 3:10-dianthraquinonylamino-benzanthrone (dark brown); trianthraquinonylamino-benzanthrone (brown); product from 3:9-dibromobenzanthrone and 2 mols. of 1-amino-4-anilinoanthraquinone (alkaline condensation, olive-grey, and similar shade after treatment with sodium aluminium chloride); product from 3:9-dibromobenzanthrone and 2 mols. of 1-amino-5-benzamidoanthraquinone (olive-brown).

C. HOLLINS.

**Manufacture of stable reduction compounds of vat dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 338,104, 11.2.30. Addn. to B.P. 334,878; B., 1930, 1144).—6:6'-Dihalogeno-4:4'-dimethylthio-indigos are hydrogenated in aqueous alkaline suspension in presence of a nickel catalyst, e.g., at 110°/20 atm., to give a stable reduction product, insoluble in cold caustic alkali, useful for printing (cf. B.P. 334,907; B., 1931, 59). C. HOLLINS.

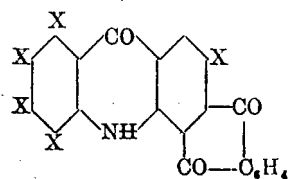
**Manufacture of arylamino-[anthraquinone] derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 338,412, 23.12.29. Switz., 22.12.28).—A leucohydroxyanthraquinone, which may contain amino- or alkylamino-substituents and/or sulphonic groups, is heated with a primary arylamine and boric acid in water and the product is oxidised. The leuco-compound may be produced *in situ*, and *β*-sulphonic groups may be removed during condensation. 1-Amino-4-hydroxyanthraquinone-8-sulphonic acid with *p*-aminophenol, boric acid, and sodium hyposulphite is heated at 40°, and then at 90–95° with acetic acid; alkaline oxidation gives 1-amino-4-*p*-hydroxyanilinoanthraquinone-8-sulphonic acid. 1:8-Diamino-4:5-dihydroxyanthraquinone-3:6-disulphonic acid and aniline gives 1:8-diamino-4:5-dianilinoanthraquinone and its monosulphonic acid. 1:8-Diamino-4:5-dihydroxyanthraquinone is similarly condensed with aniline. The non-sulphonated products are dyes for acetate silk or pigments for varnishes; the sulphonic acids dye wool. C. HOLLINS.

**Dyestuffs [of the anthraquinonephenazine class].** W. SMITH, S. G. WILLIMOTT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 338,486, 13.5.29).—1:4- or 1:8-Dichloroanthraquinone is condensed with *o*-nitroaniline in nitrobenzene in presence of sodium carbonate and



copper at 205–210°, and the di-*o*-nitroanilinoanthraquinone is reduced with sodium sulphide to the tetrahydroanthraquinonebisphenazine, which may be oxidised to the anthraquinonebisphenazine. C. HOLLINS.

**Manufacture of [vat] dyes of the anthraquinone-acridone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 338,182 and 338,216—7, [A] 7.8.29, [B] 5.7.29 and 5.5.30, [C] 6.7.29).—(A) 2:7:8:9-Tetrahalogeno-3:4-phthaloylacridones are obtained by halogenating (chlorinating if the 2-position is free) 3:4-phthaloylacridones, halogenated at least in the 8-position, but not in the 6-position, in an organic solvent; or by condensing a 1:4-dihalogenoanthraquinone-2-carboxylic acid or ester with a 2:3:4-trihalogenoaniline, and closing the acridone ring. The tetrachloro-compound is a bluish-red vat dye. (B) 3:4-Phthaloylacridones halogenated in 6:8- or 6:7:8:9-positions are obtained by condensing a 1-halogenoanthraquinone-2-carboxylic acid with aniline halogenated at least in *m*-position but having a free *o*-position, and closing the acridone ring; or by brominating a 3:4-phthaloylacridone halogenated in 6:8-, 6:7:8-, or 6:8:9-positions, to give a tetrahalogeno-derivative; or chlorinating such an acridone and removing one atom of halogen (*e.g.*, by dimethylaniline) to give a tetrahalogeno-derivative; or by removing one atom of halogen from a 2:6:7:8:9-pentahalogeno-3:4-phthaloylacridone. 6:8-Dichloro- (orange vat dye), 6:7:8:9-tetrachloro- (orange-yellow), and 6:8-dichloro-7:9-dibromo- (orange) -3:4-phthaloylacridones are described. (C) 2:6:7:8:9-Pentahalogeno-3:4-phthaloylacridones (annexed formula) are obtained by condensing



a 1:4-dihalogenoanthraquinone-2-carboxylic acid with a 2:3:4:5-tetrahalogenoaniline, and closing the acridone ring. Alternatively, a 6:8-di(or higher)-halogeno-3:4-phthaloylacridone is halogenated (or if the 2-position is free, chlorinated) in an organic solvent, *e.g.*, with sulphuryl chloride in nitrobenzene. The pentachloro-compound is a bluish-pink vat dye. C. HOLLINS.

**Manufacture of monoazo dyes [for wool and silk].** J. R. GEIGY SOC. ANON. (B.P. 337,577, 15.10.29. Fr., 15.10.28).—Dyes giving red shades on wool and silk from a neutral bath are obtained by coupling a diazotised *o*-aminophenol aryl or aralkyl ether with a 1-arylsulphonamido-8-naphtholdisulphonic acid. Examples are: benzenesulphonyl- or *p*-toluenesulphonyl-H-acid or -K-acid with diazotised *o*-aminodiphenyl ether or *o*-aminophenyl benzyl ether. C. HOLLINS.

**Manufacture of [azo or azoxy-]compounds having an affinity for cotton.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 337,902, 18.9.29. Addn. to B.P. 313,636 and 323,710; B., 1929, 672; 1930, 365).—Aromatic diaminocarboxylic acids carrying in one or both amino-groups a nitrobenzoyl substituent, or aromatic nitroaminocarboxylic acids similarly nitrobenzoylated, are submitted to mild reduction, *e.g.*, with alkaline sodium sulphide, to give azoxy- or azo compounds which can be diazotised and coupled on the

fibre. Examples are the reduction of sodium 2:5-di-(*p*-nitrobenzamido)benzoate, sodium 2-amino-5-*p*-nitrobenzamido benzoate or its acetyl derivative, and sodium 2-nitro-5-*p*-nitrobenzamido benzoate. C. HOLLINS.

**Manufacture of azo dyes containing chromium.** I. G. FARBENIND. A.-G. (B.P. 338,595, 22.8.29. Addn. to B.P. 306,843; B., 1930, 811).—The process of the prior patent is applied to aminosulphones derived from salicylic acid, in which the hydroxyl group is *ortho* instead of *para* to the sulphone group. C. HOLLINS.

**Manufacture of [water-soluble] azo dyes [for lakes and wool].** J. HUISMANN, H. SCHWEITZER, and R. STÜSSER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,788,299 and 1,788,300, 6.1.31. Appl., [A] 19.1.28, [B] 25.6.28. Ger., [A] 19.1.27, [B] 4.7.27).—See B.P. 283,897 and 293,352; B., 1929, 591; 1930, 51.

**Dis- and poly-azo dyes.** W. HENTRICH, M. HARDTMANN, R. KNOCH, and J. HILGER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,788,295, 6.1.31. Appl., 29.5.28. Ger., 10.6.27).—See B.P. 315,451; B., 1929, 809.

**Urea derivatives (B.P. 338,314). Azo dyes from mono-oxamic acids (B.P. 337,860).—See III.**

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Growth of moulds on sulphite pulp, and the accompanying decomposition of fibre.** W. DRECHSEL (Papier-Fabr., 1930, 28, 709–713, 729–736, 848–854; 1931, 29, 5–9).—The action of a number of moulds during their growth on unbleached and bleached sulphite pulps has been investigated. Pure cultures of the *Penicillium*, *Cladosporium*, and *Aspergillus* types were used, and their growth on pulps observed microscopically in moist chambers. In most of the cases examined the fibres are attacked, becoming after a time amorphous masses. As nutrient media other than the pulps themselves are rigorously excluded, the growth of the mould stops after a period of time which differs according to the type of mould. Bleached pulps are found to yield microscopic crystals of calcium oxalate, oxalic acid being a product of the action of the mould. These crystals are absent in the case of unbleached pulps, where the calcium content is low. The principal products of the action of the moulds are carbon dioxide, organic acids, and humic substances (not all identified), and traces of dextrose. The formation of carbon dioxide has been used as a means of measuring the extent of the action on the fibres, a stream of sterile air free from carbon dioxide being passed through the inoculated pulp under investigation. Carbon dioxide is determined as barium carbonate in the effluent air. Unbleached pulps yield greater quantities of carbon dioxide than do bleached pulps on a 75-day test, and the curve for daily production of the gas shows a high peak in the former case after 20–30 days. Similar experiments carried out in a stream of nitrogen yielded negative results, the moulds being anaerobic in the presence of the pulps. The loss in weight after washing the affected pulps with water varies from 3% to 6%, being highest for *Penicillium La coir*. Photomicrographs showing the effect of the moulds at various stages and tabular details of the time effects are given. T. T. PORTS.



**Destruction or recovery of sulphite [pulp] waste liquors?** M. STEINSCHNEIDER (*Papier-Fabr.*, 1931, **29**, 17). H. KÄSELAU (*Ibid.*, 18—20).—A contribution to a symposium on the subject of sulphite-waste disposal from the economic point of view. Statistics are given in support of the contention that the preparation of fuel from waste liquors is not economical.

KÄSELAU criticises the efficacy of utilising waste sulphite liquors for fuel from the viewpoint of the thermal value of the resulting fuel. T. T. POTTS.

**Determination of nitrogen and acetyl content of cellulose nitroacetate.** W. M. BILLING and J. S. TINSLEY (*Ind. Eng. Chem. [Anal.]*, 1930, **2**, 380—382).—After hydrolysis of the cellulose nitroacetate with 80% sulphuric acid the nitrogen is determined as usual with the nitrometer, and the residue, after dilution, is adjusted to  $p_H$  3.6—3.8 by means of phosphoric acid and sodium phosphate and steam-distilled from a special flask fitted with a sealed-on trap to prevent entrainment; precautions should be taken to prevent the entry of carbon dioxide into the distillate. By replacement of the free sulphuric acid by phosphoric acid prior to the distillation, decomposition of the cellulose and consequent high values for the acetic acid are eliminated. The average mean error of the acetyl determination, after application of a correcting factor to eliminate the effect of a small quantity of volatile acid derived from the cellulose nitrate, is  $\pm 0.5\%$ . The nitrogen determination by the nitrometer is less accurate with mixtures of the esters, and the error increases with decrease of the nitrogen content. H. F. GILLBE.

**Producing kraft paper from southern [American] pines.** R. H. STEVENS (*Chem. and Met. Eng.*, 1930, **37**, 734—735).—If, in the digester, 2% of the caustic soda is replaced by sodium hydrosulphide the pulp becomes stronger and methyl mercaptan is formed. With 15% of the active alkali as hydrosulphide the pulp acquires the characteristics of a sulphate pulp. Addition of further hydrosulphide has little effect until 50% is reached, after which the effectiveness diminishes. The alkali used varies from 15—18%  $Na_2O$  on the weight of dry wood for kraft pulp, to 25% for bleachable pulp. Digestion is carried out with live steam at 90 lb./in.<sup>2</sup> pressure, but the tendency to employ higher temperatures with shorter digestion period is indicated. A little of the black liquor from a previous digestion appears to have a catalytic effect in the digester. The pulp is separated on rotary filters, presses, or centrifugal machines. The alkali is recovered as carbonate by evaporation and furnacing, and these operations can be most economically effected by the use of a pressure concentrator in conjunction with a spray-in type of furnace. Saltcake is added and this is reduced in the furnace by the carbon dissolved from the wood to give the necessary hydrosulphide. In certain cases it may be economical to replace some of the saltcake by gypsum as the calcium sulphide formed saves lime in the subsequent causticising. D. K. MOORE.

**Maize stems as a raw material for paper production.** L. K. ARNOLD (*Iowa Eng. Exp. Sta. Bull.*, 1930, No. 100, 78 pp.).—Paper prepared from maize stems by digestion at 40—45 lb. pressure showed increasing

bursting and tensile strengths as the concentration of caustic soda used was raised from 5 to 20%, and a slight decrease with 25% soda. Digestion in water for 1 hr. at 50 lb. pressure followed by soda-digestion produced a paper of maximum bursting strength with 10% soda and maximum tensile strength with 20—25% soda. Similar tests with the outer fibre and the pith used separately are recorded. Ageing of the pulp considerably reduced bleach consumption. Pre-fermentation of the stems at 55° markedly decreased soda consumption. The strongest sulphate pulp from whole stems was obtained by digestion for 5 hrs. at 50 lb. pressure with 4.4% soda and 1% sodium sulphide. Wood kraft paper had 25% greater bursting strength and 50% less tensile strength than the best samples from maize stems. Trials by other pulpmaking processes are recorded.

A. G. POLLARD.

**Null-point method of potentiometric titration for determining acid or alkali in paper.** B. L. CLARKE and L. A. WOOTEN (*Ind. Eng. Chem. [Anal.]*, 1930, **2**, 385—387).—A null-point method employing the quinhydrone electrode in conjunction with the calomel electrode is applicable to the determination of paper acidity since the *E.M.F.* of the *N*-calomel cell at 25° is equal to that of the quinhydrone electrode in a solution having  $p_H$  7.03. The results obtained by this method differ from those given by the A.S.T.M. standard method by from —61 to +83%, the discrepancy being due to buffering of the paper extract by fatty materials; the indicator correction employed in the A.S.T.M. method is invalid, owing to this buffering action. The error of the null-point method is of the order of  $\pm 0.0002$  milliequiv./g. By variation of the potassium chloride concentration in the calomel electrode the method may be extended to the titration of weak acids or bases of which the neutralisation points lie between  $p_H$  6.13 and 7.67. H. F. GILLBE.

**Measurement of gloss [of paper] with the step-photometer.** H. SOMMER (*Papier-Fabr.*, 1931, **29**, 20—24).—The results of the examination of five papers by means of the Klughardt step-photometer are given.

T. T. POTTS.

**Application of dialysis etc.** BOUTARIC.—See I.

See also A., Feb., 202, **Action of acids on cellulose** (BOUCHONNET and others). 203, **Benzylcellulose** (OKADA). **Nitration of cellulose** (BERL and RUEFF). **Adsorption of bases by Willstätter's lignin** (KALB and others). **Beech-wood alkyl-lignins and their fission** (VON WACEK).

PATENTS.

**Preparation of bast fibres for spinning.** VICKERS, LTD., and O. D. LUCAS (B.P. 339,808, 23.12.29).—After de-seeding and removal of part of the woody matter, the straw is kept for 12—24 hrs. at 25° in contact with water containing a portion of liquor from the first stage of a previous treatment. A portion of the treating liquor is then run off, fresh water at 25° is added, and the temperature slowly raised to 37° and maintained thereat until retting is completed; the material is finally washed and dried. F. R. ENNOS.

**Pulping of fibrous material.** S. D. WELLS, ASSR. to MINE & SMELTER SUPPLY CO. (U.S.P. 1,769,811,

1.7.30. Appl., 7.11.28).—Fibrous vegetable material is soaked in cold caustic alkali solution and the soaked material is treated with steam at atmospheric pressure in a rod mill.

F. R. ENNOS.

**Manufacture of artificial wood.** S. SCHLESS (B.P. 339,853, 27.1.30).—A mixture of powdered calcium oxide or hydroxide and silica with cellulose or other fibrous material, wood meal, or wood dust is brought to a felt-like and loose condition and blown into a chamber, where it is allowed to settle. After compressing and heating, it is hardened by treatment with carbon dioxide at ordinary or raised temperature and finally oiled, impregnated, or polished as required.

F. R. ENNOS.

**Manufacture of [woollen] cloth [which develops neither gloss nor shine].** W. M. FULLER (U.S.P. 1,769,055, 1.7.30. Appl., 23.5.25).—The flexibility and lustre of wool fibres are reduced by the partial removal of the oil therein (by cleaning or scouring), and to compensate for this loss the wool is interlaced with strands of silk having greater flexibility and lustre, a portion of the silk being brought to the surface of the cloth during its manufacture.

F. R. ENNOS.

**Manufacture of cellulose [mixed] esters.** KODAK, LTD., Assees. of C. J. STAUD and C. S. WEBBER (B.P. 338,201—2, 16.8.29. U.S., [A] 18.2.29, [B] 25.2.29).—Cellulose acetate (or other ester) is digested with (A) an  $\alpha$ -hydroxycarboxylic acid or (B) a ketocarboxylic acid, e.g., in water at about 100° under reflux, whereby the new acid replaces a part of the acyl groups present. Examples are: cellulose acetate (33—40% acetyl) with (A) lactic, glycollic, tartaric, mandelic, malic, or (B) pyruvic or lævulic acid.

C. HOLLINS.

**Manufacture of viscose.** F. G. C. KLEIN (B.P. 314,504, 28.6.29. Ger., 28.6.28).—Soda lye of 5—13% concentration is emulsified at 0—5°, by vigorous mixing, with carbon disulphide and 4—20% of cellulose as such or in the form of alkali-cellulose.

F. R. ENNOS.

**Manufacture of artificial fibres.** I. G. FARBENIND. A.-G. (B.P. 339,740, 1.11.29. Ger., 2.11.28. Addn. to B.P. 307,829; B., 1930, 760).—Unripened viscose prepared from ripened instead of from unripened alkali-cellulose is used.

H. ROYAL-DAWSON.

**Manufacture of improved artificial silk from acylcellulose solutions.** I. G. FARBENIND. A.-G. (B.P. 318,646, 7.9.29. Ger., 7.9.28).—The coagulated thread, after leaving the spinning bath, is treated with a swelling agent (dilute formic or acetic acid, zinc chloride, thiocyanates) while under tension, and the stretched thread is afterwards passed through a hardening or setting bath during the winding operation. (Cf. B.P. 309,558; B., 1930, 986.)

F. R. ENNOS.

**Manufacture in continuous lengths of bodies which are hollow, or contain gaseous bubbles, or have a tubular structure from solutions or pseudo-solutions of cellulose esters or ethers.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 318,970, 10.9.29. Fr., 13.9.28).—One or more threads or solid film supports, which may or may not have the same composition as the cellulose derivative, are coated with the solution and the whole is heated so as to cause ebullition of the

volatile solvent; the supports may remain embedded in the manufactured product or may be separated.

F. R. ENNOS.

**Manufacture of [heavy] filaments, threads, etc. from organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 318,643, 7.9.29. U.S., 8.9.28).—Heavy filaments, such as artificial straw, bristles, etc. of denier 500—2000, are made by dry-spinning solutions containing at least 30% by wt. of organic derivatives of cellulose.

F. R. ENNOS.

**Manufacture of moisture-proof paper.** A. M. HINKSON (U.S.P. 1,769,513, 1.7.30. Appl., 30.7.28).—Equal parts of wax paper and asphalt waste are beaten with hot water and the resulting mass is sheeted and coloured.

F. R. ENNOS.

**Treatment of hard-paper material.** H. RÖMMLER A.-G. (B.P. 318,490, 29.8.29. Ger., 3.9.28).—In order to render it moistureproof, hard-paper material is coated on both sides with a liquid intermediate condensation product of a phenol with an aldehyde, or the coatings are applied by means of a carrier such as linen which has been dipped in or sprayed with the liquid artificial resin; the coated material is afterwards subjected to heat and pressure.

F. R. ENNOS.

**Treatment of cellulose sheets.** W. DÜTZMANN (B.P. 341,450, 12.10.29).

**Production of multi-coloured and/or marbled paper.** P. T. GOLDNEY (B.P. 341,621, 16.11.29).

**Laminated glass** (B.P. 339,762 and 339,872).—See VIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Dyeing of wool and silk with acid dyes.** K. QUEHL (Z. ges. Textilind., 1930, 33, 540—541, 558—559; Chem. Zentr., 1930, ii, 2057).—From the chemical point of view the behaviour of animal fibres towards acid dyes depends on the amino-acid and acid amide contents. Chromophoric groups, as in azobenzene or anthraquinone, do not enter into combination with these substances, but auxochromic groups do so. Acetic acid is less firmly held than tartaric or citric acid. According to these results the dyes combine with the amino-acids and amides; *o*-hydroxyazo compounds are fixed only when they also contain a sulphonic group. Adsorption processes are also concerned.

A. A. ELDRIDGE.

**Rotenone as a mothproofing agent [for wool].** E. A. BACK, R. T. COTTON, and R. C. ROARK (J. Econ. Entom., 1930, 23, 1014).—A solution of rotenone (up to 1—2%) in acetone forms an effective mothproofing agent for wool.

A. G. POLLARD.

**Testing mothproofing compounds.** W. MOORE (Ind. Eng. Chem. [Anal.], 1930, 2, 365—368).—Precautions necessary when conducting the half-circle and whole-circle Petri-dish methods of determining the efficacy of mothproofing compounds are described. In the former method the two specimens of cloth should be identical as regards texture, nap, and dye, and should be covered with a disc of muslin in order that the larvæ may operate between two layers of cloth, a condition for which they show preference. For the

determination of feeding preferences partly grown individuals should be used, whereas for investigation of the damage due to cocoon formation larvae ready to pupate should be employed; more than 10 larvae per dish should not be used. The whole-circle test indicates whether the preparation is toxic when consumed, and shows also that a toxic preparation will not prevent damage during cocoon formation. H. F. GILLBE.

**Sodium metasilicate.** VAIL.—See VII.

#### PATENTS.

**Bleaching [of furs, hairs, skins, feathers, etc.].** G. BAUM, Assr. to NIAGARA ELECTRO CHEM. CO., INC. (U.S.P. 1,759,341, 20.5.30. Appl., 9.7.28. Austr., 30.6.27).—The materials are bleached by exposure in a warm chamber (50°) to the vapours arising from stabilised 30% solutions of hydrogen peroxide contained in shallow trays. The skins of furs are protected by a previous brushing with a suitable fat. A. J. HALL.

**Dyeing of regenerated cellulose materials.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 338,111, 3.7.29).—An aminobenzoylphenylenediamine, or nuclear substitution product thereof, is tetrazotised and coupled with 1 mol. of a 2:8-, 1:8-, or 1:5-aminonaphthol-mono- or -di-sulphonic acid (or an *N*-substituted or azo derivative) or of a phenyl- or benzoyl-J-acid having no external amino-group and 1 mol. of a coupling component other than J-acid and aminoaryl-J-acids. Alternative routes are claimed. Examples are: *m*-aminobenzoyl-*m*-phenylenediamine with *N*.W.-acid and  $\gamma$ -acid (brown-red), or with salicylic acid and phenyl- $\gamma$ -acid (yellow-brown); *m*-aminobenzoyl-*p*-phenylenediamine with *N*.W.-acid and phenyl-J-acid (crimson) or with salicylic acid and H-acid (claret); *p*-aminobenzoyl-*p*-phenylenediamine with  $\gamma$ -acid (alkaline) and M-acid (claret), or with  $\gamma$ -acid (acid) and H-acid (maroon); *p*-aminoacetanilide-3-sulphonic acid  $\rightarrow$  *N*.W.-acid, hydrolysed, *m*-nitrobenzoylated, reduced,  $\rightarrow$  phenyl- $\gamma$ -acid (copper-red); *p*-nitroaniline  $\rightarrow$  *N*.W.-acid, reduced, *m*-nitrobenzoylated, reduced,  $\rightarrow$  S-acid (blue); cresidine  $\rightarrow$  cresidine, *m*-nitrobenzoylated, reduced,  $\rightarrow$  H-acid (green-blue). C. HOLLINS.

**Dyeing of esters or ethers of cellulose or its transformation products.** I. G. FARBENIND. A.-G. (B.P. 338,095, 9.4.30. Addn. to B.P. 304,739; B., 1930, 506).—Acetate silk etc. is dyed with 4-amino-1:8-naphthalhydrazide,  $\text{NH}_2 \cdot \text{C}_{10}\text{H}_5 < \begin{smallmatrix} \text{CO} \\ \text{CO} \end{smallmatrix} > \text{N} \cdot \text{NH}_2$ , m.p. above 350°, or the phenylhydrazide, in greenish-yellow shades. Other *N*-substituted derivatives may be used. C. HOLLINS.

**Dyeing and printing of textile fibres.** A. CARP-MAEL. From I. G. FARBENIND. A.-G. (B.P. 337,846, 13.8.29. Addn. to B.P. 324,119; B., 1930, 364; see also B.P. 330,579; B., 1930, 897).—The products of the prior patents are applied to textiles by dyeing or printing, and are developed with a caustic alkali bath. C. HOLLINS.

**Dyeing and printing of cotton by means of mordant dyes.** DURAND & HUGUENIN A.-G. (B.P. 318,469, 2.9.29. Ger., 1.9.28).—Urea (about 5%, calc.

on the weight of printing paste) is added to printing pastes, whereby mordant dyes are more efficiently fixed and the resulting shades are faster to washing and soap. A. J. HALL.

**Immunisation of yarns of vegetable origin to direct-dyeing dyes.** SOC. CHEM. IND. IN BASLE (B.P. 319,584, 23.9.29. Switz., 21.9.28).—Cotton, linen, and regenerated cellulose artificial silk fibres are immunised by esterification with an anhydride or chloride of an aromatic carboxylic acid (e.g., phthalic anhydride, benzoyl or phthalyl chloride) in the presence of a tertiary base (e.g., pyridine, quinoline, triethylamine, diethylaniline) at a moderate temperature (about 60°) and without a pretreatment with alkalis or acids. The immunised fibres have a strong affinity for basic dyes but no affinity for acid dyes, and are thus distinguishable from fibres immunised with derivatives of aromatic sulphonic acids (cf. B.P. 284,358; B., 1929, 596). A. J. HALL.

**Printing patterns on silk and other fibres.** H. W. WHISTON (B.P. 337,878, 30.8.29).—Fabric is placed upon a "resist carrier" (a sheet of waxed paper or other material) and then pressed by a heated superimposed "pattern carrier" which may be a heated (electrically or otherwise) and patterned roller or plate; the fabric is then dyed, whereon the dyeing is resisted at those parts in which wax has been absorbed from the "resist carrier." A. J. HALL.

**Mercerisation of fabrics.** C. MOCKER (B.P. 337,646, 6.12.29).—Cotton fabric is mercerised directly from the wet state by passage successively through solutions of caustic soda which increase in concentration and fall in temperature. Alkali solutions having *d* 1.11 at 30°, *d* 1.16 at 25°, *d* 1.21 at 20°, and *d* 1.26 at 15°, may be used, but the exact conditions are determined by measurements of the shrinkages of the fabric as produced by the alkali solutions. Suitable apparatus is described. A. J. HALL.

**Bucking [alkali-boiling] process for vegetable fibrous material.** G. ULLMANN (B.P. 399,850, 27.1.30. Austr., 26.1.29).—Cotton, flax, and jute materials are boiled in a liquor containing caustic alkalis (e.g., caustic soda or potash) and a metallic hydroxide (e.g., of calcium, barium, strontium, iron, or aluminium) which forms insoluble soaps, a suitable emulsifying agent being added to solubilise such soaps which form during the boiling process; the removal of impurities from the textile materials is thereby effected more efficiently than when caustic alkali alone is used. A. J. HALL.

**Preparation of dull artificial silk.** NAAML. VENN. J. A. CARP'S GARENFABR. (B.P. 337,418, 31.7.29. Holl., 24.5.29).—Regenerated cellulose artificial silks are delustrated by immersion in a cold clear solution of magnesium silicofluoride which is then gradually heated to 70–90°, whereby silicic acid separates and is deposited within the silk. Alternatively, similar deposition of silicic acid within the silk is secured by addition of magnesium silicofluoride to the spinning solution or to the coagulating bath as employed in the manufacture of the silk. The delustring is fast to soaping at 50–60°, but not to bleaching with hypochlorites. A. J. HALL.

**Manufacture of artificial silk filaments [of modified lustre].** J. A. SINGMASTER (B.P. 339,603, 9.8.29. U.S., 8.5.29).—Fine particles (0.75 micron or less) of white inorganic pigments (*e.g.*, zinc oxide, titanium oxide), which have a different refractive index from that of the main material of the spun filament and are not reactive with the chemicals used in the manufacture of the latter, are first added to the solvent, which is afterwards mixed with the cellulose derivative (nitrate or acetate) in such proportion as will not impair the continuity or materially decrease the strength of the finished product. The solution containing the dispersed pigment is passed through at least part of the filtering system and spun into filaments. F. R. ENNOS.

**Improvement [softening] of fibrous materials.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 339,858, 24.8.29).—Fibrous materials of all kinds, but particularly artificial silks, are softened and rendered more flexible by impregnation, followed by drying, with an aqueous solution of a true sulphonic acid prepared from carboxylic acids of aliphatic saturated hydrocarbons (*e.g.*, stearic, palmitic, lauric, and myristic acids); the method of sulphonation may be that described in B.P. 288,612 (B., 1928,865). A. J. HALL.

**Strengthening of cotton, artificial silk, or sheets of regenerated cellulose.** I. G. FARBERIND. A.-G. (B.P. 317,085, 8.8.29. Ger., 10.8.28).—Cellulosic material is impregnated with an aqueous solution of glyoxal or a substance capable of yielding this substance and then dried at 50–60°. After such treatment, using a 2% solution of glyoxal, cuprammonium silk having tensile strength 174 g. and extensibility 6% per 100 deniers had values of 213 g. and 9.3%, respectively. A. J. HALL.

**Waterproofing of wool and woollen materials.** MERKEL & KIENLIN GES.M.B.H. (B.P. 338,391, 4.12.29. Ger., 13.12.28).—Yarns and fabrics composed of wool are treated with an aqueous soap emulsion of an oil or fat and then with a solution of salts of metals (*e.g.*, aluminium) the hydroxides and carbonates of which are insoluble or only slightly soluble in water; the wool material is then given a prolonged heating at 60–80°. A. J. HALL.

**Protection of wool, fur, hair, etc. against textile pests [mothproofing].** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 337,823, 8.8.29).—Substituted acylthiocarbamides of the types  $R \cdot CO \cdot NR' \cdot C(NH) \cdot SH$  and  $NHR' \cdot C(NH) \cdot S \cdot COR$ , *e.g.*, hexylophenyl-, valerylphenyl-, acetylphenyl-, or acetyllallyl-thiocarbamide, are applied as mothproofing agents; preferably 1–2% of the agent is deposited on the material from a solution in petrol, tetrachloroethane, alcohol, etc. C. HOLLINS.

**Treating fabric [with cellulose].** R. R. FULTON, Assr. to KOPPERS Co. (U.S.P. 1,767,663, 24.6.30. Appl., 7.6.27).—Fabric is "filled" with cellulose by first coating it with a cellulosic pulp, treating it with a gelatinising solution (calcium thiocyanate solution, b.p. 134–140°), washing or steaming out the gelatinising liquor, and subjecting the fabric to pressure. A. J. HALL.

**Treatment of fabrics.** H. DREYFUS (B.P. 339,300, 2.8.29).—For the production of ornamental or other

effects on fabrics composed of cotton or other cellulosic fibres mixed with animal fibres and/or cellulose esters or ethers, the material is heated with a solution of a mineral acid, mineral acid salt, or a salt having an acid reaction (aluminium or ferric chloride) of suitable concentration, and the cellulosic material is removed by washing or brushing without drying in the presence of the carbonising chemical. F. R. ENNOS.

**Manufacture of nitrated fabric [as a backing for lace or embroidery].** A. LONDON and J. DATLOW (U.S.P. 1,769,702, 1.7.30. Appl., 7.8.29).—Cellulose fabric is dipped in an aqueous solution containing 50–80% of nitric acid and 40–10% of sulphuric acid at 21–52°, removed from the solution, and exposed to the same temperature for 1–3 min.; after removal of excess acid by washing, the material is treated with a neutralising and afterwards with a fireproofing and softening solution. The backing is removable from the embroidered material by dissolution in caustic soda. F. R. ENNOS.

**Treatment of [cellulose acetate] textile materials.** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 338,190, 13.8.29).—Permanent crease marks in materials containing cellulose ester and ether fibres are avoided in wet-processing them in the rope form if they are first thoroughly wetted out in open-width by steeping in water for several hours, preferably in the presence of a wetting-out agent. A. J. HALL.

**Bucking of vegetable fibrous material.** G. ULLMANN (U.S.P. 1,787,880, 6.1.31. Appl., 24.1.30. Austr., 26.1.29).—See B.P. 339,850; preceding.

**Dyeing and like machines.** C. E. OLDROYD (B.P. 341,342, 20.3.30).

**Apparatus for wet treatment of hanks of textile material.** O. VON KOHORN (O. KOHORN & Co.) and A. PERL (B.P. 341,328, 3.3.30. Addn. to B.P. 274,885).

**Sulphonation products (B.P. 337,808).** Wetting etc. agents (B.P. 337,737 and 337,774).—See III. Reduction compounds of vat dyes (B.P. 338,104).—See IV. Rubber[–cellulose] material (B.P. 339,974). Storage of rubber [fabric] (B.P. 339,730).—See XIV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Purification of caustic soda produced by the lime method.** A. KRAUSE and W. KLUKA (Przemysl Chem., 1931, 15, 6–12).—Impurities present in caustic soda produced by the addition of lime to soda solution, and consisting chiefly of sodium carbonate, chloride, sulphate, and silicate, are most readily precipitated by concentrating the solution to a titre corresponding to 72–75%  $Na_2CO_3$  and cooling to 50°. Calcium carbonate should be present in excess during the concentration process, as the solubility of sodium carbonate and sulphate is thereby diminished, probably as a result of double-salt formation. R. TRUSZKOWSKI.

**Determination of sodium carbonate in sodium bicarbonate.** A. K. BABKO (Ukraine Chem. J., 1930, 5, [Sci.], 197–207).—Sodium carbonate present in bicarbonate in excess of 3–4% can be determined by the ordinary methods. Where the content is lower, it

can be determined colorimetrically by comparison of the colour obtained on addition of phenolphthalein with that given by a standard solution. R. TRUSZKOWSKI.

**Drying of salt [sodium chloride].** B. LAGRANGE (*Chim. et Ind.*, 1930, 24, 805—808).—A rotary, hot-air dryer is illustrated and briefly described, and the economics of the drying process are discussed.

A. R. POWELL.

**Sodium metasilicate as an industrial alkali.** J. G. VAIL (*Chem. and Met. Eng.*, 1930, 37, 736—740).—Granular sodium metasilicate ( $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ ) has been prepared, m.p.  $71.8^\circ$ . It can be shipped in wooden barrels; metal should be used in moist atmospheres. For silicates varying in composition from  $\text{Na}_2\text{O} \cdot \text{SiO}_2$  to  $\text{Na}_2\text{O} \cdot 4\text{SiO}_2$  the  $p_{\text{H}}$  value of their solution increases with increasing concentration to limiting values in each case, but the greater the proportion of  $\text{Na}_2\text{O}$  in the silicate, the greater is the  $p_{\text{H}}$  of solutions of equal concentration. To give solutions having  $p_{\text{H}}$  10.2 the concentration (wt.-%) necessary is: sodium hydroxide 0.003, metasilicate 0.01, carbonate 0.02, monohydrogen phosphate 0.025, sesquicarbonate  $>4.0$ . Its action on metals and uses as a detergent in the washing of fabrics, milk containers, etc. are described. D. K. MOORE.

**Oxidation of ammonium sulphite and preparation of ammonium sulphate from gaseous sulphur dioxide, ammonia, and water vapour.** S. I. VOLFKOVICH and D. L. TZIRLIN (*J. Chem. Ind., Russia*, 1929, 6, 1323—1332, 1403—1409).—Ammonium sulphite in aqueous solution is completely oxidised by air in towers filled with silica gel or quartzite. Alternatively, the compound is caused to react with calcium sulphate, or sprayed into air at  $70^\circ$ . The catalytic action of potassium permanganate, chromic oxide, vanadium pentoxide, selenium, copper sulphate, potassium nitrate, or potassium chloride was too small to be of practical value. The solid sulphite is only slowly oxidised by air; 30—35% oxidation is effected in 2—3 hrs. at  $60$ — $70^\circ$ . Ammonia, sulphur dioxide, air, and water vapour react in a Cottrell precipitator to form a mixture of sulphite and sulphate; the former may be decomposed by air at  $60^\circ$  and the gases recirculated.

CHEMICAL ABSTRACTS.

**Oxalate method of determining the titre of potassium thiocyanate, using borax.** N. A. TANANAEV and N. A. LAZARKIEWITSCH (*Ukraine Chem. J.*, 1930, 5, [Sci.], 209—212).—Potassium thiocyanate is converted into oxalate by evaporation with oxalic acid solution, the residue is converted into carbonate by ignition, excess of standard acid is added, and the excess is determined by titration with standard borax solution. The determination occupies 3—4 hrs., and its mean error is about 0.2%.

R. TRUSZKOWSKI.

**Determination of calcium and magnesium in dolomitic limestones by means of saccharate solutions. Alkalimetric method.** A. C. SHEAD and B. J. HEINRICH (*Ind. Eng. Chem. [Anal.]*, 1930, 2, 388—389).—The limestone is decarbonated by ignition at  $1000^\circ$ , disintegrated and hydrated by addition of water, and cooled to the ordinary temperature with exclusion of carbon dioxide. An excess of 30% sucrose

solution is added, and after vigorous shaking at a low temperature the calcium monosaccharate solution is removed by filtration through filter-paper pulp in an apparatus designed to prevent entry of carbon dioxide; rise in temperature induces the formation of insoluble tricalcium saccharate. After washing, the precipitate is digested with 0.2*N*-hydrochloric acid near the b.p., and magnesium present determined by back titration of the excess of acid. The calcium cannot be determined by direct titration of the saccharate solution, as the latter is not of known composition; by digesting the limestone with standard acid, and back titrating for total calcium and magnesium, the calcium may be determined by difference. If much iron is present the method as given is not applicable, and the usual gravimetric procedure is also unsatisfactory since ammonium salts are usually not present in sufficient amount to prevent inclusion of magnesium hydroxide in the iron precipitate, and the calcium oxalate is not precipitated a sufficient number of times to render it free from magnesium; by the new method the total calcium + magnesium is satisfactory, but the distribution is incorrect.

H. F. GILLBE.

**Electrodialysis of Izjum phosphorite.** K. N. TARANOV (*Ukraine Chem. J.*, 1930, 5, [Tech.], 65—76).—Calcium and phosphoric acid can be quantitatively separated from Izjum phosphorite by electrodialysis (0.1—0.2 amp. at 15—20 volts, during 150 hrs.).

R. TRUSZKOWSKI.

**Possibility of reducing the amount of sulphuric acid necessary for the treatment of Izjum phosphorite.** M. A. EGOROV (*Ukraine Chem. J.*, 1930, 5, [Tech.], 43—64).—The extraction of phosphoric acid from phosphorites by 2% citric acid is enhanced by the addition of 2% of oxalic acid, or of alkali chlorides. The entire phosphoric acid may be extracted by using a solution containing 1% of sodium oxalate and 1% of sodium chloride. The quantity of sulphuric acid necessary for the extraction of phosphoric acid from a given weight of Izjum, but not of Podolian, phosphorite may be reduced to one third by the addition of oxalic acid, and the product so obtained is as efficient a fertiliser of sugar beet as is ordinary superphosphate.

R. TRUSZKOWSKI.

**Analysis of calcium carbide, and Czechoslovakian standards.** J. HERITES (*Chem. Listy*, 1930, 24, 493—498).—Caro's method is preferred to that recommended by Vondraček (*B.*, 1930, 659).

R. TRUSZKOWSKI.

**Analysis of calcium carbide.** R. VONDRÁČEK (*Chem. Listy*, 1931, 25, 10—14).—Polemical against Herites (cf. preceding abstract).

R. TRUSZKOWSKI.

**Anhydrous aluminium chloride, its uses and manufacture.** C. SIMON (*Chim. et Ind.*, 1930, 24, 1317—1324).—The use of aluminium chloride in oil cracking involves the use of very moderate temperatures and pressures, and gives excellent yields with little gas. Its main disadvantage is the cost of the catalyst and the difficulty of recovery when coated with carbon, and this has prevented the development of the process. Another process involving the large-scale use of aluminium chloride is the rectification of benzol, which has been the subject of experiments in France. A treatment with

4% of aluminium chloride replaces the usual acid treatment. The two processes most likely to yield aluminium chloride cheaply are those of MacAffee and Haglund. The latter process is being developed in Germany and Italy for the manufacture of aluminium. Published accounts of both are summarised, economic aspects are discussed, and it is considered that each has possibilities in suitable circumstances. C. IRWIN.

**Production of anhydrous aluminium chloride from the clays of the Donetz Valley.** I. E. ADADUROV (J. Chem. Ind., Moscow, 1929, 6, 1527—1530).—The clays (kaolinite 89.89—94.28,  $\text{Fe}_2\text{O}_3$  0.7—1.51%) are calcined at 600—750°, when  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  is formed; at 900° the free oxides are formed, and at 1000° sillimanite. The calcined clay is mixed with powdered coal, coke, or charcoal, briquetted with petroleum residues or coal-tar pitch, heated at 700—750° and then in a current of air for 15 min., and finally chlorinated for 8—10 hrs. The following reactions take place:  $\text{CO} + \text{Cl}_2 \rightarrow \text{COCl}_2 \rightarrow \text{CO} + 2\text{Cl}$ ;  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 + 3\text{C} + 3\text{CO} + 6\text{Cl} \rightarrow 2\text{SiO}_2 + 2\text{AlCl}_3 + 6\text{CO}$ . The condenser is maintained at such a temperature that the ferric chloride, m.p. 302°, condenses in the gas passages, while the aluminium chloride, subl. 183°, is collected in the vessel. The product contains 94%  $\text{AlCl}_3$ . CHEMICAL ABSTRACTS.

**Purification of regenerated nickel sulphate.** M. SERGEEV (Masloboino Zhir. Delo, 1929, No. 3, 57—62).—Solutions of nickel sulphate, regenerated from hydrogenation catalysts (e.g., Ni 1.58—4.28, Fe 0.21—0.84, Al trace—0.24, P 0.13—0.71%), are neutralised to methyl-orange, sodium carbonate is added with stirring at 50—60°, the solution is heated at 85—90°, and then filtered. The calculated quantity of copper sulphate is added in one portion, and the solution is heated to the b.p., an equivalent amount of sodium carbonate being then added and the mixture boiled for 10—15 min. If the solution still gives a positive thiocyanate test, further addition of sodium carbonate is necessary; if phosphorus is still present a small quantity of copper sulphate and sodium carbonate must be added.

CHEMICAL ABSTRACTS.

**Determination of lead peroxide and red lead.** G. BRUHNS (Chem.-Ztg., 1931, 55, 50—51).—The active oxygen content of lead peroxide can be determined by treatment with excess of ferrous nitrate solution and determination of the excess with potassium permanganate. The ferrous nitrate is prepared by mixing ferrous sulphate solution with a slight excess of lead nitrate, settling, and drawing off the clear solution. This is stirred with a weighed quantity of red lead etc. in a porcelain basin with addition of dilute nitric acid (free from nitrous acid) until all is dissolved. Permanganate solution is then run in. Neutral ferrous nitrate solution may be kept a few days without appreciable oxidation even when exposed to the air, or it may be covered with paraffin oil. Ferrous nitrate solution must not be heated, and it cannot be replaced by other ferrous salts. Examples of determinations show very consistent results.

C. IRWIN.

**Low-cost hydrogen produced by ammonia dissociation.** J. F. T. BERLINER and G. W. BURKE (Chem. and Met. Eng., 1930, 37, 751—754).—Because of the

reduction of cylinder space and compression charges, it is more economical to purchase synthetic anhydrous liquid ammonia and dissociate it into its elements by catalytic decomposition at 600° and under 1—15 atm. in an automatically controlled apparatus than to buy cylinders of compressed hydrogen. The presence of nitrogen is not detrimental to most uses of hydrogen, and in some cases may even be an advantage. By burning the dissociated ammonia with the correct quantity of air nitrogen is obtained more cheaply than it can be purchased compressed in cylinders.

D. K. MOORE.

**Recovery of iodine from phosphates.** H. JORDT (Chem.-Ztg., 1931, 55, 86).—Natural phosphates usually contain 30—70 pts. of iodine per million. One sample from Limburg contained 280 p.p.m. Iodine is actually produced as a by-product in one Danish superphosphate works. This is apparently produced electrolytically, as it contains appreciable quantities of mercury, but the process can hardly be profitable. The most likely method of economical production would be by separation from the exit gases, in which it exists partly as free iodine and partly as hydrogen iodide, by activated charcoal or by simultaneous washing with water and carbon disulphide, with treatment with hydrogen peroxide or ozone. The precipitation of sodium silicofluoride would not be impeded.

C. IRWIN.

**Hydrogen sulphide from water-gas.** FISCHER and DILTNEY. **Determination of carbon monoxide.** DITTRICH.—See II. [Alkali recovery from] pulp manufacture. STEVENS.—See V. Acid-proof products. BUDNIKOV and others.—See VIII. Potash from industrial alcohol. BROUGHTON and others.—See XVIII.

See also A., Feb., 170, **Purification of disodium phosphate** (LEMARCHANDS and TRANCHAT). 174, **Colloidal diaphragm for electrolysis of water** (JAUBERT). 176, **Catalytic formation of hydrogen cyanide** (BREDIG and ELÖD). 178, **Vanadous salts** (MEYER and AULICH). **Electrolytic preparation of zinc and lanthanum persulphates** (ARNEL). 185, **Rapid determination of selenium** (BENESCH and ERDHEIM). 186, **Determination of phosphoric acid** (VON ERDRÉDY). 265, **Reduction of nitrates by bacteria** (KORSAKOV). 266, **Bacterial reduction of mineral phosphates** (RUDAKOV).

PATENTS.

**Storing and transporting hydrocyanic acid.** HEERDT-LINGLER GES.M.B.H. (B.P. 339,717, 8.10.29. Ger., 22.10.28).—Deterioration of metal containers for hydrocyanic acid is avoided by completely expelling the air, this being effected by evacuating the containers and introducing an inert gas, or by closing them at the b.p. of hydrocyanic acid. W. J. WRIGHT.

**Purification of magnesium chloride.** A. K. SMITH and C. F. PRUTTON, Assrs. to DOW CHEM. CO. (U.S.P. 1,768,797, 1.7.30. Appl., 13.10.23).—A solution containing mixed magnesium and calcium chlorides is evaporated so that crystals of tachyrite separate, these being treated with a small amount of water to remove most of the calcium chloride. The residual

magnesium chloride, containing a small proportion of calcium chloride, is melted and evaporated until the b.p. is 160° and then cooled to 117°, the tachydrite formed being separated from the concentrated solution of magnesium chloride. W. J. WRIGHT.

**Bleaching and purification of earths, clay, china clay, quartz, silica, feldspar, bauxite, alumina, baryta, calcium and strontium sulphate and carbonate, and similar materials.** R. PERDU (B.P. 339,797, 18.12.29. Fr., 18.12.28).—During the preliminary washing the colloid matter present is peptised by the addition of non-electrolytic organic matter, such as an infusion of straw or bark. An oxidising material is then added to convert iron oxide into peroxide, which is itself converted into soluble iron protoxides by treatment with a weak acid or a weakly acid or basic salt; the bleached product is finally neutralised by means of water containing a small amount of lime.

W. J. WRIGHT.

**Manufacture of high-acid phosphates of iron.** M. GREEN and E. M. JONES, ASSRS. to PARKER RUST-PROOF CO. (U.S.P. 1,769,057, 1.7.30. Appl., 24.10.25).—Iron filings are introduced into a gaseous medium in a heated, slightly inclined, revolving drum, a 65% solution of phosphoric acid being sprayed over the filings; the temperature of all the materials is maintained at 100°.

W. J. WRIGHT.

**Storing and handling carbonic acid.** P. STAPP (B.P. 339,777, 30.11.29. Ger., 3.12.28).—Carbon dioxide, after compression, is passed into a condenser at a pressure of 65 atm. and at 15°, and thence to a storage vessel in which a pressure of only 4–5 atm. is maintained, the gaseous carbon dioxide thus formed being returned to the compressor. Oily impurities which collect on the surface of the liquid carbon dioxide in a frozen state are removed.

W. J. WRIGHT.

**Recovering carbon disulphide.** C. J. STROSACKER and J. I. JONES, ASSRS. to DOW CHEM. CO. (U.S.P. 1,768,803, 1.7.30. Appl., 25.1.26).—Loss of carbon disulphide during its manufacture, due to the presence of hydrogen sulphide, is prevented by passing the reaction products, after condensation of most of the carbon dioxide, through aqueous sodium hydroxide solutions.

W. J. WRIGHT.

**Recovery of sulphur from sulphides [or sulphide ores].** R. F. BACON (U.S.P. 1,769,819, 1.7.30. Appl., 2.6.27).—The sulphide (e.g., pyrites), strongly preheated, is alternately treated with oxygenated air, whereby the temperature is raised to 400–700°, and then with steam, the exhaust gases from the former treatment, after condensation of sulphur, being re-admitted to the furnace. No external heat is required.

W. J. WRIGHT.

**Production of concentrated sulphur dioxide.** SOC. CHEM. IND. IN BASLE (B.P. 339,926, 15.5.30. Switz., 15.5.29).—Sulphur dioxide in gaseous mixtures is absorbed by means of a countercurrent stream of water mixed with an aromatic amine (e.g., aniline) at 35°, and expelled by heat at 35–96°.

H. ROYAL-DAWSON.

**Compression of chlorine.** KREBS & CO. G.M.B.H. (B.P. 339,855, 28.1.30. Ger., 10.10.29).—The temperature of the chlorine is reduced in the compressor by admitting about 10% of liquid chlorine through the

suction tube. An air space round the cylinder avoids the necessity for water-cooling. W. J. WRIGHT.

**Treatment of marine algæ.** COMP. FRANÇ. DE L'ODE ET DE L'ALGINE (B.P. 319,333, 20.9.29. Fr., 20.9.28).—Treatment of seaweeds in a rotary digester with a mixture of water and a miscible organic solvent (e.g., alcohol, 65–75%, and/or acetone) under pressure (e.g., 1.5 kg./cm.<sup>2</sup> above atmospheric pressure), and at temperatures near the b.p. of the mixed solvent, effects dissolution of the saline and fatty substances, pigments, etc., destroys the insoluble organic iodine complex compounds, and liberates soluble iodides.

E. LEWKOWITSCH.

**Separation of zinc sulphate from sodium sulphate.** H. WEIDMANN (U.S.P. 1,787,806, 6.1.31. Appl., 6.10.27. Ger., 7.10.26).—See B.P. 278,747; B., 1928, 230.

**Manufacture of chromates.** MUTUAL CHEM. CO. OF AMERICA, ASSEES. OF O. F. TARR (B.P. 338,469, 22.3.30. U.S., 3.4.29).—See U.S.P. 1,752,863; B., 1931, 157.

**Zinc sulphide from gas** (U.S.P. 1,759,408).—See II. **Portable ozoniser** (U.S.P. 1,768,891).—See XI. **Fertilisers** (B.P. 339,969 and 340,120).—See XVI.

## VIII.—GLASS; CERAMICS.

**Firing round porcelain kilns with coal gas.** F. DIETRICH (Gas- u. Wasserfach, 1930, 73, 1066–1074, 1090–1096).—A detailed account is given of the practical methods adopted and of the theoretical considerations involved in converting two coal-fired porcelain ovens to gas-firing. The work was divided into three phases: investigation of a coal-fired oven, theoretical considerations before changing over to gas firing, and investigation of a gas-fired oven. The method of obtaining the necessary data for compiling thermal balances is explained and fully discussed. The trial kiln used for the gas-firing experiments had a glost-oven (lower chamber) capacity of 71 cub. m. In designing the fire-boxes and burners the object was to obtain sufficient mixing of the gas and air under low pressure to deliver a long, "soft" flame, in order not to cause excess loss of saggars. The thermal balance for the gas-fired kiln shows a total gas consumption of 13,790 cub. m., 3.57 cub. m. per kg. of ware, and 194 cub. m. per cub. m. of glost-oven room. The carbon monoxide content of the waste gases was considerably lower than with coal firing. Saggarr loss was reduced by about 30%.

F. SALT.

**Use of the autoclave in testing ceramic products for resistance to crazing.** R. G. MILLS (J. Amer. Ceram. Soc., 1930, 13, 903–914).—Further comparative results of service and autoclave tests are necessary to determine the true relationship between them, but investigations already indicate that such tests will become increasingly used. Tests were made on wall tile, dinner ware, sanitary ware, porcelain brick, glass bottles, and enamelled ware. With porous materials the crazing tendency in the same kind of body and glaze increases with increase in adsorption value, but the results cannot be applied to other bodies and glazes of different compositions. In the same way there is a critical condition of test for each particular ware. The pressure is probably the critical factor. The effect of



repeated tests is cumulative and more pronounced than a single test of equivalent total duration. The action of the autoclave was not found to be similar to that of the quenching or freezing test. Striking variations in the surface attack of different portions of the same piece of glass are noted.

J. A. SUGDEN.

**Advantages and use of a CO<sub>2</sub> meter on a forced down-draught periodic kiln.** D. M. McCANN (J. Amer. Ceram. Soc., 1930, 13, 944—946).—A general description and details of operation of a porcelain kiln are given. The meter enables a more uniform heating to be obtained.

J. A. SUGDEN.

**Corrosion of tank blocks by opal glasses.** C. J. UHRMANN and S. M. SLATER (J. Amer. Ceram. Soc., 1930, 13, 931—934).—A viscous layer, composed almost entirely of alumina, calcium fluoride, and alkali, was found on the bottom of the tank between the molten glass and the block. Subsequent comparative tests on the corrosion of a crucible filled with batch and slowly heated to 1370°, and another similar crucible first heated to 1370° and then filled with batch, indicated that during the slow heating the strongly corrosive ingredients of the batch first melt and without reaching a temperature high enough for reaction with the sand run to the bottom of the tank and form the viscous corrosive layer. By air-cooling the bottom blocks of the tank during melting, the corrosive layer was chilled and rendered thereby less corrosive, with a consequent decrease in the number of stones in the glass. The output of clear glass was also increased by filling the batch in three charges each of 1 ton instead of one charge of 3 tons. This allowed the bottom portion of the batch to heat up quickly enough to prevent separation of the corrosive layer.

J. A. SUGDEN.

**Effect of silicon carbide in clay bodies.** S. F. WALTON and E. L. HAUMAN (J. Amer. Ceram. Soc., 1930, 13, 935—940).—Tests on clay bodies containing 10—67% SiC by wt. show that replacement of the grog by silicon carbide greatly increases the transverse strength, resistance to slag attack, and thermal conductivity.

J. A. SUGDEN.

**Refractories in metallurgy.** H. FORESTIER (Chim. et Ind., 1930, 24, 797—804).—A review of the preparation, properties, and methods of testing refractory bricks of silica, magnesia, sillimanite, and mullite.

A. R. POWELL.

**Manufacture of heat- and acid-proof products without the use of fireclay.** P. P. BUDNIKOV, S. N. SHCHAREVITSCH, and I. G. SCHACHNOVITSCH (Ukraine Chem. J., 1930, 5, [Tech.], 93—104).—Clay is powdered and sieved (900 openings per cm.<sup>2</sup>), the fine powder is heated for 3 hrs. at 350—400°, and 85—95 pts. of the product are mixed with 5—15 pts. of fresh clay and made into a paste with a small quantity of 0.1% sodium silicate solution. The paste is pressed into moulds, dried for 2—5 days, and fired, the temperature being at first raised slowly (6—7 hrs. at 700°) and then more rapidly to 1350°, which temperature is maintained for 2 hrs.

R. TRUSZKOWSKI.

**Flux for chemical analysis of fused alumina refractories.** E. B. READ (J. Amer. Ceram. Soc.,

1930, 13, 941—943).—Complete dissolution of fused alumina refractories can be obtained by a single fusion with a mixture of sodium carbonate and borax, and the elimination of the boron by evaporation with methyl alcohol is not necessary for satisfactory commercial analysis.

J. A. SUGDEN.

**Micromanipulator.** HAUSER.—See XIV.

See also A., Feb., 161, **Adsorbed moisture in Kanbara clay (ISOBE)**. 169, **System Li<sub>2</sub>O—SiO<sub>2</sub> (KRACEK)**.

## PATENTS.

**Manufacture of glass [transparent to ultra-violet rays].** F. and R. WOLLNER, and F. NIKOLAI (B.P. 339,903, 12.3.30. Austr., 12.3.29).—Reducing agents, *e.g.*, aluminium, silicon, manganese, zinc, calcium, carbides, carbon, in quantity sufficient to reduce iron compounds to the metal, are added, before melting, to the raw materials for producing the glass.

L. A. COLES.

**Manufacture of compound glass.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 339,872, 7.2.30. Fr., 13.4.29).—Two glass sheets provided with superposed coatings of gelatin and celluloid as adhesive and an interposed cellulose acetate or nitrocellulose plastic sheet are compressed in a bath of a volatile liquid (*e.g.*, aqueous ethyl lactate, ethyl alcohol) which only when hot acts as a solvent for the plastic sheet; final adhesion is attained by heating the whole under gaseous compression in an autoclave.

L. A. COLES.

**Laminated products [*e.g.*, glass].** CELLULOID CORP. (B.P. 339,762, 18.11.29. U.S., 24.11.28).—Sheets of glass etc. coated with an adhesive are united by heat and pressure with an interposed sheet of plastic material comprising a cellulose derivative, non-volatile solvents or plasticisers (butyl tartrate, triacetin, etc.), and, if desired, stabilisers and dyes, in proportions such that the material flows readily under the heat and pressure applied.

L. A. COLES.

**Production of ceramic products.** M. LASLEY (B.P. 339,776, 28.11.29. U.S., 10.1.29).—Raw ceramic material containing organic matter is heated, preferably after crushing and grinding, at about 430° in the presence of oxygen prior to the usual firing at a higher temperature.

L. A. COLES.

**Abrasive implements.** BAKELITE CORP., Assees. of R. S. DANIELS (B.P. 319,017, 14.9.29. U.S., 15.9.28).—Porous abrasive wheels, especially those for use in the presence of water at a temperature liable to reach 100°, are immersed *in vacuo* until bubbling ceases in a bath of molten water-repelling material (naphthalene chlorination products higher than the dichloro-compound, *e.g.*, pentachloronaphthalene); when the bath has cooled almost to solidification point the articles are withdrawn and hardened in a blast of cold air.

L. A. COLES.

**Rolling and annealing of glass plates.** N.V. MIJ. TOT BEHEER EN EXPLOITATIE VAN OCTROOIEN (B.P. 341,800, 11.4.30. U.S., 13.4.29. Addn. to B.P. 325,185).

**Manufacture of sheet glass by drawing.** MONTANU. INDUSTRIELWERKE, VORM. J. D. STARCK (B.P. 341,171, 7.11.29. Czechoslov., 7.12.28).

**Manufacture of compound glass.** G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 341,631, 21.11.29).

**Bleaching of clay, silica, etc.** (B.P. 339,797).—See VII.

### IX.—BUILDING MATERIALS.

**Ground clay as a plasticising agent [in mortar].** M. G. SPANGLER (J. Amer. Ceram. Soc., 1930, 13, 927—928).—Tests on the tensile strength of two series of mortars, one containing hydrated lime and the other finely-ground clay, reveal no distinct difference between the plasticising powers of the two materials. There is a tendency for the lime mortars to be slightly stronger at the lower percentages of admixture, but weaker at the higher.

J. A. SUGDEN.

**Suggested toximetric method for wood preservatives.** H. SCHMITZ and others (Ind. Eng. Chem. [Anal.], 1930, 2, 361—363).—Various methods of determining the toxicity of wood preservatives are reviewed, and the need of a standard method is emphasised. A method is recommended wherein the influence of the preservative on the growth of a test fungus, preferably *Fomes annosus*, in nutrient agar during incubation for 14 days at 28° is determined. Since there appears to be no direct relationship between the concentration of preservative needed to inhibit growth and that which will kill the fungus, each should be determined independently. If a volatile preservative is under consideration, covered dishes or stoppered flasks should be used for the culture.

H. F. GILLBE.

### PATENTS.

**Working of lime and dolomite calcining kilns.** W. VOSS, and VULCAN-FEUERUNG A.-G. (B.P. 339,105, 27.11.29).—Water cooled nearly to 0°, so that it reaches the incandescent material in liquid form, is sprayed into the combustion and sintering zone. L. A. COLES.

**Manufacture of porous concrete by forcing air into cement slurries.** J. A. RICE, Assr. to BUBBLE-STONE Co. (U.S.P. 1,769,275, 1.7.30. Appl., 26.10.26).—Air bubbles are forced into a mixture of 100 pts. of cement slurry with 60 pts. of frothing composition comprising casein, water, calcium hydroxide, arsenious, benzoic, boric, and colloidal tungstic acids,  $\beta$ -naphthol, and diatomaceous earth solution, and the frothy mass is poured into moulds to set and harden. L. A. COLES.

**Waterproofing of concrete bricks and the like.** G. R. TUSTIAN (B.P. 340,112, 3.12.29).—The bricks etc. are coated while moist with a composition comprising about 1 pt. of cement,  $1\frac{1}{2}$  pts. of fine sand, and 2 pts. of powdered mica, and are then subjected for about 36 hrs. to steam-saturation in an airtight chamber and finally dried for 12 hrs. in the chamber with the steam supply cut off.

L. A. COLES.

**Production of artificial marble.** F. ROSÈS-GALCERÀN (B.P. 339,674, 10.9.29).—A highly-polished mould with the bottom constructed of glass etc. is charged successively with a mixture of (a) water, cement, and mineral colouring matter; (b) dry cement and sand; and (c) cement, sand, and water. After hardening and setting aside for about 4 days, the blocks are moistened with linseed oil.

L. A. COLES.

**Treatment of peat and similar [smoulder-proof] insulating materials.** E. DYCKERHOFF (B.P. 339,067, 30.10.29).—Peat or turf, in the raw state or after boiling, partial drying, or admixture with some of the dried product from a previous batch, is boiled under pressure in closed containers with phosphoric acid or phosphate solutions; after removal from the liquor the product is worked up to meal or made into sheets, blocks, etc., which may be impregnated with water-repelling materials.

L. A. COLES.

**Making bricks and such like.** PLATT BROS. & Co., LTD., and T. JENKINSON (B.P. 341,670, 14.12.29).

**Artificial wood** (B.P. 339,853). **Moisture-proof paper** (U.S.P. 1,769,513).—See V. **Anti-fouling coating** (B.P. 339,128).—See XIII. **Rubber compositions** (B.P. 339,002, B.P. 340,024, and U.S.P. 1,765,015).—See XIV.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Mechanism of the Thomas process [for production of steel].** K. THOMAS (Stahl u. Eisen, 1930, 50, 1665—1674, 1708—1712).—The effect of the form of the converter on the duration of the blow, the amount of iron oxidised, and the yield of steel in the Thomas process has been investigated on a series of 50 charges and the results are recorded in detail in 5 tables and 12 graphs. The best results were obtained when the temperature of the pig iron added exceeded 1250°.

A. R. POWELL.

**Mould coatings [for the iron foundry].** F. BEITTER (Stahl u. Eisen, 1930, 50, 1741—1744).—A review of modern methods of treating the surfaces of moulds for casting steel in order to produce castings as free as possible from surface blemishes.

A. R. POWELL.

**Influence of the emissive power on the measurement of the temperature of liquid iron.** R. HASE (Arch. Eisenhüttenw., 1930—1, 4, 261—264; Stahl u. Eisen, 1930, 50, 1813).—Comparative tests of the temperature of liquid cast iron during tapping from the furnace to the ladle showed that the optical pyrometer gives results which agree with those obtained with a thermocouple only when the brightest part of the stream just as it issues from the taphole of the furnace is observed; focussing of the optical pyrometer on the stream at lower points of its course gives lower results than the thermocouple, owing to progressive oxidation of the surface of the molten iron. Observations with the optical pyrometer on iron melted in a zirconia crucible in an argon atmosphere gave results lower than the true temperature by amounts varying from 85° to 140° as the true temperature was increased from 1250° to 1600°; oxidised surfaces gave results 10° below the true temperature when the surface was kept continuously oxidised, but progressively lower values than this when the atmosphere was kept stationary, thus allowing the oxide first formed to dissolve in the molten metal. Between 1250° and 1600° the emissive power of a clean surface of molten cast iron is  $0.44 \pm 0.03$  and that of an oxidised surface  $0.95 \pm 0.05$ .

A. R. POWELL.

**Dry granulation of liquid blast-furnace slag in granulating mills.** P. BERGER (Stahl u. Eisen, 1930, 50, 1775—1777).—The liquid slag is poured directly from the ladles in a stream 8 cm. in diam. into the refractory-lined hopper of the mill in which it meets three sprays of water, which supply just sufficient water to quench the slag; the slightly moist stream of slag then falls on to a series of three revolving beaters which throw it at a high speed against the sides of the mill, thus shattering it into coarse sand which is collected in trucks under the mill. The output is 20—30 tons/hr. with a moisture content of 7%. A. R. POWELL.

**Apparatus for the granulation of blast-furnace slag with water and air.** F. SPIES (Stahl u. Eisen, 1931, 51, 10—13).—The slag runs from the furnace along the usual channel where it is sprayed with a small quantity of water to granulate it; the sandy product is blown into storage bins by means of upwardly inclined currents of compressed air from the air-pipes feeding the furnace tuyères. A. R. POWELL.

**Regularity of the composition of basic open-hearth slag.** S. SCHLEICHER (Arch. Eisenhüttenw., 1930—1, 4, 239—244; Stahl u. Eisen, 1930, 50, 1778—1779).—Analyses of 29 basic open-hearth slags from various German steelworks showed that the sum of the lime, magnesia, and manganous oxide in the slag was  $61.4 \pm 1.5\%$ , whereas the manganous oxide varied from 12 to 28%. These results confirm the observations of Neu and of Janitzky that there is a definite relation between the manganese content of these slags and the sum of the lime and magnesia contents. Similarly there is a relation between the ferrous oxide content and the sum of the silica and phosphoric acid contents, a high acid content accompanying a low iron content and *vice versa*. These results show that addition of lime will reduce the manganese lost in the slag and addition of silica the amount of iron oxidised from the bath. On the assumption that the slag should contain not more than  $1.5\% \text{ P}_2\text{O}_5$  and  $2\% \text{ Al}_2\text{O}_3$  it is possible to calculate roughly the analysis of the slag from a knowledge of the contents of ferrous and manganous oxides. Thus with  $14\% \text{ MnO}$  and  $12\% \text{ Fe}$  the slag should contain  $47.5\% (\text{CaO} + \text{MgO})$  and  $18.5\% \text{ SiO}_2$ .

A. R. POWELL.

**Graphite formation in cast iron.** O. VON KEIL (Arch. Eisenhüttenw., 1930—1, 4, 245—250; Stahl u. Eisen, 1930, 50, 1718—1719).—Repeated melting and freezing tests on synthetic cast iron made from electrolytic iron and graphite showed that the iron solidified as white iron when the m.p. and f.p. were both low ( $1145^\circ$ ), but when both were high the metal contained needle-like graphite, and when the f.p. was low and the m.p. high the graphite separated in a finely-dispersed form. The needle graphite separates as such directly from the fused mass, and the fine graphite from the decomposition of a metastable solid phase. Experiments with pig iron with varying content of silicon and manganese showed that with increasing carbon content the metal solidifies first as white iron, then, at higher carbon content, the ledeburite first formed decomposes rapidly with the separation of fine graphite; in the neighbourhood of the eutectic composition, however, needle graphite separates

from the liquid phase owing to the retardation of the rate of cooling. Retarded and accelerated cooling tests on numerous cast irons have proved that for any given composition there is a critical rate of cooling above which the metal freezes in the metastable state and below which needle graphite separates from the liquid. Overheating of the metal reduces this critical rate of cooling to a very low value so that even after slow cooling the metal solidifies in the metastable state; repeated melting does not cause the metal to revert to its normal state.

A. R. POWELL.

**Nickel-vanadium and nickel-molybdenum cast irons.** J. CHALLANSONNET (Rev. Mét., 1930, 27, 573—603, 654—671).—Full experimental details are given of work the results of which have already been published (B., 1930, 512) and the effects of nickel and vanadium on the mechanical properties of cast iron are shown graphically; the results of similar work on nickel-molybdenum cast irons are also recorded. Nickel and vanadium both increase the compression, bending, and tensile strengths of cast iron, but the effect of these additions becomes less marked with increasing silicon content. Addition of  $0.25$ — $0.5\%$  Mo to cast iron containing  $1$ — $2\%$  Ni has little effect on the temperature of magnetic change, but it raises the Ac point, lowers the Ar point, and tends to inhibit the graphitisation effect of nickel. In grey cast iron molybdenum causes the graphite to become nodular and coarser and the hardness is increased irrespective of whether nickel is present or not. Molybdenum in small quantities also improves the tensile, compression, and bending strengths of cast iron.

A. R. POWELL.

**Nitrogen in commercial iron. IV. Simultaneous action of cold deformation and nitrogen precipitation on the magnetic properties of iron.** W. KÖSTER (Arch. Eisenhüttenw., 1930—1, 4, 289—294; Stahl u. Eisen, 1931, 51, 37—38; cf. B., 1930, 1068).—Thomas steel bars were stretched  $5$ — $20\%$  and annealed at  $100^\circ$  for 14 days to complete the separation of the nitride from solid solution; a second series of tests was made in which the annealing was done before the stretching and coercivity measurements were made of all the test pieces. In the first series the coercivity increased with increasing deformation before annealing, and subsequent annealing produced a still further increase in all cases; the total rise in coercivity decreased with increasing deformation, but annealing at  $300^\circ$  to redissolve the nitride resulted in the return of the coercivity to its original value. In the second series annealing at  $100^\circ$  produced a rise in the coercivity, which was still further increased by subsequent stretching, to an extent inversely proportional to the deformation. These results show that the specific effect of the precipitation of nitride on the coercivity decreases with increase of cold-work, and that the increase of coercivity may be considered as the sum of two effects, one due to cold deformation and the other to the precipitation of nitride; the first effect increases and the second decreases with increasing deformation. The coercivity of plain carbon steels increases linearly with the carbon content, irrespective of whether the cementite is globular or lamellar in character, but the rate of increase is smaller in the first case; the coercivity of a steel with a banded

pearlitic structure is 2—3 times that of a similar steel in which the cementite is in a globular form.

A. R. POWELL.

**Influence of silicon and manganese on the nature of the solidification of iron-carbon alloys.** O. VON KEIL and F. KOTYZA (Arch. Eisenhüttenw., 1930—1, 4, 295—297; Stahl u. Eisen, 1931, 51, 106).—Silicon acts as a promoter of the decomposition of carbide in cast iron cooled at rates up to 30°/min. The hypoeutectic region of the ternary iron-carbon-silicon alloys is characterised by a white-iron field and a field in which the graphite is present in a highly dispersed form. Addition of 0.3% Mn broadens the white zone, and at the eutectic carbon concentration causes the separation of needle graphite; with increasing addition of manganese the field of existence of needle graphite broadens until with 3.5% Mn the highly dispersed form of graphite no longer appears.

A. R. POWELL.

**Dependence of the hardness of carbon steels on the grain size of the iron carbide.** S. SHIMURA and H. ESSER (Stahl u. Eisen, 1930, 50, 1674—1675).—Steels with 0.6—1.75% C were annealed at 850° for 1 hr., quenched, and annealed for 10 min.—48 hrs. at 700—720°; the hardness was then determined and the grain size of the carbide measured. In all cases the hardness decreased almost linearly with an increase in the time of annealing, i.e., with increasing grain size of the carbide. The final hardness of all the steels after prolonged annealing was approximately the same, namely 121—142 on the Brinell scale.

A. R. POWELL.

**Stability of aluminium steels and of aluminium coatings on iron at high temperatures.** A. HAUTTMANN (Stahl u. Eisen, 1931, 51, 65—67).—The rate of diffusion of oxygen into aluminium-iron alloys at 1000° falls suddenly almost to zero with increase of aluminium between 4 and 9%; at the same time, the  $d$  of the alloys departs from the mixture rule in that the alloys are denser than the theoretical. Iron coated with aluminium by the sherardising ("Alitierung") process is much more resistant to scaling at 1000° than metal which has been coated by dipping into molten aluminium.

A. R. POWELL.

**Tensile properties of boiler plate at high temperatures.** A. POMP (Stahl u. Eisen, 1930, 50, 1737—1741).—The elastic limit, tensile strength, elongation, and reduction in area of 12 boiler-plate steels have been determined at the ordinary temperature and at 300—500°, and the results are shown in tables and graphs. These indicate that there is a relation between the tensile strength at the ordinary temperature and the elastic limit at high temperatures up to 500°.

A. R. POWELL.

**Examination of pre-Roman steel.** H. HANEMANN (Stahl u. Eisen, 1931, 51, 67—68).—Steel bars found in South Germany and dating from about 800 B.C. contained 0.44% C, 0.065% Si, 0.10% Mn, 0.042% P, and 0.012% S, as well as numerous slag inclusions. In spite of the presence of the slag, it has been found possible to forge fragments of this steel into fairly satisfactory knife blades, which can be hardened by suitable heat treatment. The microstructure of the steel as found reveals the presence of dark patches

consisting of cementite irregularly distributed in pearlite and light patches of ferrite with small inclusions of pearlite. No evidence of decomposition of cementite or of conversion of lamellar pearlite into the granular form was found.

A. R. POWELL.

**Hot-hardness characteristics of some modern tool steels and alloys.** E. G. HERBERT (Proc. Inst. Mech. Eng., 1930, 681—692).—The hardness of various high-speed cutting steels and alloys has been measured at temperatures up to 800° by means of the Herbert pendulum hardness tester, using a diamond point. The time-hardness of Widia metal falls almost linearly from 54 to 44 between 20° and 600°, then falls rapidly to below 30 at 800°. Ordinary high-speed steel behaves somewhat similarly, but there is a sharp fall at 500° and the hardness then remains constant up to 600°, above which a rapid decrease occurs; after superhardening, by the cloudburst method, the hardness of the same steel rises from 70 at 20° to 78 at 150°, remains constant to 300°, falls sharply to 68 at 400°, rises again to 75 at 420°, and remains constant at this value, which corresponds with 1000 Brinell, up to 600°, after which a rapid fall takes place to 30 at 700°. Nitrogen case-hardened steels show well-defined maxima in the hardness curve at 225° and 390° and a minimum at 350°. The maximum hardness of superhardened steels is obtained at a depth of about 0.0034 in. below the surface and the total thickness of this layer is about 0.22 in. The maximum hardness of nitrided steel (80 hrs. treatment) occurs at a depth of 0.0008 in. and a second maximum at a depth of 0.0075 in. Superhardening of cutting tools may be produced during use in cutting very hard materials.

A. R. POWELL.

**X-Ray study of grain size in steels of different hardness values.** W. A. WOOD (Phil. Mag., 1930, [vii], 10, 1073—1081).—Laue's mathematical results on the effect of particle size on the breadth of X-ray interference lines are applied to steels of different hardness. The half-breadths of the iron lines were measured photomicrometrically and found to be related to the hardness numbers of the steels. The relation between the hardness and the grain size of the steels, deduced from the half-breadths, is discussed, and agrees with the assumption that the hardness varies inversely as the superficial area of the grains.

N. M. BLIGH.

**Strength and structure of surface welds [in iron and steel].** O. DAHL and S. SANDELOWSKY (Stahl u. Eisen, 1930, 50, 1639—1642).—In making surface welds on steel, using a steel welding rod and the arc process, considerable loss of carbon takes place, so that with mild steel and a low-carbon welding rod maximum hardness and strength are obtained at the junction of the base metal and the deposited metal; with a high-carbon rod, however, the upper surface of the ridge has the maximum hardness. The microstructure of the welds consists of ferrite with irregularly embedded strips of pearlite.

A. R. POWELL.

**Manufacture and properties of non-rusting steels.** E. HOUDREMONT (Stahl u. Eisen, 1930, 50, 1517—1528).—A review of modern methods of making, working, and heat-treating non-rusting steels, with a brief account of

their microstructure and the effect of heat treatment thereon.

A. R. POWELL.

**Mechanism of ageing [of alloys].** G. TAMMANN (Z. Metallk., 1930, 22, 365—368).—Examples from recent work of the author and others are quoted in support of the theory that the increase in strength and hardness of an alloy during ageing is due to the accumulation of the atoms thrown out of solid solution along the slip planes, thus causing an increased resistance to slip by their interlocking action. Microscopical examination of a copper-aluminium alloy quenched and aged for 2 weeks at 200° shows that the copper is precipitated as minute needles of  $\text{CuAl}_2$  having a definite orientation to the crystals of the solid solution; these crystals gradually grow with the time of ageing and shrink in a similar manner to the coagulation and slow crystallisation observed in some cases of colloidal precipitation from solutions. Similar effects are observed in the separation of cementite from steels.

A. R. POWELL.

**Fatigue strength and resistance to slip [of metals].** P. LUDWIK (Z. Metallk., 1930, 22, 374—376).—Measurements of the hardness of electrolytic copper during fatigue tests under alternating stresses show that the hardness increases with the stress applied to a maximum of 65% above the normal hardness under a stress nearly equal to the fatigue strength. The values obtained with various non-ferrous metals in fatigue tests, using cylindrical rods, square bars with round notches, and rods with a circular groove, are recorded in a series of tables, and the effect of moistening the test piece with sea-water during the test has been determined. The fatigue strength in torsion tests bears the same ratio (0.575:1) to the fatigue strength in alternate-bending tests as the elastic limit in torsion bears to the tensile strength in static tests, except in the case of cast iron, where the graphite inclusions cause premature failure in the bending test.

A. R. POWELL.

**X-Ray study of the structure of metal systems.** A. WESTGREN (Z. Metallk., 1930, 22, 368—373).—A review of recent work on the study of the structure of alloys by means of X-rays, showing the value of this type of research in establishing the composition of compounds in alloy systems and the limits of solid-solubility of one metal in another.

A. R. POWELL.

**Flotation reagents [for minerals].** B. W. HOLMAN (Bull. Inst. Min. Met., Nov., 1930, 53 pp.).—The behaviour of the various reagents used as collectors, frothers, or modifiers in modern flotation practice is discussed and reviewed, and the effects of solubility, adsorption, dissociation, and structure of the reagents, their chemical and electrochemical action on the minerals, and the  $p_H$  of the circuit on the phenomena of flotation are described.

A. R. POWELL.

**Flotation of chalcopyrite in sea-water.** A. K. BURN (Bull. Inst. Min. Met., Nov., 1930, 10 pp.).—The copper ore at the Tocopilla mine, Chile, contains 4% Cu, all but 0.4% of which exists as chalcopyrite. The ore is stage-crushed to pass 40-mesh and floated in four air-agitation cells, using "Flotol" and xanthate in a sea-water circuit of  $p_H$  8. A good recovery from the cleaner cells is obtained in the form of concentrates

containing 27.5% Cu together with a small quantity of middlings assaying 5% Cu.

A. R. POWELL.

**Modern methods of concentrating minerals.** C. BERTHELOT (Rev. Mét., 1930, 27, 535—543).—A brief account of modern flotation practice as applied to zinc-lead ores.

A. R. POWELL.

**Sizing by elutriation of fine ore-dressing products.** A. M. GAUDIN, J. O. GROH, and H. B. HENDERSON (Ind. Eng. Chem., 1930, 22, 1363—1366).—An elutriator designed by the U.S. Bureau of Mines comprises a glass sorting-tube with a launder at the upper end to catch the overflow, and a funnel at the lower end connected to a Woulff's bottle, which is fed from a constant-level water tank. The rising-current velocities are adjusted by regulating the overflow per unit time, for which purpose the cross-section of the tube must be determined. The volume of water at 13.5° required for splits of various size and elutriators of various diameters is tabulated, and a second table gives the correction factors to be applied for other temperatures and when acetone is used instead of water. Acetone has the advantage that it reduces the settling time of mineral products to a quarter of that required with water. When sizes below 800-mesh are separated the pulp should be very thin, so as to lessen flocculation, and with such fine sizes it is also advisable to add sodium silicate as a dispersing agent.

W. J. WRIGHT.

**Experience with copper firebars and staybolts in England and Germany.** R. KÜHNEL (Z. Metallk., 1931, 23, 1—7).—German experiments on the causes and prevention of corrosion of locomotive firebars and staybolts have confirmed the results obtained by Hudson and others (B., 1929, 819). Hydrogen chloride and sulphur dioxide are the chief causes of rapid corrosion, and their effects are considerably enhanced by the leakage of water from the boiler. The addition of 0.45% Sn to the copper used in making the bars and bolts increases its fatigue strength at high temperatures, and this alloy appears to be almost as satisfactory in service as the alloy with a small percentage of silver recommended by the English workers.

A. R. POWELL.

**Notched-bar impact strength of some aluminium alloys, especially at low temperatures.** W. A. GÜLDNER (Z. Metallk., 1930, 22, 412—416; cf. B., 1930, 1072).—The effect of low temperatures (below —60°) on the impact strength of notched bars of aluminium alloys is only very slight, complete fracture taking place without a transition stage and the results obtained in static tests being much lower than those in dynamic. The energy consumed increases with the width of the test piece, but is always a small percentage lower than the proportional value and is little affected by decrease in temperature below the normal. Rolled alloys are particularly sensitive to the effect of even small notches, provided they are sufficiently deep to penetrate the rolling skin. The impact strength of notched bars is a parabolic function of the depth of the notch. An expression is deduced for this relationship, which is applicable to all aluminium alloys; hence the impact test is of little use in comparing the value of different aluminium alloys as constructional materials.

A. R. POWELL.

**Electrical conductivity of copper.** G. ELSNER and P. SIEBE (*Z. Metallk.*, 1930, 22, 397—401).—The effect of thermal and mechanical treatment on the electrical conductivity of electrolytic copper (99.8 and 99.9% Cu) and of standard fire-refined copper (99.8 and 99.64% Cu) has been investigated. Heat treatment of cold-drawn wires of these four types increases the conductivity to a maximum at 400—500°. The temperature range at which maximum conductivity is obtained is more restricted and lower the poorer is the quality of the copper. Annealing at temperatures above this range produces a progressive diminution of the conductivity of electrolytic copper, but the conductivity of fire-refined copper decreases slightly with the annealing temperature between 400° and 500°, then increases rapidly, but is always much below that of electrolytic copper. Subsequent cold-working of annealed copper wire decreases its conductivity again to an extent dependent on the amount of cold-work done; the effect of cold-work on the poorer qualities of copper is much less than that on electrolytic copper, possibly owing to the precipitation of impurities from solid solution during the previous annealing. A. R. POWELL.

**Dezincification of brass.** G. MASING (*Z. Metallk.*, 1931, 23, 23—25).—The addition of 0.02% As to 70 : 30 brass prevents dezincification, but does not protect the metal from corrosion in faintly acid media. A thin film of arsenic appears on the surface after some time and forms a local element with the brass; this causes corrosion to proceed more rapidly, without, however, causing secondary deposition of copper on the brass surface owing to the lower overvoltage of hydrogen at the arsenic cathode. A. R. POWELL.

**Influence of a third metal on the constitution of brasses.** III. Tin. O. BAUER and M. HANSEN (*Z. Metallk.*, 1930, 22, 287—391, 405—411; 1931, 23, 19—22; cf. B., 1930, 62).—Earlier work on the ternary system tin-zinc-copper is critically reviewed and the effect of additions of 1—5% Sn to brasses containing 50—70% Cu has been determined by micrographical and thermal analysis. The peritectic reaction  $\alpha + \text{liquid} \rightleftharpoons \beta$  takes place over a definite range of composition, indicating that the  $\alpha$ - and  $\beta$ -phases of the copper-zinc and copper-tin systems are isomorphous; the temperature of this reaction is lowered by 25°, 55°, and 85°, respectively, by the addition of 1, 2, or 3% Sn. The  $\beta$ -phase of the ternary system decomposes with fall of temperature into ternary  $\alpha$ - or ternary  $\gamma$ -solid solution, according to the composition; below 600° both  $\alpha$  and  $\gamma$  are produced simultaneously by the decomposition of the  $\beta$ -phase. Within a limited range of composition  $\beta$  is completely decomposed into the ( $\alpha + \gamma$ )-eutectoid below 600°. The boundaries of the  $\alpha$ - and  $\beta$ -fields are displaced towards the copper end of the diagram by increasing additions of tin and by rise in temperature. The solubility of tin in  $\beta$ -brass also increases rapidly with rise in temperature from 1.4% at 400° to above 6% at 700°, whereas only 0.4% Sn is soluble in  $\alpha$ -brass, hence  $\beta$ -alloys containing 0.4—1.5% Sn and 55—62% Cu yield a certain amount of brittle  $\gamma$  on slow cooling from above 400°. The composition of the  $\alpha$ -,  $\beta$ -, and  $\gamma$ -phases in stable equi-

librium with one another at 20—400° is as follows:  $\alpha = 62.3\%$  Cu, 0.4% Sn, and 37.3% Zn;  $\beta = 55\%$  Cu, 1.5% Sn, and 43.5% Zn;  $\gamma = \text{about } 52\%$  Cu, 8% Sn, and 40% Zn. Slowly-cooled alloys with 62—66% Cu and 0.5—1% Sn have a Brinell hardness of 44—60, those with 51—55% Cu and 1—3% Sn a hardness of 130—150, and those with 51—55% Cu and 5% Sn a hardness of over 200. Numerous micrographs showing characteristic structures of these ternary alloys are included together with a series of equilibrium diagrams. A. R. POWELL.

**Titanium-copper alloys capable of age-hardening.** W. KROLL (*Z. Metallk.*, 1931, 23, 33—34).—Titanium-copper alloys have been prepared by melting titanium (prepared by reduction of the tetrachloride with sodium in a nickel bomb) with copper in an atmosphere of purified argon; melting in air is impossible owing to absorption of oxygen and nitrogen. The two metals form a eutectic containing 24% Ti, m.p. 900°, and a limited series of solid solutions containing 3—4% Ti at 900° and 0.5% Ti at 350°. With less than 6% Ti the alloys can be forged, and with less than 3% Ti they are readily drawn into wire and rolled. The alloy with 3.05% Ti quenched from 850° has a tensile strength of 42.6 kg./mm.<sup>2</sup> with an elongation of 31.2%; after ageing for 24 hrs. at 350° these values become 73.4 kg./mm.<sup>2</sup> and 30%, respectively, and subsequent cold-rolling increases the tensile strength to 113.4 kg./mm.<sup>2</sup> but reduces the elongation to 6%. The electrical resistance of aged alloys is about one third that of the quenched alloys, but only the alloy with 1% Ti is suitable for use as an electrical conductor. A. R. POWELL.

**Determination of beryllium in aluminium.** H. V. CHURCHILL, R. W. BRIDGES, and M. F. LEE (*Ind. Eng. Chem. [Anal.]*, 1930, 2, 405—407).—The sample is dissolved in hydrochloric acid and heavy metals are precipitated by hydrogen sulphide. The filtrate is treated with an equal volume of ether, and hydrogen chloride is passed for 1 hr. after the liquid phase becomes homogeneous. The precipitated aluminium chloride is removed by filtration and the beryllium chloride in solution is converted into sulphate by heating with sulphuric acid. The residue is dissolved in water, filtered from silica, if present, and the beryllium is precipitated by neutralisation with ammonia, using rosolic acid as indicator. The precipitate is dissolved in hydrochloric acid and to the slightly acid solution at 60° an excess of 8-hydroxyquinoline is added; iron, aluminium, and titanium are precipitated by addition of 2*N*-ammonium acetate in slight excess, and the beryllium is finally precipitated by heating the solution to 60° and adding a slight excess of ammonia. The precipitate is ignited to oxide at 1000°. The method is more accurate than that of Havens (which does not give satisfactory results if the Al : Be ratio exceeds 100 : 1) and less expensive in application. H. F. GILLBE.

**Ageing of some aluminium sand-casting alloys.** W. SARAN (*Z. Metallk.*, 1931, 23, 32—33).—Sand castings of the light aluminium alloy, alufont (9.7% Zn, 2.1% Cu, 1.28% Fe, 0.25% Si, and 0.23% Mg), slowly increase in hardness on ageing at the ordinary temperature,



reaching a maximum after 12 days; a slow fall in hardness takes place over the ensuing 7 days, followed by a further hardening during the next 9 days. Daily variations in hardness which occur during ageing are attributed to small changes of room temperature. The elastic limit of castings of the aluminium alloy with 8% Cu increases by 21% in 12 days, and that of the alloy with 15% Zn and 2% Cu by 12% in 21 days; the corresponding increases in hardness are 6% and 10%.

A. R. POWELL.

**Expansion of light aluminium alloys during heat treatment [ageing].** H. SIEGLERSCHMIDT (Z. Metallk., 1931, 23, 26—30).—The expansion caused by precipitation of a constituent from solid solution during ageing of light aluminium alloys is first detectable at 150—200°, according to the composition, and reaches its maximum velocity at 210° with lantal, at 240° with the 5% Cu alloy, and at 230° with duralumin; the end of the precipitation process occurs at 230° with duralumin, but at higher temperatures in the other cases. Contraction due to redissolution of the precipitated constituents commences at 250° with lantal and at 300° with the other two alloys, and extends through a range of 180—200°. The thermal expansion curve of pure aluminium (99.8% Al, 0.15% Na) shows a slight minimum at 350° and a slight maximum at 280°, probably due to the high content of sodium. The coefficients of thermal expansion of the light alloys tested decrease in the range 300—500°, especially those of lantal and duralumin, which decrease to a sharp minimum at 470° followed by a sharp increase.

A. R. POWELL.

**Comparative tests with light-metal cylinder alloys.** M. VON SCHWARZ (Z. Metallk., 1930, 22, 417—419).—Comparative values for the hardness at 20—300°,  $\alpha$ , coefficient of thermal expansion, and running properties of nine light aluminium alloys, electron, and grey cast iron are shown in a table and three graphs. Of the aluminium alloys, quarzal (15% Cu, 0.5 Si, 6% Mn) showed the best running properties when used for the cylinders of internal-combustion engines, having a steady temperature curve and a constant moment of friction combined with a hardness of 160—180 at 20° and 110 at 250°. High-silicon alloys, on the other hand, are unsatisfactory, rapid rise of temperature and friction occurring with time of running.

A. R. POWELL.

**Rapid determination of calcium in lead alloys of low calcium content.** L. I. SHAW, C. F. WHITTEMORE, and T. H. WESTBY (Ind. Eng. Chem. [Anal.], 1930, 2, 401—402).—The alloy is dissolved in fuming nitric acid, a little water being added if the action is too violent, and the lead is removed by addition of sulphuric acid followed by filtration. To the filtrate a slight excess of ammonia solution and about 10% of 95% ethyl alcohol are added, and the calcium is precipitated by ammonium oxalate; the precipitate is removed, dissolved in dilute sulphuric acid, and titrated as usual with permanganate solution. The alcohol lowers the solubility of the calcium oxalate and renders unnecessary the addition of large quantities of ammonia, which retard the filtration and washing processes. A determination may be completed within 25 min. The

average deviation of the results from the mean is  $\pm 3.4\%$ .  
H. F. GILLBE.

**Determination of antimony in lead-antimony alloys of low antimony content.** L. I. SHAW, C. F. WHITTEMORE, and T. H. WESTBY (Ind. Eng. Chem. [Anal.], 1930, 2, 402—403).—Various permanganate and bromate methods for such determinations have been investigated and found to be unsuitable, and a modified permanganate method has been developed. The alloy (2 g.) is added to a mixture of 30 c.c. of concentrated sulphuric acid with 4 g. of fused potassium bisulphate heated to 320°; after heating for 20—30 min., the mixture is cooled, boiled for 15 sec. with 50 c.c. of water and 10 c.c. of hydrochloric acid ( $d$  1.16), diluted with 150 c.c. of water, and titrated slowly at 10—12° with potassium permanganate solution. The black residue formed prior to the titration is thereby eliminated and copper sulphate need not be added. The method is much more rapid than are the bromate methods, and a duplicate determination may be completed within 1 hr.

H. F. GILLBE.

(1) Beneficiation of ferruginous sands from the Great Victoria gold mine. (2) Concentration of hæmatitic ilmenite. (3) Flotation of sulphide ore from the Wiluna Gold Mines, Ltd. (4) Treatment of silver-lead ore from Durack's Lode, Kimberley gold field, N.W. Australia. (5) Treatment of ore from the Riverina Proprietary gold mine. (6) Bromocyanidation of Kalgoorlie ores. B. H. MOORE (Sch. Mines W. Australia, Bull., 1928, No. 3, 5—10, 11—13, 18—31, 32—36, 37—40, 40—47; 1929, No. 4, 3—5, 34—43).

(1) Slimes from the Surprise lead mine. (2) Cyanidation of "Lady Carman" pyritic gold ore. (3) Treatment of flotation concentrates. A. S. WINTER and B. H. MOORE (Sch. Mines W. Australia, Bull., 1927, No. 2).

**Refractories.** FORESTIER.—See VIII.

See also A., Feb., 157, System cadmium-magnesium (DEHLINGER). 158, Magnesium-cadmium alloys (GRUBE and SCHIEDT). System aluminium-magnesium silicide and aluminium-antimony alloys of high purity (DIX and others). Aluminium-iron-silicon alloys (FINK and VAN HORN). Strontium amalgams (DEVOTO and RECCHIA). System iron-phosphorus-silicon (HUMMITZSCH and SAUERWALD). 159, X-Ray study of iron and some iron-nickel alloys (ROBERTS and DAVEY). Transformation of austenite. (DAVENPORT and BAIN). 169, Equilibrium  $\text{FeS}_2 \rightleftharpoons \text{FeS} + \text{S}$  (DE RUDDER). 170, Iron-iron carbide-oxygen equilibrium (PINGAULT). 172, Electrode properties of stainless steel (NEWBURY). 173, E.M.F. of alloys formed in the wet way (PAGE). High-voltage anodic layer on aluminium (LILIENFELD and others). Passivity of chromium (MÜLLER and ESSIN). 178, Electrodeposition of gold (PAWECK and WEINER). 187, Electrodeposition of copper (HÖLEMANN).

PATENTS.

**Converter for bessemerising.** E. I. WILLIAMS (U.S.P. 1,768,649, 1.7.30. Appl., 22.4.29).—In a bessemerising process, e.g., for copper matte, air is introduced



through one set of tuyères until the lining adjacent is eroded, and then through a second set on the opposite side of the furnace which builds up accretions on the eroded part. The direction of the air is then alternated as required. C. A. KING.

**Metal-melting furnace.** PNEULEE, LTD., and H. J. PARRY (B.P. 339,802, 20.12.29).—The tapping plug of a crucible enters a pouring spout through the side of the furnace, which also is provided with a vertical slot or groove in the lining to give access for the tapping plug when the crucible is lowered into or removed from the furnace. C. A. KING.

**Treatment of a molten mass of metal or alloy, particularly a mass of molten steel, in a ladle.** F. KRUPP A.-G. (B.P. 339,579, 31.3.30. Ger., 30.4.29. Addn. to B.P. 312,063; B., 1930, 823).—When heating the steel etc. in a ladle by the heater described in the prior patent, the ladle is arranged in a chamber in which a vacuum is created. H. ROYAL-DAWSON.

**Melting of scrap metal.** HIRSCH, KUPFER- u. MESSINGWERKE A.-G. (B.P. 340,375, 15.11.29. Ger., 19.1.29).—The charge of loose scrap in the furnace is kept moving up and down to prevent the formation of bridges above the already molten metal. A. R. POWELL.

**Direct reduction of iron from its ore.** S. L. MADORSKY, Assr. to GATHMYS RES. CORP. (U.S.P. 1,762,622, 1.7.30. Appl., 25.11.25).—Preheated hydrogen is blown through a mass of molten iron oxide in a converter, and the excess hydrogen is burned above the surface of the molten mass by the injection of oxygen into the upper part of the converter; in this way the temperature is maintained sufficiently high to maintain the charge in a molten condition. The hydrogen and oxygen are obtained from the electrolysis of water. A. R. POWELL.

**Production of sponge iron from iron ores.** F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 340,641, 11.10.29. Ger., 11.10.28).—Gas from the iron blast furnace is passed at 800° upwards through a subsidiary shaft containing iron ore, part of the gas being burned in this shaft in order to maintain the temperature above 800°. A. R. POWELL.

**Treatment of gases containing sulphur dioxide from roasting furnaces to render innocuous combustible contact poisons, e.g., arsine. [Roasting of blende.]** METALLGES. A.-G. (B.P. 340,736, 6.12.29. Ger., 1.2.29).—Blende is roasted in Spirlet furnaces until its sulphur content is reduced to about 8.5% and then completely desulphurised in a Dwight-Lloyd apparatus. The gases from the second treatment may contain arsine due to reduction of the arsenic trioxide at the surface of the ironwork in the sintering apparatus, and are therefore passed through the Spirlet furnace to reoxidise the arsine to arsenic trioxide. A. R. POWELL.

**Desulphurisation of ferrous metal.** H. L. FREVERT (U.S.P. 1,770,395, 15.7.30. Appl., 21.2.28).—Sulphur may be removed from cast iron by treating the molten metal with sodium carbonate mixed with 10% of fluorspar or other common fluoride. A. R. POWELL.

**Hardening of cast iron.** W. MEACHER (B.P. 340,530, 27.9.29).—A thin copper sheet or shroud is placed over the part to be hardened and by the passage of a heavy current between the copper and the iron the former is caused to diffuse some distance into the iron, thereby considerably hardening it. A. R. POWELL.

**Manufacture of [steel] articles requiring high resistibility to the action of hot gases, vapours, and liquids, e.g., pressure receptacles.** F. KRUPP A.-G. (B.P. 340,819, 25.1.30. Ger., 23.4.29).—The steel used contains 0.05–0.2% C, 5–12% Cr, 0.2–1% Mo, and 0.2–0.5% V, and is heat-treated by quenching from above the Ac3 point and tempering at 500–750°. A. R. POWELL.

**Rust-proofing of iron or steel.** A. W. SCHLUCHTER, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,761,936, 3.6.30. Appl., 24.12.28).—The metal is heated at 480–980° in a current of ammonia containing 0.1% P or 0.1% S as sulphur dioxide. A. R. POWELL.

**Inhibitor [for use in pickling steel].** G. BARSKY, Assr. to AMER. CYANAMID CO. (U.S.P. 1,771,702, 29.7.30. Appl., 2.7.29).—The inhibitor comprises the product obtained by heating with 5–15% S the condensation product of an aldehyde (e.g., acetaldehyde) and an aromatic amine (e.g., aniline). A. R. POWELL.

**Non-corrosive [to iron] alcoholic solution.** W. S. CALCOTT and H. W. WALKER, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,771,985, 5.8.30. Appl., 5.10.28).—The corrosive action of dilute solutions of mono- or poly-hydric alcohols on iron and steel is prevented by the addition of small quantities of the condensation product of an aldehyde with ammonia or with aliphatic or aromatic amines, with or without the addition of carbon disulphide. Dilute alcohol solutions so treated are suitable for use as anti-freeze cooling media in automobile radiators. A. R. POWELL.

**Cast-iron alloy.** C. TAYLOR and H. L. GREENE, Assrs. to MOLYBDENUM CORP. OF AMERICA (U.S.P. 1,762,109, 3.6.30. Appl., 21.4.27).—A cast-iron alloy containing 1–4% Ni, 0.25–1% Mo, 0.7–2.5% Si, and 1.5–4% C is claimed. Sand castings have a Brinell hardness of about 200 and chill castings a surface hardness of 700–800. A. R. POWELL.

**Stable surface alloy steel.** R. P. DE VRIES (U.S.P. 1,768,578, 1.7.30. Appl., 20.1.26).—Steel containing 1–10% Cr, 1–6% Al, 0.5–5% Cu, and 0.05–1% C is claimed. The preferred composition is 1% Cr, 2% Al, 1% Cu, and 0.25% C. A. R. POWELL.

**Pickling of [chromium-iron] alloys.** L. L. SATLER, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,770,712, 15.7.30. Appl., 25.11.27).—The alloys are pickled in 50–70% nitric acid at 82–115°. A. R. POWELL.

**Manufacture of [chromium] steel alloys.** H. C. BIGGE and P. G. PARIS, Assrs. to BETHLEHEM STEEL CO. (U.S.P. 1,770,508, 15.7.30. Appl., 6.1.25).—Chromium ore is reduced in an electric furnace with an excess of ferrosilicon so as to obtain a ferrochromium alloy containing an excess of silicon. The slag is tapped off and discarded and the molten alloy tapped into a second furnace containing a charge of molten, low-carbon steel scrap. The resulting chromium steel is refined by

treatment with sufficient chrome-iron ore to remove the excess silicon and the metal is then tapped from below the slag. This second furnace and its slag content are then utilised for the reduction of further quantities of chrome-iron ore.

A. R. POWELL.

#### Manufacture of [nickel-chromium] alloy steel.

R. E. BISSELL, Assr. to THOMPSON PRODUCTS, INC. (U.S.P. 1,761,866, 3.6.30. Appl., 8.2.28).—An austenitic steel containing 11.5–13% Cr, 7–8% Ni, 2–3% Si, and 0.25–0.35% C is annealed at 900° for 2–6 hrs., then cooled slowly at 28°/hr. to 705°, and finally air-cooled to produce a martensitic structure. The alloy is suitable for the manufacture of valves for internal-combustion engines.

A. R. POWELL.

#### [Chromium-manganese] alloy steels.

E. ALLEN & Co., LTD., CHATWOOD SAFE Co., LTD., and L. K. EVERITT (B.P. 340,382, 20.11.29).—Steel containing 1–4% C, 15–25% Cr, and 11–20% Mn, preferably 3% C, 20% Cr, and 14% Mn, is claimed. Such steel is highly resistant to the oxyacetylene flame, is very hard, and withstands shock.

A. R. POWELL.

#### Froth-flotation concentration of minerals.

H. LAVERS, and MINERALS SEPARATION, LTD. (B.P. 340,598, 2.10.29).—Minerals containing soluble salts which act as flotation inhibitors, *e.g.*, ferrous sulphate, are ground with sufficient alkali dichromate, permanganate, persulphate, or chlorate to convert the inhibitor into an innocuous substance.

A. R. POWELL.

[Frothing] reagent for use in the [flotation] concentration of ores. C. G. QUIGLEY (U.S.P. 1,771,550, 29.7.30. Appl., 8.8.28).—The reagent comprises a metal salt of an alkylsulphuric acid, *e.g.*, calcium amylsulphate.

A. R. POWELL.

#### Refining of copper.

ELECTRICAL RES. PRODUCTS, INC., Assees. of J. E. HARRIS and J. H. WHITE (B.P. 340,497, 30.9.29. U.S., 8.3.29).—Electrolytic copper is melted in a crucible lined with alumina and the molten metal is deoxidised with calcium boride prior to casting. The product has a high electrical conductivity and is not rendered brittle by heating in a reducing atmosphere. [Stat. ref.]

A. R. POWELL.

#### Refining [degasifying and deoxidising] of copper.

M. G. CORSON (U.S.P. 1,769,986, 8.7.30. Appl., 26.4.27. Renewed 12.11.29).—Copper containing sulphur and oxygen is melted under reduced pressure, whereby the sulphur is completely removed as sulphur dioxide; the metal is then deoxidised with a mixture of calcium carbide with calcium or sodium fluoride or cryolite.

A. R. POWELL.

#### Welding of magnesium.

I. G. FARBENIND. A.-G. (B.P. 339,923, 12.5.30. Ger., 7.6.29. Addn. to B.P. 313,487; B., 1930, 1034).—The agent claimed in the prior patent is applicable also in the case of magnesium.

H. ROYAL-DAWSON.

#### Welding of magnesium and its alloys.

A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 340,894, 31.3.30).—A film of a metal which forms a solid solution with magnesium, *e.g.*, aluminium, zinc, or cadmium, is inserted between the heated pieces to be welded and pressure is applied.

A. R. POWELL.

**Aluminium alloys.** H. C. HALL and T. F. BRADBURY (B.P. 340,331–2, 1.10.29. Addns. to B.P. 323,353; B., 1930, 198).—(A) The magnesium content of the alloy claimed in the chief patent is reduced to 0.03–0.1%. (B) The molten alloy is treated with 0.01% of sodium just prior to casting.

A. R. POWELL.

#### Plating of metals with aluminium or its alloys.

A. R. and J. M. HETZEL (VEREIN. SILBERHAMMERWERKE HETZEL & Co.), Assees. of H. WACHWITZ (B.P. 340,498, 30.9.29. Ger., 3.10.28).—The metal to be coated with aluminium is provided with a thin, almost transparent film of alumina, a sheet of aluminium or one of its alloys is placed on this prepared surface, and, after heating to welding temperature, the composite metal is passed through heavy rolls so that the oxide layer is broken up and the aluminium welded to the base metal without forming therewith a thick brittle layer of intermetallic compound.

A. R. POWELL.

#### Zinc-base alloys.

NEW JERSEY ZINC Co., Assees. of W. MCG. PEIRCE and E. A. ANDERSON (B.P. 341,054, 9.8.29. U.S., 12.3.29).—A die-casting alloy comprising zinc with 0.05–2% Cu and 0.005–5% Ni is claimed. Part or all of the nickel may be replaced by any one or two of the following: cadmium, lithium, manganese, or magnesium. The alloys have a high resistance to slow plastic deformation and cold flow.

A. R. POWELL.

#### Production of high-grade nickel or nickel alloys.

BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (B.P. 340,632, 8.10.29. Austr., 29.4.29).—After a preliminary deoxidation with manganese or zinc, the molten metal or alloy is treated with up to 0.1% Li, preferably in the form of an alloy of 15% Li and 85% Mg. More magnesium may subsequently be added to render any sulphur present innocuous.

A. R. POWELL.

#### Manufacture of electrolytic iron.

R. D. PIKE (U.S.P. 1,769,605, 1.7.30. Appl., 13.3.26).—Sponge iron containing copper is dissolved in a countercurrent system by means of a solution containing ferric and ferrous chlorides and the resulting ferrous chloride solution is electrolysed to obtain electrolytic iron and ferrous ferric chloride solution which is returned to the leaching tanks. The final leach liquor from the clean-up of the head tank in the series contains copper, and is therefore by-passed from the circuit and treated with sufficient scrap or sponge iron to reduce all the ferric chloride to ferrous chloride and to precipitate all the copper; the purified liquor is passed directly to the electrolytic cells.

A. R. POWELL.

**Chromium-plating of brass or other metal articles.** METALS PROTECTION CORP. (B.P. 340,350, 11.10.29. U.S., 26.8.29).—The article to be plated is annealed at 300° to relieve internal stress, pickled, and chromium-plated in the usual way. Alternatively, the annealing and cooling may be carried out in a reducing or neutral atmosphere so as to avoid the necessity of pickling.

A. R. POWELL.

#### Mechanical [ore-roasting] kiln.

G. BALZ, Assr. to BALZ-ERZRÖSTUNG GES.M.B.H. (U.S.P. 1,787,504, 6.1.31. Appl., 29.3.28. Ger., 22.2.27).—See B.P. 317,341: B., 1929, 856.

Flotation concentration of ores, coals, graphite, etc. W. SCHÄFER (U.S.P. 1,788,331, 6.1.31. Appl., 25.4.28. Ger., 4.5.27).—See B.P. 289,848; B., 1928, 899.

Treatment of molten magnesium and its high-percentage alloys. A. BECK, W. SCHMIDT, and G. SCHREIBER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,788,185, 6.1.31. Appl., 20.8.28. Ger., 4.3.27).—See B.P. 311,998; B., 1929, 562.

Determining creep stress (B.P. 339,890).—See I. Ore-reduction furnaces (B.P. 340,117). Aro-welding electrodes (B.P. 339,930).—See XI. Coating [metal] surfaces (B.P. 338,983). Anti-fouling coating (B.P. 339,128).—See XIII. Securing rubber to metal (B.P. 339,421).—See XIV.

## XI.—ELECTROTECHNICS.

Electrolysis of montanic acid. FISCHER and HORN.—See II. Titration of acid or alkali in paper. CLARKE and WOOTEN.—See V. Electrodialysis of phosphorite. TARANOV.—See VII. Magnetic properties of iron. KÖSTER. Conductivity of copper. ELSNER and SIEBE. Titanium-copper alloys. KROLL.—See X. Rubber mixtures. JACOBS.—See XIV.

See also A., Feb., 143, Quantitative chemical analysis by spectra (LUNDEGÅRDH). 153, Magnetic properties of rhenium (AKULOV). Resistance of wires of iron, nickel, and of their alloys (MEDICI). 154, Magnetic properties of platinum- and palladium-cobalt alloys (CONSTANT). 172, Electrode properties of stainless steel (NEWBURY). 173, *E.M.F.* of alloys formed in the wet way (PAGE). High-voltage anodic layer on aluminium (LILLENFELD and others). Passivity of chromium (MÜLLER and ESSIN). 174, Diaphragm for electrolysis of water (JAUBERT). 178, Preparation of zinc and lanthanum persulphates (ARNEL). Electrodeposition of gold (PAWECK and WEINER). 187, Electrodeposition of copper (HÖLEMANN). 189, Tube furnaces for micro-analysis (THIESSEN).

### PATENTS.

Electric furnace [for treatment of metals and metal waste]. A. ISLIKER, Assr. to AJAX METAL CO. (U.S.P. 1,769,223, 1.7.30. Appl., 23.7.23. Renewed 26.6.29. Switz., 23.8.22).—Air or other gas, together with, if desired, scorifying or slagging agents, is blown into the bottom of the charge through one or more tuyères in an induction furnace or converter having one or more submerged induction channels or canals.

J. S. G. THOMAS.

Electric rotary furnaces particularly for reduction of iron ore. B. M. S. KALLING and C. V. DELWIG (B.P. 340,117, 7.12.29. Swed., 10.12.28).—Electrodes supplied with polyphase current and of large area are spaced in planes along the length of the furnace and are either not in direct contact with the furnace wall or are in contact with the periphery of the wall only at those points which are at a lower temperature than that of the electrically-heated zone. Treated material leaves the furnace through a lock which prevents escape of gas, and is surrounded by a cooling device.

J. S. G. THOMAS.

Semi-conductive coating for electric conductors, more particularly for high potentials. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 318,971, 12.9.29. Ger., 13.9.28).—Woven material saturated with a homogeneous semi-conducting liquid, especially tolyl phosphate, is applied to the conductor. [Stat. ref.]

J. S. G. THOMAS.

[Aluminium oxide] electric insulating bodies. SIEMENS & HALSKE A.-G. (B.P. 339,726, 15.10.29. Ger., 17.10.28).—Insulators formed of aluminium oxide are vitrified in an atmosphere free from reducing gases or vapours containing carbon, *e.g.*, in a 3:1 nitrogen-hydrogen mixture.

J. S. G. THOMAS.

Manufacture or impregnation of insulating materials. STANDARD TELEPHONES & CABLES, LTD., T. R. SCOTT, and T. N. RILEY (B.P. 339,990, 18.9.29 and 5.3.30).—Insulating material is dried, exhausted, and then treated with the vapour of one or more aromatic or paraffin hydrocarbons, *e.g.*, diphenyl or methylnaphthalene, which assist in expelling residual air.

J. S. G. THOMAS.

Manufacture of [arc-]welding electrodes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. M. WEED (B.P. 339,930, 8.7.30. U.S., 8.7.29).—A core of metal wires, washed in a caustic bath and etched in dilute hydrofluoric acid, is coated by dipping in a thin paste composed of calcium carbonate 1 pt., barium carbonate 1 pt., titanium oxide 2 pts., and sodium acetate 4 pts.

J. S. G. THOMAS.

Manufacture of [outer] electrodes of electric batteries. P. ROSENBERG (B.P. 339,799, 18.12.29. Ger., 28.11.29).—A phenol- or cresol-formaldehyde condensation product heated at 150–200° is pressed into, and seals liquid-tight, one end of an electrically conducting tubular casing.

J. S. G. THOMAS.

[Getter for] electric incandescence lamps. EGYE-SÜLT IZZÓLÁMPÁ ÉS VILLAMOSSÁGI RÉSZVÉNYTÁRSASÁG (B.P. 339,798, 18.12.29. Hung., 18.12.28).—A getter substance consisting, at least in part, of an inorganic water-abstracting agent which is gaseous at ordinary room temperature, more especially potassium silicofluoride, is claimed.

J. S. G. THOMAS.

Luminous electric-discharge tube. RAINBOW LIGHT, INC., Assees. of R. R. MACHLETT (B.P. 319,016, 14.9.29. U.S., 14.9.28).—The internal surface of the tube is cleansed by means of the vapour of an alkali metal before the tube is filled with a pure rare gas, *e.g.*, neon, helium, or argon, at a pressure exceeding 10 mm. Hg (preferably above 20 mm. Hg).

J. S. G. THOMAS.

Thermionic cathodes of vacuum electric tube devices. WESTINGHOUSE LAMP Co., Assees. of F. H. DRIGGS (B.P. 319,008, 9.9.29. U.S., 15.9.28).—An insulator composed of 90–99% of fritted or sintered rare-earth oxide, *e.g.*, thoria, and 10–1% of inert refractory material of lower m.p., *e.g.*, magnesium silicate ("isolantite"), is arranged between and makes contact with a heating element and a surrounding, hollow, thermionically-active metal body.

J. S. G. THOMAS.

[Target electrode support for] X-ray tubes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. K. KEARSLEY (B.P. 339,891, 26.2.30. U.S., 27.2.29).—An

X-ray target support or backing composed of an alloy of copper with 0.25–2% Al is claimed.

J. S. G. THOMAS.

[Portable] ozone-producing device. D. W. DAVIS and T. M. HARDY, Assrs. to NAT. LABORATORIES CORP. (U.S.P. 1,768,891, 1.7.30. Appl., 26.6.29).—A portable device suitable for domestic or public use is described.

W. J. WRIGHT.

Electro-osmotic purification of liquids. L. MELERSH-JACKSON. From SIEMENS-ELEKTRO-OSMOSE GES. M.B.H. (B.P. 339,673, 7.9.29).—Part of the liquid which has been treated in at least one central chamber is used for flushing the adjacent spaces which are separated from the central chamber by diaphragms.

J. S. G. THOMAS.

Copper oxide and like rectifiers for electric current. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of R. D. AMSDEN (B.P. 341,827, 17.5.30. U.S., 18.5.29).

Incandescence-cathode X-ray tubes. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 339,715, 7.10.29).

Hardening cast iron (B.P. 340,530).<sup>1</sup> Refined copper (B.P. 340,497.) Electrolytic iron (U.S.P. 1,769,605). Chromium-plating (B.P. 340,350).—See X. Silver salt emulsions (B.P. 339,769).—See XXI.

## XII.—FATS; OILS; WAXES.

Micropenetrometer; its application to the testing of fats. E. FREYER (Ind. Eng. Chem. [Anal.], 1930, 2, 423–424).—A micropenetrometer has been devised in which the molten fat is allowed to flow into cylindrical holes (6 mm. diam.) in a copper block which contains also larger holes for circulating water at constant temperature. After solidification the excess of fat is shaved off to a plane surface, and the penetrability is determined by measuring the depth to which a steel needle, weighing about 0.4 g., penetrates when dropped from a height of about 20 cm. As certain fats when cooled have a relatively hard crystalline core, the needle is dropped at about 1 mm. from the edge of the hole, and a series of measurements may thus be made with one specimen. Reproducible temperature-penetrability curves are readily obtainable, and duplicate readings agree to within 0.2 mm.

H. F. GILLBE.

Determination of fats and moisture in oil seeds and cottonseed cakes, the soluble part of siccatives, etc., by infusion with back-filtration. Y. I. LURIE (J. Chem. Ind., Russia, 1929, 6, 1530–1531).

CHEMICAL ABSTRACTS.

Conductometric determination of soap in technical baths. H. C. STUHLMANN and H. FABER (Chem. Fabr., 1930, 531–532).—The solution is titrated conductometrically with 0.5N-sulphuric acid; a sharp rise in conductivity takes place when all the fatty acid is precipitated. The end-point is taken as usual at the intersection of the two branches of the titration curve.

A. R. POWELL.

Spontaneous coloration of soap lyes. M. SERYAKOV (Masloboino Zhir. Delo, 1929, No. 7, 44–46).—Examination of a substance giving a red colour

which disappeared in presence of acid but returned on addition of alkali led to conflicting results; some of the data indicated the presence of tetrahydroxylinoleic acid.

CHEMICAL ABSTRACTS.

Oxidation of castor oil by potassium permanganate. Study of triazelain. G. SCHUSTER (J. Pharm. Chim., 1931, [viii], 13, 5–12).—Oxidation of castor oil (iodine value 83, saponif. value 183) by Hilditch's method gave 63.58% of the triglyceride of azelaic acid, which has also been prepared from epichlorohydrin and sodium azelate. Its properties are described. Neither stearic nor dihydroxystearic acid was identified in the oxidation products of the oil examined. The triglyceride can be separated from free azelaic acid as the insoluble magnesium salt.

E. H. SHARPLES.

Vegetable oils of the Union of S.S.R. VI. Oils of the *Cruciferae* in connexion with the climate of district of origin. S. IVANOV, A. P. SALTSHCHINKIN, and A. S. VOROBEJEV (Chem. Umschau, 1930, 37, 349–354; cf. B., 1930, 568).—The iodine value of oils from various genera of *Cruciferae* increases with more northerly origin of the seed, e.g., from 104 to 134 for the oils of *Thlaspi arvense* grown in Germany and in lat. 65° 10' N. In general, the differences are not so large as in the case of the drying oils. Fruits of *Crambe koktebelica*, N. Busch, *C. aspera*, MB., and *C. maritima*, L., respectively, yielded 15.5, 10.8, and 9.1% (27.1, 25, and 22.4% on the kernels) of pale odourless oils having acid value 5.4, 6.1, 13.2; saponif. value 183.6, 181.1, 179.9; iodine value 112.9, 110.2, 106.9; thiocyanogen value 79.6, 82.4, 80.0; butyrometric value 71, 70.5, 70.5. Thiocyanometric analysis gave the respective compositions: linoleic acid 38.42, 33.08, 31.04%; oleic acid 53.8, 63.44, 62.70%; saturated acids 7.78, 3.45, 7.96%. Oxidation (Hazura-Sayzeff) of the liquid fatty acids (12.24%) from the oil of *C. koktebelica* indicated the presence of oleic and linoleic acids and of an acid yielding a hydroxy-acid, m.p. 117°. Seeds of *Capsella bursa pastoris*, Moench., and of *Thlaspi arvense*, L., yielded, respectively, 35.5 and 32.35% of oils having  $d_{4}^{15}$  0.9222, 0.9124; butyrometric value —, 73/22°; acid value 2.89, 3.85; saponif. value 162.46, 180.4; iodine value 128.1, 103.6; thiocyanogen value —, 82.65. The same hydroxy-acid (m.p. 118) was found among the products of oxidation. The seeds of *Hesperis matronalis*, L., yielded 28.78% of a bitter drying oil rich in sulphur compounds and having acid value 3.8, saponif. value 180.3, Hehner value 96.3, iodine value 150, thiocyanogen value 115.2, insoluble hexabromides 10.8%,  $d_{4}^{15}$  0.9340, butyrometric value 86/25°. The action of myrosin on sinigrin (in *Brassica nigra*) is weaker at 4° than at 15°; at 15° the hydrolysis is almost complete in 1 hr.

E. LEWKOWITSCH.

"Periodine" values of fats and oils and their practical significance. S. JUSCHKEVITSCH (Masloboino Zhir. Delo, 1929, No. 3, 43–47).—"Periodine" values were determined as follows: linseed oil 238.0, hempseed oil 230.6–234.4, sunflower oil 204.6, olive oil 118.0, castor oil 162.0. The compositions of mixtures of oils having the same iodine value (e.g., olive, castor, almond, rape) were determined with an accuracy of

5%. The difference iodine value serves for the determination of the fatty acid composition.

## CHEMICAL ABSTRACTS.

**Sodium metasilicate.** VAIL.—See VII. **Emulsions for leather.** CLAFLIN.—See XV. **Insecticidal soap solutions.** VAN DER MEULEN and others.—See XVI. **Analysis of butter.** GUTHRIE etc. **Benzoic acid in margarine.** DINGEMANS.—See XIX.

See also A., Feb., 164, **Benzene dispersions of basic soaps of nickel and iron** (SOYENKOFF). 192, **Infusorial earth from Caucasus** (WEINBERGER). 198, **Dehydrogenation of ricinoleic acid** (BÖESEKEN and HOEVERS). 249, **Fatty acids of brains of ox, pig, and sheep** (BROWN and AULT). 260, **Isotonic soap for [medical] injections** (PICOU).

## PATENTS.

**Treatment of lard.** L. B. PARSONS, Assr. to CUDAHY PACKING Co. (U.S.P. 1,767,999, 24.6.30. Appl., 1.6.29).—Lard is deacidified by treatment with an excess of sodium bicarbonate (=2–3 times the content of free fatty acids) and 0.25–1% of water for 10–30 min., at temperatures between 60° and 94°; water is evaporated during the process and the dry soap formed is removed by filtration after the addition of about 0.4% of a filter aid.

E. LEWKOWITSCH.

**Fatty oil composition.** A. A. SOMERVILLE, Assr. to R. T. VANDERBILT Co., Inc. (U.S.P. 1,767,264, 24.6.30. Appl., 17.1.27. Cf. U.S.P. 1,742,791; B., 1930, 426).—Fatty oil compositions (with or without mineral oils) for lubrication, belt dressing, etc. are protected against rancidification by the addition of a small amount of a non-volatile compound of an aldehyde (e.g., aldol) with a nitrogenous base (e.g.,  $\alpha$ -naphthylamine).

E. LEWKOWITSCH.

**Manufacture of castor oil [medicinal] soap.** W. M. BILLING, Assr. to W. S. MERRELL Co. (U.S.P. 1,767,041, 24.6.20. Appl., 14.4.26).—A liquid aqueous solution containing 25–40% of castor oil soap, which may be readily diluted and does not attack glass, is prepared by liquefying a neutral castor oil soap by the addition of a small excess of castor oil fatty acids.

E. LEWKOWITSCH.

**Recovery of oil from blubber, meat, bones, and other parts of sea animals.** A. F. BURGESS. From C. O. JOHNSON (B.P. 339,305, 31.8.29).—A wet-rendering process is described in which the water containing the charge is kept boiling under pressure (e.g., 25–28 lb./in.<sup>2</sup>), by means of live steam; steam is continuously vented above the level of the liquid, and the products are kept under top pressure (e.g., 4–5 lb./in.<sup>2</sup>), while settling and drawing off the oil.

E. LEWKOWITSCH.

**Processing of animal matter.** E. T. MEAKIN (U.S.P. 1,766,031–3, 24.6.30. Appl., [A] 13.11.22, [B, C] 18.2.24).—(A) Continuously acting rendering plant is described; the comminuted material is cooked *in vacuo* while passing through a horizontal oven provided with propelling scraper arms and rotary inlet and outlet valves, interconnected so as to open alternately, and is conveyed thence by screws through a continuous cage-type press. (B) The material is immersed

and cooked at about 74° in deep grease (rendered from the same fatty material) in a vacuum cooker of the type (c) and pressed. Loss of nitrogen is avoided and the continuous process rapidly yields a high-quality fat and a nutritive residue suitable for human consumption (e.g., as fillers in sausages). (c) Apparatus for the process (b) consists of a cylindrical vacuum cooker of the type described in (A), inclined upwards from the inlet valve, in order that entering material may be immediately immersed in grease, and provided with a dam at the upper (outlet) end to keep the grease-bath at a constant level.

E. LEWKOWITSCH.

**Detergent.** B. F. CARVER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,345, 24.6.30. Appl., 21.5.23).—The detergent comprises the mixed alkaline or ammonium soaps of coconut oil (fatty acids or other fatty oil) and the sulphonates derived from acid-treated mineral oil sludge.

E. LEWKOWITSCH.

**Wetting etc. agents** (B.P. 337,737 and 337,774). **Basic product from oleic acid** (B.P. 317,325).—See III. **[Fats from] marine algæ** (B.P. 319,333).—See VII. **Solutions of drying agents** (B.P. 339,865 and 339,922).—See XIII.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Composition of the alcoholic fraction of turpentine.** V. KRESTINSKI, F. SOLODKI, and G. TOLSKI (J. Appl. Chem., Russia, 1930, 3, 691–698).—The alcoholic fractions of turpentine from *Pinus sylvestris* contain  $\alpha$ -pinene,  $\Delta^3$ -carene, *d*-camphor, probably  $\Delta^1$ -menthen-4-ol, a saturated alcohol, a sesquiterpene (m.p. 82–83°, probably cadinene), and other components.

CHEMICAL ABSTRACTS.

**Composition of turpentine from *Pinus sylvestris*.** V. KRESTINSKI, A. LIVEROVSKI, and V. MALMBERG (J. Appl. Chem., Russia, 1930, 3, 533–540).—Turpentine distilled in the presence of alkalis had  $d_{20}^{20}$  0.8660,  $n_D^{20}$  +13.64°,  $n_D^{20}$  1.47163, and contained only  $\alpha$ -pinene and (*l*)- $\Delta^3$ -carene. The residue, washed with alcohol and crystallised from methyl-alcoholic chloroform, had m.p. 51° and empirical formula  $C_{10}H_{20}$ . Another crystalline substance had m.p. 90°. CHEMICAL ABSTRACTS.

**Lævorotatory turpentine from *Pinus sylvestris*.** V. KRESTINSKI and L. BASHENOVA-KOSLOVSKA (J. Appl. Chem., Russia, 1930, 3, 681–689).—The turpentine, of which all the components were lævorotatory, contained  $\alpha$ -pinene 69, camphene 5, carene fraction 14.5, phellandrene 1.5, alcohols and sesquiterpenes 10%.

CHEMICAL ABSTRACTS.

**Manufacture of terpinol from Russian turpentine.** A. K. SHUMEIKO (J. Appl. Chem., Russia, 1930, 3, 541–553).—The use of 65% sulphuric acid (1 : 1.5–4.0 of turpentine) is recommended; the maximum permissible temperature is 5°. CHEMICAL ABSTRACTS.

**["Chalking" of paint (films).]** H. WAGNER (Farben-Ztg., 1931, 36, 738).—A reply to criticisms by van Hoek, mainly polemical. The weathering of the vehicle has not been ignored, but is bound up with the question of reaction between pigment and vehicle. It is also stressed that variation of degree of wetting of different pigments by one and the same

vehicle is a different problem from that of the behaviour of one pigment when wetted by vehicles of various surface tension etc. S. S. WOOLF.

**Comparison of some light sources for accelerated weathering tests [on paints etc].** G. G. SWARD and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1931, Circ. 375, 53—58).—Paints, varnishes, nitrocellulose lacquers, and auto-top enamels were submitted to accelerated weathering cycles, using as light sources enclosed and "flaming" types of carbon arcs and mercury arcs. Details of products tested, light sources used, and relative costs thereof are given, and the results of the exposures are analysed. The types of deterioration induced by the various lamps are indicated. S. S. WOOLF.

**Field tests on quick-drying house paints.** H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1931, Circ. 374, 32—52).—An account is given of a series of exposure tests recently undertaken (B., 1927, 851) on a range of paints comprising various pigment mixtures in vehicles containing linseed oil, bodied oils, "lead tungate," synthetic resin varnishes, etc. Compositions, spreading rate, brushing, and general properties are detailed. S. S. WOOLF.

**Identification of aluminium and magnesium in printing inks.** L. M. LARSEN (Ind. Eng. Chem. [Anal.], 1930, 2, 416—417).—Four test strips,  $\frac{3}{8}$  in.  $\times$   $1\frac{1}{2}$  in., prepared by printing on alum-free paper with the ink to which has been added a little viscous linseed varnish and cobalt drier, are boiled with about 3 c.c. of 2*N*-hydrochloric acid for 1 min., and to the acid extract is added an equal volume of a solution containing 250 g. of ammonium acetate, 10 g. of acetic acid, and 1 g. of aurintricarboxylic acid per litre; after boiling and allowing to cool, a red flocculent precipitate, unchanged by shaking in the cold with an alkaline ammonium carbonate solution, indicates that aluminium is present in the ink. Iron should be absent. In the detection of magnesium, 4 strips are boiled with 5% acetic acid for 2 min., and to the extract are added an equal volume of 5% sodium hydroxide solution and 1 drop of a 0.025% benzopurpurine 4B solution per c.c. of solution; a rose-coloured precipitate, formed within 2—5 min. in the cold, indicates the presence of magnesium. If the pigment produces a coloration which masks the test, it should be destroyed by addition of a few drops of a 2% chloramine-T solution at the b.p. H. F. GILLBE.

**Lacquers.** A. W. VAN HEUCKEROTH and J. R. STEWART, JUN. (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1931, Circ. 371, 1—20).—A series of brief notes is presented on the use of vegetable oils as plasticisers for nitrocellulose lacquers, examination of various chemical plasticisers (phenyl phthalate offering possibilities in comparison with the commonly used butyl ester and tolyl phosphate), the final examination of a range of lacquers containing various synthetic resins exposed in 1930 (cf. B., 1930, 1119), a new apparatus for drying nitrocellulose for laboratory tests, physical properties of films produced from nitrocellulose of different viscosities, a chart correlating composition and viscosity of mixtures of nitrocellulose of different

viscosities, and a comparison of various "viscosity cups" in use in industry. S. S. WOOLF.

**Nitrocellulose lacquers.** H. WOLFF and B. ROSEN (Farben-Ztg., 1931, 36, 736—737).—The use of natural or "run" kauri copal in nitrocellulose lacquers gives rise to inequalities (not always apparent) in the film, owing to the gelling tendency of the resin. A sample of a proprietary "extracted kauri" was examined for suitability as a substitute for ester gum in two lacquer formulations and was found to be superior to the ester gum in hardness, abrasion-resistance, adhesion, elasticity, etc. In a further series of tests, using an alcohol-soluble nitrocellulose, the "extracted kauri" was compared with shellac and manila, and was found to be less resistant to abrasion but more elastic than shellac, which it otherwise resembled in behaviour, both being superior to manila. Attention is drawn to the irregular trend of the physical properties of different nitrocellulose lacquers with time. S. S. WOOLF.

**Kauri gum in nitrocellulose lacquer.** L. S. SPACKMAN (New Zealand J. Sci. Tech., 1930, 12, 65—93).—Lacquers which compare favourably in colour, finish, and hardness with those containing best ester gums may be prepared from kauri gum. No single solvent is satisfactory and various systems have been studied and compared with those using denatured absolute alcohol, which it is neither necessary nor desirable to use in large quantities. Ethylene glycol monoethyl ether mixed with *n*-butyl alcohol forms the best solvent, and this may be considerably diluted with toluene or benzene; petroleum hydrocarbons may be used to a more limited extent. T. McLACHLAN.

**Photomicrographs of some dull and crystalline [novelty] finishes [with lacquers and varnishes].** H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1931, Circ. 373, 28—31).—Photomicrographs of matt and crystalline lacquer and varnish films are given, with a brief note on the strength of such films in respect of modifying agents and solvents used. S. S. WOOLF.

**Exposure tests [on varnishes].** H. A. GARDNER, G. G. SWARD, and S. LEVY (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1931, Circ. 372, 21—27).—An account is given of a series of exposures on tung oil and tung oil-linseed oil varnishes containing various natural and synthetic resins. No specific results or evaluations are given, as the tests are not considered satisfactory, but general conclusions on the carrying out, inspection, and recording of exposures of oil varnishes etc. are drawn, and photomicrographs illustrating types of failure are included. S. S. WOOLF.

**Determination of acid value of copals.** Z. LEPPERT (Przemysl Chem., 1931, 15, 1—5).—Powdered copal (1 g.) is dissolved under a reflux condenser in 20 g. of boiling terpeneol, the hot solution is diluted with 20 c.c. of methyl alcohol, and the solution is titrated, using 0.1*N*-potassium hydroxide. R. TRUSZKOWSKI.

**Lead peroxide and red lead.** BRUHNS.—See VII. Soluble part of siccatives. LURIE.—See XII.

## PATENTS.

**Painting processes.** E. FRENKEL (H. FRENKEL) (B.P. 339,789, 9.12.29. Ger., 10.12.28).—Paint compositions containing as binding agents fatty oils that have been rendered viscous by treatment with air or gases containing ozone are claimed. Such paints gel throughout from the inside, and permit subsequent coatings containing the same or other vehicles to be applied while the first coat is still wet. S. S. WOOLF.

**Production of perfectly homogeneous solutions of drying agents.** I. G. FARBENIND. A.-G. (B.P. 339,865 and Addn. B.P. 339,922, [A] 4.2.30, [B] 5.5.30. Ger., [A] 22.2.29, [B] 14.6.29).—(A) Solutions of naphthenates of heavy metals and/or alkaline earths (cf. "Soligen driers") in fatty acids of drying or semi-drying oils, *e.g.*, of linseed, tung, soya bean, poppyseed, are claimed. (B) The fatty acids used as solvents are partly replaced by the fatty oils themselves. S. S. WOOLF.

**Production of [anti-fouling] coating materials.** N. J. SHIELDS (B.P. 339,128, 17.12.29).—Coating materials containing 25–50% of their weight of (preferably yellow) mercuric oxide, together with iron oxide, zinc oxide, and lead pigments if desired, suspended in a suitable vehicle, *e.g.*, an emulsion of turpentine with a wood alcohol solution of shellac, or a mixture of linseed oil, turpentine, and brown japan, are claimed. S. S. WOOLF.

**Pigment having insecticidal and fungicidal properties and its manufacture.** E. A. TAYLOR, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,766,412, 24.6.30. Appl., 5.8.26).—An insoluble copper salt, *e.g.*, copper chromate, is precipitated on a finely-divided, insoluble, arsenical insecticide, *e.g.*, calcium arsenate, as substratum. S. S. WOOLF.

**[Heat-insulating] coating composition.** H. J. HAIN, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,768,447, 24.6.30. Appl., 9.6.27).—Mixtures of mineral pigments, *e.g.*, whiting, with linseed meal in smaller amount and water are claimed. S. S. WOOLF.

**Manufacture of intaglio printing inks.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,733, 23.10.29).—Such inks comprise cellulose esters soluble in ethyl alcohol, *e.g.*, nitrocellulose containing 10.5–11.5% N, colouring matter, and solvent mixtures which contain at least 70% of ethyl alcohol but are free from aliphatic homologues of 1:4-dioxan. S. S. WOOLF.

**Manufacture of homogeneous masses and solutions applicable as lacquers.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 339,271, 3.9.29. Cf. B.P. 318,549; B., 1930, 1120).—Condensation products of aldehydes (or agents yielding them) with phenol derivatives, *e.g.*, salicylic acid, phenol ethers, phenoxy-fatty acids, are combined with vinyl ester polymerides by fusion together or mixing solutions of the components. S. S. WOOLF.

**Production of [cellulose] varnishes.** INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 312,923, 30.5.29. Ger., 1.6.28).—Homogenising media consisting of benzene derivatives which are good solvents for "glyptal" resins, *e.g.*, nitro-

benzene, pyridine, benzaldehyde, are added to cellulose varnishes containing condensation products of the "glyptal" type, a suitable solvent for the homogenising means, *e.g.*, "ethyl" glycol, and, if desired, plasticisers. S. S. WOOLF.

**Spray-drying of cellulose acetate solutions.** C. J. STAUD, Assr. to EASTMAN KODAK Co. (U.S.P. 1,762,937, 10.6.30. Appl., 2.3.28).—A solution containing cellulose acetate acetic acid, and a highly volatile organic liquid, *e.g.*, ethylene dichloride, is spray dried by atomising it into a desiccating gaseous vehicle which rapidly takes up the vapours of the acetic acid etc., thereby disrupting the particles and producing a rough-surfaced powder highly permeable to liquids. S. S. WOOLF.

**Producing resins [from cracked petroleum distillates].** J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,766,927, 24.6.30. Appl., 3.1.28. Renewed 21.12.29).—A mixture of polymeric hydrocarbons, *e.g.*, cracked petroleum distillates, is heated with phenolic substances, *e.g.*, wood- or coal-tar acids, to 150° in the presence of an oxidising gas, *e.g.*, air, ozone, or oxides of chlorine, until the desired viscosity is attained. S. S. WOOLF.

**Manufacture of [resinous] condensation products from diolefines.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 338,152, 14.5.29).—Resinous, non-rubber-like products are obtained by treating diolefines with substantial quantities (*e.g.*, 1–5 pts.) of an oxygenated inorganic acid, preferably of sulphur or phosphorus, or an organic derivative thereof, such as benzenesulphonic acid. C. HOLLINS.

**Manufacture of condensation products from cyclic hydrocarbons [and diolefines].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 338,109, 9.5.29).  $\alpha$ -Butadiene etc. is condensed in presence of aluminium chloride etc. with cyclic hydrocarbons having at least one aromatic nucleus and no olefinic side-chains, or with alkyl, halogeno-, nitro-, alkoxy-, or carbalkoxy-derivatives of these, to give thermo-hardening bases for lacquers. Examples are:  $\alpha$ -butadiene passed into mixtures of aluminium chloride with tetrahydronaphthalene, benzene, *m*-xylene, chlorobenzene, or nitrobenzene. C. HOLLINS.

**Solid resinous condensation product.** W. O. HERRMANN and H. DEUTSCH, Assrs. to CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,767,759, 24.6.30. Appl., 4.4.21).—Alkaline substances, *e.g.*, hydroxides, carbonates, sulphides, are added at above 30° to aliphatic aldehydes with more than 1 carbon atom, *e.g.*, metaldehyde, or their liquid intermediate condensation products, *e.g.*, aldol, the reaction being continued until practically complete conversion into a solid resin, insoluble in alkaline lye, has ensued. The product is treated with mineral acid and the resin is precipitated by water. S. S. WOOLF.

**Manufacture of condensation products containing sulphur.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 338,604, 24.8.29).—Polyhydric alcohol-polybasic acid oily to resinous products containing unsaturated acids, especially drying oil acids, are heated below 200°



with less than 5% of sulphur to give varnish bases soluble in benzene, petroleum, butyl acetate, oils, etc.

C. HOLLINS.

**Production of condensation products of urea and formaldehyde.** S. GOLDSCHMIDT and R. MAYRHOFER (B.P. 316,144, 22.7.29. Ger., 23.7.28).—The liquid or viscous condensation product obtained from phthalic anhydride and polyhydric alcohols is added to an aqueous mixture of urea and formaldehyde prior to, during, or after formation of a condensation product by heating in the presence of 2*N*-sulphuric acid.

S. S. WOOLF.

**Manufacture of condensation products of formaldehyde and urea.** KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 339,601, 10.6.29. Austr., 11.5.29).—Urea (1 mol.) is condensed with formaldehyde (less than 1.4 mols.) at temperatures not above 70°, and a condensation product of urea (1 mol.) with formaldehyde (at least 2 mols.), prepared at a higher temperature in the presence of an acid, is gradually admixed in such quantity as will give a final composition of 1 mol. of urea to 1.4—1.8 mols. of formaldehyde. The formation of undesirable by-products is thus avoided. S. S. WOOLF.

**Manufacture of artificial resins.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 338,937, 29.7.29).—Urea and formaldehyde, a methylol compound of a urea, the anhydride thereof, or mixtures of such initial materials are condensed in the presence of halogeno-aryl-substituted aliphatic alcohols, *e.g.*, mono- or dibromobenzyl alcohols, ethylene glycol monobromophenyl ether, preferably in the presence of an acid catalyst, *e.g.*, acetic or hydrochloric acid, potassium tetroxalate, urea nitrate, or buffer mixtures furnishing a constant *p<sub>H</sub>* value (*e.g.*, 4—6.5). The reaction may be further accelerated by heating to 80—120°. The condensation products are freed from the solvents in which they have been prepared by distillation, precipitation, etc. The resins produced are combustible with difficulty or incombustible. S. S. WOOLF.

**Manufacture of synthetic resins.** E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,767,696, 24.6.30. Appl., 14.8.26).—Resorcinol (1 mol.) is condensed with paraformaldehyde ( $\frac{1}{2}$  mol. or less) in the absence of a catalyst at 100—150° until the desired viscosity is attained, and the mass is cooled below 100° before the reaction product becomes infusible. Additional paraformaldehyde may be admixed as hardening agent. The product is a fusible, water-insoluble resin. S. S. WOOLF.

**Coating of [metal] surfaces with artificial resin.** "HEROLD" A.-G. (B.P. 338,983, 31.8.29. Ger., 9.3.29).—Metal surfaces, particularly pipes, are heated to the m.p. of a fusible artificial resin and the latter is applied as powder, in the absence of solvent. Alternatively, the resin may be applied in suspension in a non-solvent, *e.g.*, water or heavy benzol, or as a briquette which is rubbed over the surface. The surface coated with the unhardened resin is treated superficially with a solvent or solvent vapour during the subsequent hardening of the resin. A filler may be applied simultaneously with, although separately from, the resin. Freedom from blistering and rapid hardening are claimed.

S. S. WOOLF.

**Manufacture of [lace-artificial resin] decorative moulded objects.** A. V. KELLER (B.P. 338,915, 28.8.29).

**Modified resins etc.** (B.P. 337,732—6 etc.).—See III. [Pigment from] anthraquinone derivatives (B.P. 338,412).—See IV. Hard-paper material (B.P. 318,490).—See V. [Pigments from] marine algæ (B.P. 319,333).—See VII. Coating for rubber (U.S.P. 1,761,814).—See XIV. Artificial horn (B.P. 313,455).—See XV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Consistency of the particles in balata latex.** New developments in the use of the micromanipulator. E. A. HAUSER (Kautschuk, 1931, 7, 2—3; cf. B., 1925, 514).—With the aid of a new dark-field condenser for use with the micromanipulator, it is found that the particles in balata latex are of two distinct types: the larger particles (diam. 0.5—3.5  $\mu$ ) are plastic and adhesive and consist of balata resin; the smaller, considerably below 0.5  $\mu$ , have a viscid interior, are plastic, and almost elastic at the surface, and constitute balata hydrocarbon. Coagulants barely affect the consistency of particles of the former type, but the latter coalesce easily and solidify. An instrument is described for the automatic production of glass needles of the necessary fineness for micromanipulator work. D. F. TWISS.

**Protective action of some anti-oxidants.** F. KIRCHHOF (Kautschuk, 1931, 7, 7—11).—Some nitrogenous anti-oxidants such as aldol- $\alpha$ -naphthylamine in alcoholic solution give intense colorations with certain metallic chlorides, *e.g.*, ferric or cupric chloride; the coloured solutions probably contain the metal chloride attached at the tertiary nitrogen atom or atoms. These products are considered to be of analogous structure to hæmin and, like this substance, they act as marked oxygen carriers, their catalytic effect likewise being suppressed by hydrocyanic acid. It is possible that the gradual discoloration of rubber goods containing anti-oxidants is due to the formation of such coloured products by the action of traces of iron. D. F. TWISS.

**Technical [rubber] mixtures and their electrical properties.** F. JACOBS (Kautschuk, 1931, 7, 4—7).—Investigation of the influence of various common mineral pigments on the dielectric resistance of rubber showed that ochre and lampblack were definitely unfavourable. Of several cheap filling materials, kieselguhr and marble powder proved the best; slate powder was less good, whilst chalk, precipitated calcium carbonate, and clay were poor. D. F. TWISS.

**Laboratory evaluation of [rubber] flex-cracking resistance.** L. V. COOPER (Ind. Eng. Chem. [Anal.], 1930, 2, 391—394).—Owing to the low compressibility of rubber, cracking during flexure may be regarded as a result of elongation, and a machine based on this principle has been evolved for evaluating the resistance to cracking under repeated flexure. One end of a dumbbell-shaped test piece is attached to a beam which makes 400 oscillations per min., the maximum elongation of the test piece being 100%; between oscillations the piece returns to zero stress. Average tyre-tread

stock exhibits cracks in about 8 hrs., and the machine reproduces closely actual running conditions. Neither the abrasion-resistance nor the rigidity of the stock affects the determination, and the machine, being compact, may be operated, if desired, in direct sunlight or in an enclosure at any given temperature. The detrimental influence of excessive pigmentation, and the advantage of adding reclaimed rubber to a mix in preference to clay, are well demonstrated. Diphenylamine greatly increases the resistance of the rubber to cracking.

H. F. GILLBE.

See also A., Feb., 150, **X-Ray investigations on rubber** (OTT). 228, **Isoprene and caoutchouc** (STAUDINGER and others).

#### PATENTS.

**Treatment of rubber latex.** E. HOPKINSON and M. C. TEAGUE, ASSRS. to AMER. RUBBER CO. (U.S.P. 1,765,015, 17.6.30. Appl., 18.8.23).—Rubber latex containing a protective colloid and a vulcanising agent is made into an uncoagulated mixture with cellulosic material, such as straw or wood flour. Mineral oil or solvent naphtha may also be present to increase the cohesiveness of the wet mass. The mixture is dried with retention of the serum solids and vulcanised. The products are porous, have a low sp. gr., and are suitable for use as structural material, *e.g.*, for covering walls or floors.

D. F. TWISS.

**Treatment of rubber latex.** W. B. WESCOTT, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,762,494, 10.6.30. Appl., 8.7.27).—By mixing rubber latex with hæmoglobin (*e.g.*, 15% on the rubber content), and spray-drying in air of controlled temperature and humidity, it is possible to obtain a non-tacky rubber dust which is readily miscible with dry compounding ingredients; the product is also re-dispersible with water.

D. F. TWISS.

**Purification of dispersions of vegetable resins [such as rubber latex].** K.D.P., LTD. (B.P. 338,766, 17.12.29. Ger., 20.12.28).—Dispersions such as rubber latex are heated, with concurrent evaporation, until the substances impeding the separation of the vegetable resin from the serum have become inoperative; the dispersion is then submitted to the usual methods of purification, *e.g.*, by centrifuging, with or without previous dilution.

D. F. TWISS.

**Composition of matter. [Rubber dispersion.]** W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,762,194, 10.6.30. Appl., 18.5.25).—If a colloid substance, *e.g.*, casein (15 pts.) is worked into crude or reclaimed rubber (100 pts.), and the mixture is then dispersed at 120–160° in a "non-solvent" medium, *e.g.*, a molten mixture of rosin (60 pts.), montan wax (20 pts.), "mineral rubber" (10 pts.), and gum copal (10 pts.), a thermoplastic product is obtained which has almost the same m.p. as that of the mixture without the rubber, but has greatly superior adhesive and cohesive properties. By the addition of sulphur to the product and heating, the characteristics of the mass can be further modified.

D. F. TWISS.

**Rubber-containing compositions.** N. SWINDIN, and NORDAC, LTD. (B.P. 339,002, 5.9.29).—An aqueous liquid is dispersed into rubber by kneading, the rubber

being warmed or softened by a solvent; the addition of a hard, porous, inert powder such as coke dust aids distribution of the aqueous liquid, and compounding ingredients such as inert fillers and sulphur may also be introduced into the dispersion. A water-setting cement, such as Portland cement, is then incorporated, and causes the composition to set gradually. The addition of "cumar" resin or gilsonite counteracts any retarding effect of rubber solvent on this setting operation. Such rubber cements are suitable for application as surfacing material for floors or roads, or as acid-resisting coatings.

D. F. TWISS.

**Manufacture of rubber emulsion.** S. E. SHEPPARD and L. W. EBERLIN, ASSR. to AMER. ANODE, INC. (U.S.P. 1,769,717, 1.7.30. Appl., 6.1.23).—A solution of rubber, with or without compounding ingredients, is emulsified into water containing an emulsifying agent such as a soap or colloidal clay. The emulsification is effected by agitation, and is facilitated by previously reducing the viscosity of the rubber solution, *e.g.*, by heat or exposure to ultra-violet radiation.

D. F. TWISS.

**Preparation of age-resisting rubber composition.** H. W. ELLEY (U.S.P. 1,763,615, 10.6.30. Appl., 29.5.29).—Secondary aromatic amines, particularly those derived from  $\alpha$ -naphthylamine by replacement of hydrogen by an alkyl-substituted benzene ring, are used as anti-oxidants. The examples given are phenyl- $\alpha$ -naphthylamine and diphenylamine.

D. F. TWISS.

**Rubber composition and method of preserving rubber.** H. W. ELLEY, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,763,293, 10.6.30. Appl., 7.12.27).—Ageing of rubber is improved by incorporation or surface-application of *m*-tolylenediamine.

D. F. TWISS.

**Preservation of rubber and age-resisting rubber products produced thereby.** B. F. GOODRICH Co., ASSEES. of P. C. JONES (B.P. 339,834, 13.1.30. U.S., 21.1.29).—For preservative purposes there is incorporated in rubber a tetra-substituted hydrazine such as tetraphenylhydrazine or its alkyl or alkoxy-derivatives.

D. F. TWISS.

**Coating of rubber with cellulosic derivatives.** H. A. BRUSON, ASSR. to RESINOUS PRODUCTS & CHEM. Co. (U.S.P. 1,761,814, 3.6.30. Appl., 5.3.29).—The application of solutions of nitrocellulose or other cellulose derivatives, to which has been added a resin formed by the condensation of glycerol with a long-chained dibasic aliphatic acid, such as sebacic acid, is claimed.

D. F. TWISS.

**Caoutchouc mixture or rubber-leather imitation.** O. G. BOHLIN (B.P. 340,305, 25.9.29).—Rubber is mixed with finely-powdered vegetable substances, such as straw meal in place of all or part of the usual inorganic filling materials, the proportion added being sufficiently large to enable the mass to retain its shape during vulcanisation.

D. F. TWISS.

**Manufacture of conversion products of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD., and E. B. ROBINSON (B.P. 339,398, 30.9.29).—Rubber which has been rendered liquid by excessive mastication followed by heating, *e.g.*, in carbon dioxide at 250–270° for 24 hrs., is

treated with an isomerising agent, *e.g.*, sulphuric acid or a sulphonic acid, with or without the addition of phenolic substances. The isomerising process, which may occupy 1 hr. or so at 150°, yields a fairly hard black mass which becomes soft at 100°. D. F. TWISS.

**Manufacture of rubber-like masses and articles therefrom.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,255, 30.8.29).—By polymerising a mixture of an aliphatic conjugated diolefin such as butadiene or isoprene (2 pts.) with styrene (up to 1 pt.), either constituent, if desired, being already in a partly polymerised condition, an intimately mixed polymerisate is obtained. The new products are of technical value and those of rubber-like character can be compounded with carbon black and vulcanised. D. F. TWISS.

**Manufacture of [rubber] tread stock.** W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,767,234, 24.6.30. Appl., 28.3.24).—Partly vulcanised rubber-fibre particles, *e.g.*, the comminuted product of old tyre carcasses, is mixed with a relatively small proportion of alkali-preserved latex, the latex rubber constituting, for example, 10% of the final dry mass. Vulcanising agents also may be incorporated. The uncoagulated mixture is drained and dried, the resulting slabs being then cut into shape for making moulded articles. D. F. TWISS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., E. A. MURPHY, A. NIVEN, and D. F. TWISS (B.P. 338,975, 30.8.29).—Crumb-like compositions are obtained by the coagulation of rubber latex by precipitating therein one or more compounding ingredients in relatively large proportion. These ingredients are formed by the interaction of one or more water-soluble reagents having normally no coagulant effect (such as carbonates, silicates, sulphates, or sulphides of the alkali metals) with one or more water-soluble substances an ion of which may incidentally have coagulating influence (such as soluble salts of magnesium, aluminium, barium, or zinc). The mixture is stirred during the compounding process. On account of its crumb-like character the wet product can be washed easily for the removal of soluble reaction products before being dried and applied to further manufacturing processes. D. F. TWISS.

**Manufacture of goods of or containing rubber or similar resins.** DUNLOP RUBBER CO., LTD., W. G. GORHAM, and E. A. MURPHY (B.P. 339,676, 13.9.29).—Rubber sheet, made direct from compounded latex, *e.g.*, by spreading, is cut in known manner, the resulting strips or thread being vulcanised subsequently. This procedure gives improved resistance to chafing. D. F. TWISS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 339,974, 18.6.29).—Cellulosic material such as fabric or thick paper which has been previously shaped is coated, by dipping, with a frothed and, if desired, compounded aqueous dispersion of rubber or similar material. The sponge-coated material is dried and vulcanised. D. F. TWISS.

**Correcting for heat loss due to condensation in treating rubber articles.** INDUSTRIAL PROCESS CORP.

(B.P. 339,303, 26.8.29. U.S., 26.11.28. Cf. B.P. 305,577; B., 1930, 645).—In the known method of vulcanising rubber articles, using as heating medium a mixture of an inert gas and a vapour, *e.g.*, carbon dioxide and steam, in a container such as an "air bag," the pressure of the gas is corrected (by calculation from knowledge of the sp. heat of the material to be heated, the desired rise in temperature, etc.) so as to allow for the reduction in the effective capacity of the container due to condensation of steam. [Stat. ref.] D. F. TWISS.

**Aldol condensation product and vulcanisation of caoutchouc therewith.** L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,754 865, 15.4.30. Appl., 10.11.24).—Vulcanisation is accelerated by the incorporation of the reaction product of an aldol, *e.g.*, acetaldol, with an aromatic amine containing a single benzene nucleus, *e.g.*, aniline or benzylamine. D. F. TWISS.

**Vulcanisation of rubber.** D. H. POWERS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,763,325, 10.6.30. Appl., 31.8.26).—Vulcanisation is accelerated by benzthiazyl monosulphide; this is conveniently prepared by the action of cyanogen chloride on an aqueous solution of sodium mercaptobenzthiazole. D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,762,531, 10.6.30. Appl., 5.1.28).—Vulcanisation is accelerated by the reaction product of a straight-chain aldehyde, *e.g.*, butaldehyde, with a dialkylenethiuram disulphide, such as diallylthiuram disulphide. D. F. TWISS.

**Vulcanisation of rubber.** H. WADE. From RUBBER SERVICE LABS. CO. (B.P. 339,352, 7.9.29).—Rubber is vulcanised with the aid of an accelerator prepared by the interaction of an aldehyde-amine, *e.g.*, heptaldehyde-aniline or butylideneisoamylamine, with a substituted unsaturated aldehyde such as  $\alpha$ -ethyl- $\beta$ -propylacetaldehyde preferably in equimolecular proportions and in the presence of an organic acid such as butyric acid. D. F. TWISS.

**Vulcanisation of rubber or rubber compositions.** DEUTS. HYDRIERWERKE A.-G. (B.P. 339,826, 6.1.30. Ger., 8.1.29).—Vulcanisation is effected in the presence of one or more esters of cyclic alcohols, *e.g.*, cyclohexyl benzoate or bornyl adipate. D. F. TWISS.

**Manufacture of vulcanised rubber.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 340,083, 1.11.29).—Vulcanisation is effected with the aid of a reaction product of a halogen derivative of benzene, *e.g.*, dinitrochlorobenzene, and a dithiocarbamate, *e.g.*, potassium piperidine-1-carbothionolate. D. F. TWISS.

**Manufacture of hollow bodies consisting of vulcanised rubber.** W. GENTH, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,765,666, 24.6.30. Appl., 14.3.29. Ger., 21.3.28).—Metaldehyde is used as an aerating or inflating agent in the manufacture of hollow or sponge-like articles of vulcanised rubber. D. F. TWISS.

**Production of rubber.** H. BECKMANN (B.P. 338,698, 25.10.29. Ger., 26.10.28. Addn. to B.P. 240,430; B., 1925, 1000).—In the production of micro-porous rubber, greater uniformity and control are ensured by adding

to the latex a substance capable of forming a hydrophilic gel, *e.g.*, silicic acid, aluminium hydroxide, or an albuminoid substance; on treatment of the mixture with a coagulant both the rubber and the additional substance are coagulated. The product is then vulcanised.

D. F. TWISS.

**Production of rubber articles [for aeroplanes etc.].** DUNLOP RUBBER Co., LTD., and E. W. MADGE (B.P. 340,024, 19.9.29).—Light "board," suitable, *e.g.*, for use in aeroplane construction, comprises a layer or layers of sponge ebonite prepared from frothed compounded latex (cf. B.P. 332,525; B., 1930, 1040), between two or more layers of material such as metal, fabric, plywood, or ebonite.

D. F. TWISS.

**Storage and packing of sheet rubber and the like.** GOODYEAR TIRE & RUBBER Co., Assees. of P. BEEBE (B.P. 339,730, 21.10.29. U.S., 26.2.29).—Sheets of unvulcanised rubber or rubberised fabric are stored with intervening plies of "liner" fabric which has been treated with a solution of soap, *e.g.*, coconut-oil soap, and dried.

D. F. TWISS.

**Manufacture of waterproof cement for securing rubber to metal.** GOODYEAR TIRE & RUBBER Co., Assees. of C. M. CARSON (B.P. 339,421, 21.10.29. U.S., 23.2.29).—Rubber is attached to metal by first coating the latter with a cement containing latex, hæmoglobin, vulcanising agents, and a tanning agent, such as formaldehyde or potassium dichromate; the cement coating is preferably dried and then baked at 120° for at least 1½ hrs. before applying the rubber stock and vulcanising. Unions so produced are stronger and more resistant to moisture than those produced with latex-hæmoglobin cements containing no tanning agent.

D. F. TWISS.

**Ebonite composition and its production.** H. GRAY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,769,506, 1.7.30. Appl., 2.4.23).—Comminuted ebonite is mixed with a substantial proportion of a rubber softener, such as pontianak resin or factice, and heated, *e.g.*, at 149–177°, to produce a homogeneous mass. The product can be moulded at a lower temperature than previously; it also is miscible with rubber and can be dissolved in rubber solvents. A similar product may be obtained by adding the softener to a raw ebonite mixture before vulcanisation.

D. F. TWISS.

**Modified isocolloids** (B.P. 337,732 etc.).—See III.

## XV.—LEATHER; GLUE.

**Emulsions and suspensions in the leather industry.** A. A. CLAFLIN (J. Amer. Leather Chem. Assoc., 1930, 25, 544–550).—Colloid mills are suggested for the production of such emulsions etc. The stability and creaming of emulsions is discussed and for satisfactory results the grinding of equal quantities of oil and water in the mill is recommended, the product being diluted as desired. Dilute emulsions with no tendency to "cream" can readily be obtained by the use of colloids, *e.g.*, gum, casein, gelatin, as emulsifying agent. "Creaming," with no actual separation of the oil, is not objectionable since the original emulsion is restored by stirring. In the manufacture of suspensions, a stabiliser (soap) is necessary to prevent the reaggregation of the solid disperse phase.

D. WOODROFFE.

**Influence of oils on the water absorption of leather.** G. POVARNIN and P. KOPELOVICH (Vestnik, 1928, No. 9, 142).—Within certain limits, depending on the nature of the oil used, the absorption of water decreases with increase in oil content. With cod oil, anomalous results are sometimes obtained.

CHEMICAL ABSTRACTS.

**Influence of some factors on the elastic properties of chrome-tanned leathers.** G. POVARNIN (Vestnik, 1928, No. 2–3, 80).—Chrome-tanning lowers the tensile strength of the hide. Increase of concentration of chrome liquors increases the elastic properties of the leather, but decreases the tensile strength. Other conditions being the same, a basicity of 55° gives a leather of maximum elasticity and elongation, but minimal tensile strength. The absorption of chromic oxide increases with basicity, but only slightly with increase in concentration of chromium.

CHEMICAL ABSTRACTS.

**Salt stain in the manufacture of leather.** P. WHITE and F. O. CAUGHLEY (New Zealand J. Sci. Tech., 1930, 12, 108–113).—The conclusions of other workers that "pink stain" is caused by halophilic bacteria is confirmed. Sodium fluoride or silicofluoride (0.5% on the wt. of salt used) appears to inhibit the growth of the bacteria, but causes shrinkage of the hides. Long-period tests are being carried out with these preservatives.

T. MCLACHLAN.

**Determination of total sulphuric acid in leather.** K. A. BUSCHMAN (Ukraine Chem. J., 1930, 5, [Tech.], 113–116).—The leather (4–5 g.) is dissolved in a boiling solution of 10 g. of sodium hydroxide in 25 c.c. of water, the solution is cooled and diluted to 100 c.c., and chlorine is passed for 15 min., after which sulphuric acid is determined as barium sulphate.

R. TRUSZKOWSKI.

**Drying equipment for badan root.** I. ENTIN (Vestnik, 1928, No. 2–3, 95).—The root is air-dried and then dried at 45° in plant which is described. The root, which contains 60–75% of moisture, is subject to rapid fermentation with loss of tannin.

CHEMICAL ABSTRACTS.

**Preparation and preservation of sheepskin skivers for colour tests [on tanning materials].** Report of a Committee of the American Leather Chemists' Association. C. A. BLAIR (J. Amer. Leather Chem. Assoc., 1930, 25, 550–561).—In the preferred method, a pickled sheepskin skiver is agitated for 4–5 hrs. with a 0.8% borax solution, transferred to a fresh borax solution, set aside overnight, transferred to a 0.1% acetic acid solution for 2 hrs. with frequent stirring, changed to another acetic acid solution of the same concentration, and after 3 hrs. with occasional agitation put into a buffer solution ( $p_H$  4.8) prepared from equal parts of 0.059*N*-acetic acid and 0.143*N*-sodium acetate. After remaining overnight in this solution the skiver is preserved in a solution containing 1% phenol and 2% boric acid. In making colour tests, a piece of skiver (4 × 6 in.) is drained, weighed, and shaken for 1 hr. with 400 c.c. of a solution of one half of its weight of the sample of tannin extract to be tested. After allowing the skiver piece to remain in the solution overnight, it is shaken for ½ hr., washed, nailed out

(grain side down) on a filter paper, and dried without artificial heat.

D. WOODROFFE.

**Determination of moisture in tanning extracts.** H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1930, 25, 578—580).—The higher results obtained by the direct method and recommended by Parker (B., 1929, 1024) are attributed to oxidation as a result of prolonged drying.

D. WOODROFFE.

**Production of artificial tanning materials.** ANON. (Vestnik, 1928, No. 2—3, 91).—Coal-tar distillates, b.p. 280—360°, are preferably sulphonated at 120—125° for 6—10 hrs. A satisfactory product is obtained in 4% yield by sulphonation of birch tar. By removal of the fraction of anthracene oil having b.p. below 170°, sulphonation may be effected in 1—1.5 hrs. with ordinary sulphuric acid at 100°.

CHEMICAL ABSTRACTS.

See also A., Feb., 227, [Determination of mixed] solutions of gallic acid and tannin (STEENHUISEN).

#### PATENTS.

**Manufacture of products resembling natural albumin [artificial horn].** PFENNING-SCHUMACHER-WERKE, G.M.B.H. (B.P. 313,455, 11.6.29. Ger., 11.6.28).—Albumin-like products are prepared by adding aqueous ormaldehyde to a solution of urea in aqueous ammonium sulphide; on evaporation the syrupy product gives first a gel and finally a clear, highly refractive solid mass which may be powdered and moulded by pressure and heat. The same products are obtained by adding urea to a mixture of aqueous formaldehyde and ammonium sulphide.

C. HOLLINS.

**Drying and cleaning of hides [by hot air and suction].** P. HERTZSCH (B.P. 340,940, 1.10.29).

**Bleaching skins etc.** (U.S.P. 1,759,341).—See VI. Imitation leather (B.P. 340,305).—See XIV.

### XVI.—AGRICULTURE.

**Chemistry of soil.** L. WOLF (Z. angew. Chem., 1930, 43, 922—928).—A centrifuge suitable for determination of the sand content of different particle sizes in soil is described. The function of chalk in soil and the causes of acidity of soils are discussed.

J. W. SMITH.

**Atterberg consistency constants [of soil].** L. D. BAVER (J. Amer. Soc. Agron., 1930, 22, 935—948).—Factors affecting the upper and lower plastic limits and the plasticity number are discussed. The constants appear to offer a means of predicting the values of the dynamic properties of any plastic soil.

CHEMICAL ABSTRACTS.

**Comparison of effects of blast-furnace slag and limestone on an acid soil.** F. H. CRANE (J. Amer. Soc. Agron., 1930, 22, 968—973).—On the basis of weight, the limestone was superior in its effects on the growth of red clover and on soil reaction, but on the basis of calcium carbonate content, as determined by titration, using phenolphthalein as indicator, the materials were equally effective.

CHEMICAL ABSTRACTS.

**Effect on plant growth of treating soils with copper-carrying pyrite.** H. V. SMITH (J. Amer. Soc. Agron., 1930, 22, 903—915).—Application of waste pyrite (1.26—1.63% Cu) increased the sulphate content

of the soils; the  $p_H$  of acid soils was lowered, but that of alkaline soils was unchanged. The pyrite may be applied to calcareous soils in fairly large amounts, but acid soils should be treated sparingly unless lime is also added.

CHEMICAL ABSTRACTS.

**Retrograde power of soil under various conditions.** F. V. TRIFONOV (Ukraine Chem. J., 1930, 5, [Tech.], 77—92).—The solubility of phosphates added as fertiliser to Ukrainian black soil is diminished by about 30% with augmentation of the water content from 30 to 100% saturation, whilst Izjum phosphate, containing citric acid, is 63% more soluble at complete saturation of the soil. The solubility of ordinary phosphate fertiliser decreases with rise in temperature, whilst that of Izjum phosphates increases. The action of the soil in diminishing the solubility of phosphate varies according to the locality, but is on the whole greater in more fertile soil.

R. TRUSZKOWSKI.

**Determination of readily available phosphorus in soils.** E. TRUOG (J. Amer. Soc. Agron., 1930, 22, 874—882).—The readily available ("available") phosphorus probably exists as calcium phosphate, and the difficultly available ("unavailable") as basic ferric phosphate. In the determination of the former in 1 g. of soil, 200 c.c. of 0.002*N*-sulphuric acid, buffered with ammonium sulphate to  $p_H$  3, is employed, with agitation, for 30 min., the phosphorus in the extract being determined by Denigès' colorimetric method.

CHEMICAL ABSTRACTS.

**Field test of different sources of phosphorus [for plants].** C. F. NOLL, F. D. GARDNER, and C. J. IRVIN (Penn. Agric. Exp. Sta. Bull., 1930, No. 252, 14 pp.).—The order of efficiency of the fertilisers tested was basic slag > steamed bone meal > superphosphate > rock phosphate. Yields from triple equivalent dressings of rock phosphate approached those of a single equivalent of superphosphate. The residual effect of rock phosphate in subsequent seasons was no greater than that of superphosphate. Supplementary applications of sulphur and of gypsum increased the efficiency of rock phosphate.

A. G. POLLARD.

**Solubility of soil phosphates and silicates as a basis for the determination of phosphate requirement.** E. HOFFMANN (Landw. Jahrb., 1930, 72, 791—834).—The plant-assimilable phosphate is satisfactorily characterised by means of water extracts in soils poor in bases, and by calcium bicarbonate extracts in soils rich in bases. Repeated extraction of soils with either solvent, whilst demonstrating differences in solubility ratios, does not yield information as to their phosphate requirements. The leaching of bases resulting from repeated water extraction permits the decomposition of soil phosphates which would not be dissolved under normal soil conditions. As extraction proceeds, a final condition is attained in which constant proportions of phosphorus appear in the leachate. The total reserve of easily soluble phosphate in soils cannot be determined by repeated extraction with solvents. The use of citric acid for this purpose may lead to the formation of complex phosphorus compounds or to reduced dissociation due to citrates formed. An acid buffer solution which cannot form complex compounds is essential for

the extraction of assimilable phosphate. The water-soluble silica content of soils bears no relation to the soluble phosphate content, but is to some extent related to productivity. This effect appears to depend on the ability of silicates to counterbalance the depression of phosphate solubility caused by calcium carbonate.

A. G. POLLARD.

**Tests of tobacco fertilisers comparing nitrogen, phosphorus, and potassium from different fertiliser sources.** T. B. HUTCHESON and T. L. COPLEY (Virginia Agric. Exp. Sta. Tech. Bull., 1929, No. 35, 10 pp.).—Inorganic sources of nitrogen were more efficient than was organic material in tobacco-leaf production. Among the organic materials examined, animal tankage, cottonseed meal, and fish scrap were the most successful. Superphosphate was the most satisfactory of the phosphate fertilisers. Potash fertilisers containing chlorides, when not used in excessive amounts, improved the yield and quality of the leaf. Chlorides increase the resistance of the plants to drought spot, but, used in large quantities, diminish the burning qualities of the leaf.

A. G. POLLARD.

**Lysimeter investigations. I. Nitrogen and water relations of crops in legume and non-legume rotations.** R. C. COLLISON and J. E. MENSCHING (N.Y. State Agric. Exp. Sta. Tech. Bull., 1930, No. 166, 90 pp.).—The amount of drainage from lysimeters was largely controlled by the dry-matter production in the crops. Nitrogenous fertilisers decreased the utilisation of water by crops. As the available nitrogen in the soil increased, the transpiration ratio for barley decreased. The inclusion of lucerne in the rotation increased the yields of the other crops to a greater extent than did applications of nitrogen as dried blood. This effect was mainly apparent in the succeeding crop, and decreased rapidly. Both lucerne and timothy exhausted the soil of nitrate during the growth season, but the nitrogen available for subsequent crops was about 3 times as great after lucerne as after timothy. Drainage losses of nitrogen during rotations including lucerne were high. Heaviest losses of nitrogen from soils occurred by crop removal, the amounts averaging 4–6 times that lost in drainage. When soil was fallowed after lucerne, nearly one half of the available nitrogen was lost in drainage. The total nitrogen lost from lysimeters during 12 years was approximately one fifth of that originally present in the soil. Much available nitrogen in the soil increases the nitrogen content of straw. Lysimeters 8 ft. deep produced 8% more dry matter, but lost 25% more water and 47% more nitrogen than those 2 ft. deep.

A. G. POLLARD.

**Relation of nitrogen to the storage quality of fruit.** J. H. GOURLEY (Sci. Agric., 1930, 11, 130–136).—Storage decay in apples was not appreciably increased by the application of nitrogenous fertilisers to the trees, but was most prominent among highly coloured fruit of high carbohydrate content. In general, applications of nitrates produced less highly coloured fruit with higher water content, greater catalase activity, and higher nitrogen content, but had no definite effect on the  $p_H$ , total acid, or soluble pectin contents, or on respiration.

A. G. POLLARD.

**Nitrogen and organic matter as related to soil productivity.** W. L. POWERS and R. D. LEWIS (J. Amer. Soc. Agron., 1930, 22, 825–832).—The nitrogen and total carbon are increased by irrigation, crop rotation, or manuring, but the ratio is unchanged. The nitrate-supplying power of the soil appears to increase.

CHEMICAL ABSTRACTS.

**Normal magnesium carbonate in comparison with "light" and "heavy" oxides and carbonates in the soil.** W. H. MACINTIRE and W. M. SHAW (J. Amer. Soc. Agron., 1930, 22, 919–934).—Comparative lysimeter experiments were performed on magnesian materials at 8-ton and 32-ton lime-equivalent rates. The crystalline carbonate  $MgCO_3 \cdot 3H_2O$  gave the greatest magnesium outgo. All the materials depressed the outgo of lime and potash; sulphate outgo was enhanced. Nitrification is accelerated by additions at the lower, and repressed by those at the higher, rates.

CHEMICAL ABSTRACTS.

**Behaviour of berries towards various potassium salts.** F. WEISKE [with T. REMY] (Ernähr. Pflanze, 1930, 26, 269–277; Chem. Zentr., 1930, ii, 1565).—Experiments with red currants show that lack of potash, besides diminishing the yield, causes pathological leaf coloration and premature fall of leaves. If potassium is administered as chloride, the yield does not fall appreciably, but chloride injury appears. Gooseberries behave similarly when treated with potassium chloride. Black currants are much less sensitive to both effects.

A. A. ELDRIDGE.

**Potato. III. Effect of potash fertilisation on sensitivity to frost of the potato plant.** H. WARTENBERG (Arb. biol. Reichsanst. Land- u. Forstwirtschaft., 1929, 17, 377–384; Chem. Zentr., 1930, ii, 1384).—Potash fertilisation is of no practical value as protection against frost. The sensitivity of the plants is in fact increased.

A. A. ELDRIDGE.

**Production of artificial farm manures.** F. B. SMITH, W. H. STEVENSON, and P. E. BROWN (Iowa Agric. Exp. Sta., Res. Bull. No. 126, 1930).—Straw was composted with various nitrogenous materials in the preparation of farmyard manure. Analytical data show the effects of aeration, water content, and nature of nitrogen supply on the rate and manner of decomposition of the material, losses of dry matter and nitrogen, and the nitrogen content of the product. In most cases inoculation of the rotting stack with infusions of soil or manure did not markedly increase the rate of its decomposition.

A. G. POLLARD.

**Effects of artificial farm manures on soils and crops.** F. B. SMITH and P. E. BROWN (Iowa Agric. Exp. Sta. Res. Bull. No. 127, 1930).—Comparison is made of the effects on soil and plant growth of natural and artificial farmyard manure (cf. preceding abstract). Very similar bacteriological conditions and nitrate accumulation and assimilation are recorded.

A. G. POLLARD.

**Artificial manuring of flax.** F. HIEKE (Ernähr. Pflanze, 1931, 27, 26–29).—Applications of calcium cyanamide increased the yield of straw and more especially of the seed of flax, but the yield and quality of fibre were not correspondingly improved. Kainite increased

the yield of retted fibre. The effect of basic slag on the crop was small.

A. G. POLLARD.

**Finger-and-toe [in *Brassicæ*].** SCHUMACHER (Ernähr. Pflanze, 1931, 27, 30—31).—In addition to the customary methods for eradicating finger-and-toe, balanced manuring is an important preventive measure. Applications of potash and phosphate are frequently omitted for these crops, but are valuable in increasing the resistance of plants to infection.

A. G. POLLARD.

**Influence of soil conditions, fertiliser treatment, and light intensity on growth, chemical composition, and enzymic activities of sugar beet.** J. TYSON (Michigan Agric. Exp. Sta. Tech. Bull., 1930, No. 108, 44 pp.).—The mineral contents of the leaves and roots of sugar beet are markedly influenced by soil conditions. The intensity of light to which plants are exposed affects the utilisation of minerals rather than their absorption. Optimum nutrient requirements for beet are examined and the mineral constituents of crops recorded. In general, the proportion of minerals in the plants was highest in spring and low in summer, rising again in autumn on sufficiently rich soils. Potash fertilisers increased the sugar content of the roots only on deficient soils. In most cases the maximum sugar content occurred among the heaviest crops, except where sodium nitrate had been applied in excessive amounts or late in the season. The sugar content was not influenced by light intensity until the latter was sufficiently decreased to inhibit further growth. Catalase activity was positively correlated with the vigour of growth and the size of the plant. Oxidase activity was greatest under conditions which inhibited further growth.

A. G. POLLARD.

**Control of thistles.** ROSING (Ernähr. Pflanze, 1931, 27, 31—32).—Thistles in meadowland were almost completely eradicated by broadcasting kainite while the plants were in the early stages of growth.

A. G. POLLARD.

**Changes occurring in stored alcoholic plant extracts.** J. E. WEBSTER (Science, 1931, 73, 77—78).—Plant samples prepared in the usual manner by adding an excess of calcium carbonate have been found to be generally acid, the acidity depending on the material preserved. The adjustment of  $p_H$  values of alcoholic solutions to 5.8—6.0 with 0.1*N*-caustic soda is preferred to the addition of calcium carbonate. Acidity is an important factor in the nitrogen changes, the continuous increase in ammonia formed on keeping being greater in alkaline solutions.

L. S. THEOBALD.

**Natural nicotine-free and nicotine-poor tobaccos.** P. KOENIG (Forschungen u. Fortschritte, 1930, Dec., Reprint, 1 p.).—The low nicotine content of the pure breeds of tobacco plants described is hereditary, and the quantity of nicotine in the tobacco made from the leaves is independent of the manner of growth of the plant; the albumin content also remains constant.

E. H. SHARPLES.

**Production of natural nicotine-free, nicotine-poor, and nicotine-rich tobaccos.** P. KOENIG (Die Umschau, 1930, 40, Reprint, 1 p.).—Results of an examination of a large number of tobacco plants are

described. Variations in nicotine content of the leaves under varying conditions are discussed; the content is highest in the highest leaves (shortly before ripening), and is lower in greenhouse plants, close-planted, and irrigated plants than in manured, open-air plants. Pure breeds of plants, even when grown under widely varying conditions giving green leaves of different nicotine contents, after drying, give tobaccos of constant nicotine content, and by breeding stocks of low and no nicotine content, tobaccos are obtained having aroma and flavour indistinguishable from high-nicotine tobacco.

E. H. SHARPLES.

**Relation between insecticidal action and physical properties of soap solutions.** P. A. VAN DER MEULEN (J. Econ. Entom., 1930, 23, 1011—1012).—The varying toxicity of different soap solutions is associated with their ability to form tough adherent films on the surface when exposed to air.

A. G. POLLARD.

**Petroleum insecticides.** C. W. WOODWORTH (J. Econ. Entom., 1930, 23, 848—851).—A general survey of the grades of oil used in insecticides and of their varied effects on plants.

A. G. POLLARD.

**Physical properties of certain dormant oil emulsion-sulphur [insecticidal] combinations.** M. D. FARRAR and M. A. SMITH (J. Econ. Entom., 1930, 23, 979—985).—Insecticidal combinations of sulphur with oil emulsions were much more effective when the latter were emulsified with gum than with fish-oil soap, caseinate, or petroleum soaps. When water, emulsifier, and sulphur are mixed before the addition of oil, sulphur disperses in the water phase, and only to a small extent in the emulsifier interface. From such preparations sulphur settles rapidly. If a stock oil emulsion and sulphur are combined, sulphur enters the emulsifier interface and the oil droplets carry a coating of sulphur. Dilution of such preparations does not lead to the separation of sulphur. Addition of water to the stock oil emulsion when mixing with sulphur causes a decrease in the diameter of the oil droplets, and also in the number of droplets carrying sulphur. The particle size of the sulphur used is a minor factor in controlling the stability of the emulsion, but the small amount of electrolyte present in certain flotation sulphurs increases the adherence of sulphur to the oil droplets. Increasing proportions of sulphur in the mixture cause an increase in the size of the oil droplets and in the proportion carrying sulphur.

A. G. POLLARD.

**Relative value as insecticides of some constituents of *Derris*.** W. M. DAVIDSON (J. Econ. Entom., 1930, 23, 877—879).—The order of toxicity of *Derris* constituents toward aphids, white-fly larvae, thrips, and red spider mites was: rotenone > deguelin > tephrosin > toxicarol. Each of the first two substances named was more toxic than nicotine to aphids.

A. G. POLLARD.

**Rotenone as a contact insecticide.** W. M. DAVIDSON (J. Econ. Entom., 1930, 23, 868—874).—Comparative tests are recorded of rotenone, used as a dust and in wet sprays, against numerous classes of insects.

A. G. POLLARD.

**Nicotine in paint for woolly aphid control.** L. CHILDS (J. Econ. Entom., 1930, 23, 883).—Addition



of nicotine to tree paints satisfactorily controlled woolly aphids. A. G. POLLARD.

Neonicotine and certain other derivatives of the dipyridyls as insecticides. C. R. SMITH, C. H. RICHARDSON, and H. H. SHEPARD (J. Econ. Entom., 1930, 23, 863—867).—Among numerous dipyridyl derivatives examined, neonicotine (pyridylpiperidine) proved the most toxic towards aphids. A. G. POLLARD.

Comparison of the toxicity of *p*-dichlorobenzene and naphthalene to the confused flour beetle (*Tribolium confusum*, Duv.). R. S. LEHMAN (J. Econ. Entom., 1930, 23, 958—966).—Air saturated with *p*-dichlorobenzene acts as a strong anæsthetic to *T. confusum*. On the basis of time required to kill 50% of the insects, naphthalene was 10—14 times as toxic as *p*-dichlorobenzene at similar concentrations.

A. G. POLLARD.

Effect of boron on powdery mildew and spot blotch of barley. F. M. EATON (Phytopath., 1930, 20, 967—972).—In sand cultures spot blotch on barley increased in severity with increasing concentrations of boron in the nutrient. None developed where boron was absent. On the other hand, powdery mildew appeared only where boron-free nutrients were used. The intake of boron by wheat and barley increased with the concentration of that element in the nutrient.

A. G. POLLARD.

Killing eggs of the codling moth on harvested fruit. E. J. NEWCOMER (J. Econ. Entom., 1930, 23, 798—802).—Treatment of apples and pears with a 1% oil emulsion in a machine used for removing spray residues kills adhering codling moth eggs without injury to the fruit.

A. G. POLLARD.

Tests of miscellaneous chemicals against the codling moth. L. C. McALLISTER (J. Econ. Entom., 1930, 23, 907—922).—Laboratory tests with over 200 substances against newly-hatched larvae of the codling moth are recorded.

A. G. POLLARD.

Trap baits for capturing codling moth. M. A. YOTHERS (J. Econ. Entom., 1930, 23, 923—929).—Addition of geraniol to trap baits containing syrup etc. increased their efficiency in codling moth control.

A. G. POLLARD.

Effect of chemical treatment of seeds on the tropic stimuli of *Gramineæ*. J. NEUKIRCHEN (Planta, [Z. wiss. Biol.], 1930, 12, 505—531).—Treatment of seeds with a variety of common fungicides and insecticides modified the geotropic and phototropic phenomena and affected the rate of growth after germination.

A. G. POLLARD.

Aeroplane dusting in the control of cotton boll worm (*Heliothis obsoleta*, Fab.). F. SHERMAN (J. Econ. Entom., 1930, 23, 810—813).—Applications of 5—6 lb. of calcium arsenate per acre were successful in controlling the cotton boll weevil, but damage by the cotton boll worm appeared to be increased.

A. G. POLLARD.

Insecticides against cattle grubs (*Hypoderma* Sp.). F. C. BISHOPP, E. W. LAAKE, R. W. WELLS, and H. S. PETERS (J. Econ. Entom., 1930, 23, 852—863).—

Dusts containing ground *Derris* root gave excellent results in the control of cattle grubs. Powdered tobacco and dusts containing nicotine and nicotine sulphate were good, whilst pyrethrum, *p*-dichlorobenzene, and colloidal sulphur were inferior.

A. G. POLLARD.

Determination of sulphur in insecticides and fungicides by carbon disulphide extraction. R. EDGEMOND (Ind. Eng. Chem. [Anal.], 1930, 2, 371—373).—Soluble material is first removed by treatment with 1:2 nitric acid, and the residue, after thorough washing, is heated at 105—110° for 4—4.5 hrs.; by this procedure all the sulphur present is converted into a form soluble in carbon disulphide, with which it may be extracted in the usual manner, and the danger of reaction between the sulphur and the other components of the mixture is practically eliminated. Equally satisfactory results may be obtained by heating the material overnight (17—18 hrs.) at 98—102°. The loss of sulphur during the heating at 110° does not exceed about 0.3%. If the surface of the material be moistened with dilute (1:3) alcohol before the treatment with acid, more thorough penetration by the latter is assured.

H. F. GILLBE.

Water-soluble arsenic in spray material. H. C. YOUNG (Ohio Agric. Exp. Sta. Bull., 1930, No. 448, 22 pp.).—Summer-strength mixtures of lime-sulphur and lead arsenate contain dangerous amounts of soluble arsenic which increase with the dilution of the lime-sulphur up to 1:100. Arsenic solubility is reduced by the addition of freshly-slaked high-grade lime. Lime containing much magnesia is less effective. Commercial manganese arsenate (Manganar) does not react with lime-sulphur to produce soluble arsenic. Wettable sulphur sprays react only slightly with Manganar or lead arsenate.

A. G. POLLARD.

Toxicity of sodium chlorate [to animals]. E. SEDDON and T. T. McGRATH (Agric. Gaz., N.S.W., 1930, 41, 765—766).—Sodium chlorate is palatable to cattle which can consume sufficient, supplied as a salt lick, to cause death. The possibility of injury to cattle from chlorate weed-killers is discussed.

A. G. POLLARD.

Analysis of insecticides. LANDIS and others.—See III. Sulphuric acid-treated phosphorite. EGOROV.—See VII. Potash from industrial alcohol. BROUGHTON and others.—See XVIII. Digestibility of cottonseed meal for cattle. WATKINS.—See XIX.

See also A., Feb., 186, Determinations of phosphoric acid (VON ENDRÉDY). Determination of carbonate in soils (NICLOUX). Volumetric determination of sodium (BLENKINSOP). 192, Moors of Tekir-Ghiol and Agigea (DELEANU and HOFMANN). Development of soil profile in N. Wales (ROBINSON). Types of clay in south-west Finland (AARNIO). Tuff-soils of Maifeld (PFEFFER). 265, Aërobic cellulose-decomposing soil bacteria (KALNINS; MAKRIKOV and STEPANOVA). Bacterial fixation of free nitrogen (BAZAREWSKI). Assimilation of phosphorus by *Azotobacter* (ZIEMIECKA). 266, Biological reduction of mineral phosphates (RUDAKOV). 272, Assimilation of carbon dioxide by plants (NEYDEL). Development and ripening of peaches (NIGHTINGALE and others). 273, Nutrition of fruit trees (DAVIS). Effects of

potassium deficiency on plants (NIGHTINGALE and others).

## PATENTS.

**Manufacture of fertilisers.** ARMOUR FERTILIZER WORKS (B.P. 340,120, 11.12.29. U.S., 11.12.28).—Fertiliser mixtures containing acidic material, *e.g.*, superphosphates, are neutralised rapidly in a mixer by the addition of liquid anhydrous ammonia.

L. A. COLES.

**Drying of agricultural produce.** V. I. PETTERSSON (B.P. 339,563, 24.2.30. Ger., 22.2.29).—The material, *e.g.*, cut grass and stalked cereals, is piled up so as to form hollow cylinders and passed on a guiding track through a chamber swept by air.

H. ROYAL-DAWSON.

**Investigating [electrically] the geological structure of the sub-soil.** R. AMBRONN (B.P. 339,964, 12.9.29. Ger., 5.12.28).

**Fungicide** (U.S.P. 1,766,412).—See XIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Investigation of dry beets.** G. S. BENIN (Nauk. Zapiski Tzuk. Prom., 1930, 9, 273—287).—Cossettes containing 5—6% of water are ground; those containing more than 6% of water are mixed with an equal quantity of water, the time of preparation of the sample being short in both cases. Convenient quantities for the determination of sucrose (polarimetric) and invert sugar (Fehling's solution) are mentioned. The elimination of lead from the filtrate is unnecessary. The water is determined by distillation with xylene.

## CHEMICAL ABSTRACTS.

**Carbonatation and sulphitation to low  $p_H$  of remelt brown beet and raw cane sugars.** I. B. MINTZ and B. E. KRASILSHCHIKOV (Nauk. Zapiski Tzuk. Prom., 1930, 9, 319—330).—The remelt is preferably treated with lime (2% of the weight of sugar), carbonated to  $p_H$  9—10, and filtered; the filtrate is sulphited to  $p_H$  7.0—7.5. Raw cane sugar melt is treated similarly, the sulphitation being a little higher.

## CHEMICAL ABSTRACTS.

**Polarisation of raw sugars.** F. W. ZERBAN (Facts about Sugar, 1930, 25, 438—440).—Tabulated values of direct polarisation of samples examined over a period of 20 years at the New York Sugar Trade Laboratory are given.

## CHEMICAL ABSTRACTS.

**Vitamins in sugar-cane juice and in some cane juice products.** E. M. NELSON and D. B. JONES (J. Agric. Res., 1930, 41, 749—759).—Juice from the upper portions of cane stalks is richer in the antineuritic vitamin than that from lower portions, although both are poor. Juice obtained from bagasse by high-pressure methods is richer in this vitamin than ordinary cane juice. Sugar-cane juice contains small amounts of vitamin-A and little, if any, of vitamin-D. Vitamin-B could not be demonstrated in cane syrup, blackstrap molasses, or cane cream.

A. G. POLLARD.

**Use of kieselguhr for clarification of thick syrups and centrifugal syrups.** A. A. RAGOZIN (Zhur. Sakh. Prom., 1930, 4, 181—186).—The surface tension of thick and centrifugal syrups increases after treatment

with dry kieselguhr and addition of milk of lime to an alkalinity of 0.05% CaO. Further increase of surface tension is obtained by treating the syrups with the same amount of kieselguhr previously mixed with a small quantity of thick syrup and milk of lime. Subsequent sulphitation to  $p_H$  6.8—7.0 is recommended, and filtration is carried out at 92°. CHEMICAL ABSTRACTS.

**Colour in the sugar industry. III. Preparation of asbestos for use as a filter aid.** J. F. BREWSTER and F. P. PHELPS (Ind. Eng. Chem. [Anal.], 1930, 2, 373; cf. Peters and Phelps, B., 1927, 710).—The asbestos (25 g.) is boiled for 30 min. with 25% sodium hydroxide solution (250 c.c.), filtered hot with suction, and washed repeatedly; after pressing out most of the water, the material is heated on the water-bath for 30 min. with hydrochloric acid ( $d$  1.20, 250 c.c.) and nitric acid ( $d$  1.42, 25 c.c.); hot water is added and the asbestos is filtered, washed with hot water until free from acid, and dried at 110°. Colloidal impurities and iron oxide, which may cause a change of colour of the sugar solution, together with the very fine fibres, are removed by this treatment, which may be completed within 2 hrs.

H. F. GILLBE.

**Limiting decomposition of starch under the action of distillery malts of various origins.** A. JOSZT and A. KLEINDIENST (Przemysl Chem., 1930, 14, 537—541).—The limiting hydrolysis of starch attained by the action of various barley malts varies from 51.7 to 56.2%.

R. TRUSZKOWSKI.

**Reactivation of carbon.** SIPYAGIN and SERKIN.—See II. **Sugar beet.** TYSON.—See XVI.

See also A., Feb., 187, **Determination of reducing sugars** (INTONTI). 199, **Titration of aldose sugars** (KLINE and ACREE). **Determination of reducing sugars** (SCHUETTE and TERRILL). 202, **Hydrolysed inulin** (JACKSON and McDONALD). 263, **Fermentation of sugar mixtures by Sauterne yeast** (SOBOTKA and REINER).

## PATENT.

**Radioactive chocolate** (B.P. 339,034).—See XIX.

## XVIII.—FERMENTATION INDUSTRIES.

**Potato flakes as raw material in distilleries.** E. LÜHDER (Z. Spiritusind., 1931, 54, 7—9).—Two detailed methods are given for the use of potato flakes in the mash from which alcohol is produced. In the first of these the flaked potatoes are cooked and liquefied previous to their saccharification, whereas in the second method the preliminary stages are omitted and saccharification is proceeded with directly. In the latter method, provided the mash is kept liquid and free from "balling" by vigorous stirring, the yield of alcohol is slightly greater. Modifications of the methods are necessary when the potato flakes are partly substituted by raw potatoes or by some cereal such as maize.

C. RANKEN.

**Bottling [of beer].** A. HADLEY (J. Inst. Brew., 1931, 37, 11—15).—The relative merits of the slow, the semi-rapid, and the quick systems of chilling are discussed in connexion with the bottling of beer. Filtra-

tion, collection of fermentation carbon dioxide, bottle cleansing, and labelling are also dealt with.

C. RANKEN.

**Haze in bottled beer.** H. L. HIND (J. Inst. Brew., 1931, 37, 15—19).—The haze which is liable to form in beer in bottle is largely of protein origin, and is chiefly due to the disturbance of the equilibrium among the colloidal constituents. Changes in the  $p_H$  of the beer, which may be produced either by bacterial development with the production of acidity or by the dissolution of alkali from the glass, and traces of metals are frequent causes of these alterations in equilibrium. The haze may be prevented or minimised by the removal of the undesired proteins by the addition of adsorbents, or by their conversion into a more soluble form by adding proteolytic agents, such as papain, to the beer. Cold storage and filtration by the Metafilter or by the Seitz filter have their advantages, but also tend to remove the desirable head-retaining colloids.

C. RANKEN.

**Potash from industrial alcohol.** L. B. BROUGHTON, H. L. MARSHALL, and N. C. THORNTON (Maryland Agric. Exp. Sta. Bull., 1928, No. 300, 37—61).—In the manufacture of alcohol from molasses, ash obtained from boiler furnaces fired with molasses residue contained 34% of water-soluble  $K_2O$ , 1.7% of citric-soluble  $P_2O_5$ , and a small proportion of nitrogen. The material was too alkaline for use as a substitute for potash salts in compound fertilisers and caused reversion of acid phosphate and loss of ammonia from ammonium salts. Satisfactory mixtures are described in which the material is used in admixture with other potash salts or is compounded with tankage, ammonium sulphate (or sodium nitrate), and superphosphate.

A. G. POLLARD.

**Action of distillery malt on starch.** JOSZT and KLEINDIENST.—See XVII.

See also A., Feb., 160, Adsorption of invertase by charcoal (MILLER and BANDEMER). 252, Lactenin (JONES and SIMMS). 263, Fermentation of sugar mixtures by Sauterne yeast (SOBOTKA and REINER). Growth of yeast by aeration (CLAASSEN). Activator-Z (PHILIPSON). Bios (SUZUKI and others). 264, Lipase of *Aspergillus niger* (JURACEC). Citric fermentation (KOTOVSKI). 265, Reduction of nitrates by bacteria (KORSAKOV). 266, Lactic fermentation (VIRTANEN and TIKKA). Biochemical preparation of optically active glyceraldehyde (NEUBERG).

#### PATENTS.

**Manufacture of yeast.** G. S. BRATTON, Assr. to ANHEUSER-BUSCH, INC. (U.S.P. 1,767,646, 24.6.30. Appl., 6.3.26).—Propagation of yeast is initiated in a setting solution which is rich in yeast-assimilable proteins and nutrient salts, but deficient in assimilable sugars. A feeding solution which is rich in assimilable sugars but deficient in assimilable proteins and nutrient salts is added during propagation continuously or intermittently to the diluted and aerated setting solution.

C. RANKEN.

**Malting of cereals.** T. R. DIXON (B.P. 339,047, 7.10.29).—The enzymic action of the grain is accelerated by sprinkling the steeped and germinating grain with a 10% solution of lactic acid as soon as the acrospire

penetrates through the testa. After further germination, the grain is steeped in a lactic acid solution of about 20% concentration for about 24 hrs. prior to being transferred to the kiln.

C. RANKEN.

**Treatment of beverages.** A. GUSMER (U.S.P. 1,765,667, 24.6.30. Appl., 29.7.25).—Fermented beverages of cereal or fruit origin are heated to remove the alcohol in excess of that specified by the National Prohibition Act. Sulphurous acid and sugars are then added and the boiling is continued, the entire treatment being carried out under a vacuum.

C. RANKEN.

**Leavened bakery products** (B.P. 340,072). **Grape juice** (U.S.P. 1,767,399).—See XIX.

#### XIX.—FOODS.

**Relation between the quantities of fat and protein in normal milk.** A. E. PERKINS (Ohio Agric. Exp. Sta. Bull., 1930, No. 446, 126—127).—From analyses of grouped samples of milk the following formula is derived: protein (%) =  $2.78 + [0.42 (\text{fat \%} - 2.78)] \pm 0.25$ . The probable error of single determinations from mixed milk is likely to be smaller than that shown above.

A. G. POLLARD.

**Fluctuation of  $p_H$  in koumiss from mare's milk. Changes in sp. gr. and in fat and amino-nitrogen in koumiss.** K. S. ARKHPOV (Ark. Biol. Nauk, 1930, 30, 475—485).—The  $p_H$  varies from 3.4 to 4.3 according to the temperature and duration of keeping. The amino-nitrogen varies from 0.064 to 0.084% according to the temperature and duration of fermentation. The sp. gr. varies from 1.005 to 1.007. The fat content depends on that of the milk and on the duration of fermentation.

CHEMICAL ABSTRACTS.

**Determination of *B. coli* [in milk].** A. VAN RAALTE (Chem. Weekblad, 1930, 27, 663).—Milk brought to specified dilutions by addition of peptone solution is kept 24 hrs. at 37°, and the indole test applied. The milk may be graded according to the dilution range within which a positive result is obtained.

S. I. LEVY.

**Methylene-blue reductase test [for milk].** H. R. WHITEHEAD (New Zealand J. Sci. Tech., 1930, 12, 100—107).—A résumé of work carried out on this test as a method of grading milk for cheese manufacture. The number of *Streptococci lactis* is the chief factor affecting the reduction time, though other organisms may increase or retard the reaction. It is as accurate as the direct microscopical or the plate count, both of which require skill, whilst the last is too costly and time-consuming for industrial purposes.

T. McLACHLAN.

**Modification of the methylene-blue reductase test and its comparative value in determining the keeping quality of milk.** C. K. JOHNS (Sci. Agric., 1930, 11, 171—190).—The modifications ensure greater convenience and accuracy of working (especially with high-grade milks) and closer correlation with the keeping quality. A preliminary incubation at 12.8° for 18 hrs. is adopted, and in the subsequent incubation at blood-heat tubes not discoloured after 6 hrs. are shaken to redistribute bacteria carried to the surface with the rising butter fat.

A. G. POLLARD.

**Factors affecting the flavour of butter.** I. Effect of various starter cultures. II. Effect of various neutralisers. C. C. WALTERS (Ark. Agric. Exp. Sta. Bull., 1930, No. 249, 1—10, 11—26).—I. Commercial starters containing yeasts did not affect the flavour of the butter.

II. [With M. S. LIBBERT.] Chemical analyses of various neutralisers for sour cream are recorded. The alkalinity of these materials varied widely. Lime neutralisers containing magnesium had a higher neutralising value than purely calcic materials. The time of reaction of the neutralisers averaged 1—3 min. for soda compounds, 4—5 min. for lime neutralisers, and 10—15 min. for lime neutralisers containing magnesium. Reduction of acidity due to pasteurisation was greater for lime than for soda neutralisers. Butter flavour was improved by the use of neutralisers in all cases, the effect being more pronounced in fresh than in stored butter.

A. G. POLLARD.

**Analysis of butter.** E. S. GUTHRIE and others (J. Dairy Sci., 1930, 13, 380—393).—The A.O.A.C. and Kohman methods have been compared. For the determination of water the Kohman method is the more accurate, whilst for the fat determination the A.O.A.C. method is preferred. The Kohman and A.O.A.C. methods, respectively, gave average variations and ranges as follows: water 0.031 (0.001—0.087)%, 0.081 (0.006—0.221)%; fat 0.113 (0.002—0.337)%, 0.104 (0.004—0.129)%. CHEMICAL ABSTRACTS.

**Acidity changes associated with the keeping quality of apples under various storage conditions.** H. H. PLAGGE and F. GERHARDT (Iowa Agric. Exp. Sta. Res. Bull. No. 131, 1930).—Decreased acidity of apples during storage varied with variety and season, becoming more rapid with rising temperature. The appearance of "soggy breakdown" was definitely associated with changes in titratable acidity and  $p_H$  value, being more extensive where acid losses were small. Susceptibility to Jonathan spot was inversely proportional to the total acid loss during storage. The storage capacity of some varieties of apples was correlated with the density of the juice.

A. G. POLLARD.

**Preserving fruits by freezing.** I. Peaches. II. Figs. J. G. WOODROOF (Georgia Agric. Exp. Sta. Bull., 1930, Nos. 163 and 164).—I. The effect on the coloration of peaches of numerous chemicals added prior to freezing is examined; of these, 2% citric acid, 4% phosphoric acid, and 2% sodium chloride tended to whiten the flesh.

II. [With J. E. BAILEY.] The use of very low temperatures for freezing figs destroyed the pigment of the seed cavity.

A. G. POLLARD.

**Sampling of apples for arsenical spray residue determinations.** J. R. NELLER (Ind. Eng. Chem. [Anal.], 1930, 2, 382—384).—Study of the arsenic content of apples washed with hydrochloric acid and containing 0.017—0.019 grain/lb. shows the average probable error of single samples of 6 apples each to be 8.2%, and of duplicate samples 5.36%, of the total arsenic present; since the average probable error of the analysis is 7.40%, 2 samples of 6 apples each provide sufficiently accurate sampling. The rate of reduction of

the sampling error with increase of the number of samples falls off rapidly. These results are applicable to apples containing 0.01 grain or less of arsenic per lb.

H. F. GILLBE.

**Detection of benzoic acid in jams, fruit juices, and margarine.** J. J. J. DINGEMANS (Chem. Weekblad, 1930, 27, 640).—The colour test based on nitration to *m*-dinitrobenzoic acid and treatment with ammonia and ammonium sulphide is not always satisfactory. A more sensitive test is to nitrate and add hydroxylamine hydrochloride. Salicylic acid, if present, is first oxidised with alkaline permanganate.

S. I. LEVY.

**Digestibility of cottonseed meal as a supplemental feed for range cattle in New Mexico.** W. H. WATKINS (New Mexico Coll. Agric. Tech. Bull. No. 178, 1929).—Addition of cottonseed meal to a ration of wheat straw for steers increased the digestibility of the following straw constituents: dry matter, total organic matter, total nitrogen, protein nitrogen, and ether extract.

A. G. POLLARD.

**Sodium metasilicate.** VAIL.—See VII.

See also A., Feb., 185, Rapid determination of water [in wheat] (LONGINESCU and PIRTEA). 190, Measurement of surface temperature [of milk-drying rolls] (ROESER and MUELLER). 245, Optical rotations of cereal glutelins (CJONKA and others). 251, Human milk: carbohydrates and analysis (POLONOVSKI and LESPAGNOL). 252, Antirachitic vitamins of human milk (MACCHI and SCALFATI). Lactenin (JONES and SIMMS). 256, Food proteins of milk, eggs, and meats (CLAYTON and CUMMINGS). Nutritional potency of liver (McHARGUE and others). Nutritive value of cereal breakfast foods (BLOUGH and others; MATTILL and CLAYTON). Effect of cooking on digestibility of meat (CLIFFORD). Synthetic milk (RANDOIN and LECOQ). 257, Carbohydrate content of foods (McCANCE and LAWRENCE). 269, Vitamins in grape juice and wine (RANDOIN). Vitamins of water-cress (MENDEL and VICKERY). Determination of vitamin-A (COWARD and others). 270, Isolation of vitamin-B<sub>1</sub> (VAN VEEN). Vitamin-B in leafy vegetables (ROSCOE). 271, Vitamin-B<sub>2</sub> in egg-white (CHICK and others). Assay of vitamin-B<sub>2</sub> (READER).

## PATENTS.

**Production of leavened bakery products.** STANDARD BRANDS, INC., Assees. of A. D. BLANK, H. A. KOHMAN, and A. SCHULTZ (B.P. 340,072, 18.10.29, U.S., 22.10.28).—A mixture is described which aids the maturing of yeast-raised dough and reduces the fermentation period. The mixture consists of phosphoric acid or an acid phosphate, an ammonium salt, potassium bromate or iodate, and an enzyme preparation such as papain, pepsin, or diastase.

E. B. HUGHES.

**Cleaning and preparing of rice.** C. J. ROBINSON, and T. ROBINSON & SON, LTD. (B.P. 339,014, 16.9.29).—To reduce the amount of broken rice obtained in the polishing process, the hulled rice is steeped in water and then steamed. The grains are dried before milling.

E. B. HUGHES.

**Production of cereal coffee substitutes.** H. OHLHAVER (B.P. 339,821, 2.1.30).—Cereal substances are covered with water and maintained at about 25° for 36–48 hrs., during which self-fermentation (non-alcoholic) takes place. The product is then drained and roasted. E. B. HUGHES.

**Production of radioactive chocolate.** G. SENFTNER (B.P. 339,034, 28.9.29).—A radioactive solution is used to hydrate anhydrous lactose or dextrose etc., and is retained in the water of crystallisation of the sugar. This crystallised sugar is then used in chocolate in place of part or all of the sucrose. E. B. HUGHES.

**Production of grape juice.** L. C. REILING (U.S.P. 1,767,399, 24.6.30. Appl., 17.5.27).—After the aromatic vapours have been extracted by heating the grape juice *in vacuo*, the colloids are coagulated by raising the temperature to 65–88°. The juice is then rapidly cooled to below 10° to precipitate the excess of tartrates, filtered, concentrated, and finally injected with the previously separated aromatic substances. C. RANKEN.

**Processing of animal matter** (U.S.P. 1,766,031–3).  
**Oil from blubber etc.** (B.P. 339,305).—See XII.  
**Beverages** (U.S.P. 1,765,667).—See XVIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Notes on alcohol determination [in pharmaceutical preparations].** R. E. SCHOETZOW (J. Amer. Pharm. Assoc., 1930, 19, 1060–1062).—Alkalis, such as milk of magnesia, prevent “bumping” in the distillation of alcohol, when added to preparations such as tincture benzoin compound in sufficient amount to combine with the resins present. Other preparations which are subject to excessive foaming can be distilled successfully if a slight excess of calcium chloride be added; it precipitates or combines with the foam-producing constituents. In the decolorisation of tincture of iodine with sodium thiosulphate, an excess must be avoided, otherwise the distillate will be acid or will contain sulphur compounds. In the determination of alcohol in spirit of nitrous ether according to the U.S.P. method, it is better first to decompose the ethyl nitrite with potassium iodide and sulphuric acid, decolorise the iodine as above with thiosulphate and sodium hydroxide, and make the necessary correction for the extra amount of alcohol. In the removal of volatile oils from distillates by extraction with light petroleum previous to redistillation, it is advisable to use the fraction b.p. 35–80°. Some preparations do not give clear distillates, but if magnesium carbonate be added to the alcoholic mixture after extracting with light petroleum, after agitation, chilling, filtering, and redistilling, a clear distillate is usually obtained.

E. H. SHARPLES.

**Phenol-water mixtures.** G. J. W. FERRY (Pharm. J., 1931, 126, 58).—The f.p. of the “liquid carbolic acid” mixture proposed for the new B.P. is 3.1° and the  $d_{4}^{15.5}$  is 1.0615. The f.p. limits are suggested as a useful pharmaceutical specification. F. J. WILKINS.

**Strychnine. II. Action of certain substances in masking the taste of strychnine.** J. C. WARD and J. C. MUNCH (J. Amer. Pharm. Assoc., 1930, 19,

1057–1060; cf. A., 1930, 1456).—The bitter taste of strychnine may be masked by a number of chemically unrelated substances, such as salts, sucrose, and yerba santa extract and in the salts the cation is the significant factor in the masking action, the efficiency of which decreases in the order: magnesium (78%), calcium (25%), sodium (8%), potassium (2%). Addition of sodium bicarbonate increases the bitterness, and the most efficient masking action was possessed by yerba santa extract, which increased the threshold of strychnine from its normal value of 5.4 micrograms to 36.4, on the addition of 5%. No relationship was found between acuity and speed of perception of the bitter taste of strychnine. No consistent masking effect could be attributed to the anions studied. E. H. SHARPLES.

**Lobeline. I. Evaluation of lobeline preparations and comparison with lobelanine and with the total alkaloids of *Lobelia inflata*.** M. NISISITA (Okayama Ig. Zasshi, 1929, 39, 1985–1986).—Of the preparations examined, a fresh solution of crystalline lobeline had the greatest toxicity and the greatest effect on respiration. The emetic action, which was greatest with brownish crystals of “lobeline sulf.” (Merck), appears to be produced by a decomposition product of lobeline. CHEMICAL ABSTRACTS.

***Polypodium occidentale*.** L. FISCHER and F. J. GOODRICH (J. Amer. Pharm. Assoc., 1930, 19, 1063–1071).—The rhizome (loss in air 75.44%, loss at 110° 3.48%, acid-insoluble ash 0.26%, acid-soluble ash 2.42%, water-insoluble ash 2.06%, water-soluble ash 0.62%) of *P. occidentale* (licorice fern), grown on the Pacific coast, gives 41.19% of aqueous extract (on the drug dried at 75°), and contains 13.6% of reducing sugars, 1.26% of starch, 6.22% of pentosans, 3.67% of tannins (chiefly of the pyrocatechol variety), 0.005% of volatile oil, a yellow colouring matter (dyes silk a fast yellow), 2.36% of glycyrrhizin and ammoniated glycyrrhizin (?). It contains no appreciable amounts of alkaloids and is non-toxic to white rats, even in large amounts. E. H. SHARPLES.

**Preparation of Carstanjen's compound.** A. A. HARWOOD (J. Amer. Pharm. Assoc., 1930, 19, 1171–1173).—Difficulties occurring in the preparation of this compound (potassium sulphite-thymoquinone) can be avoided by starting from freshly prepared sulphite as follows. A solution of 100 g. of potassium hydroxide in 100 c.c. of water is saturated with sulphur dioxide. An equal amount of potassium hydroxide is then added and then an equimolecular amount of potassium bicarbonate. The solution is heated to 80° and thymoquinone added in small amounts, with shaking, until an excess is present as indicated by the presence of an oily layer on the surface. Sulphur dioxide is then passed in until partial decolorisation occurs and, on cooling at 0°, the compound crystallises out. Attempts to prepare the compound from potassium bisulphite were unsuccessful owing to reduction to thymoquinol. The author, in disagreement with Carstanjen's explanation, that the amounts of potassium hydroxide liberated by the repeated addition of equal quantities of thymoquinone to the potassium sulphite should be the same, finds that the amount of alkali used

in converting the bisulphite into the normal sulphite diminishes with each increase in thymoquinone added.

E. H. SHARPLES.

**Stabilisation of solution of arsenious and mercuric iodide, U.S.P. X [Donovan's solution].** W. J. HUSA and W. W. F. ENZ (J. Amer. Pharm. Assoc., 1930, 19, 1228—1230).—Donovan's solution, stabilised by various methods, after storage for 10 months in well-filled, amber-coloured bottles, showed the following percentage deterioration: control 12.4, authors' modified formula (cf. B., 1930, 585) <1, neutralised with potassium hydroxide 10.1, saturated with carbon dioxide 8.7, neutralised with potassium hydroxide and saturated with carbon dioxide 6.1, air replaced by hydrogen 12.5, neutralised with potassium hydroxide and air replaced by hydrogen 7.3, calcium carbonate added 6.0, terpin hydrate added 8.3. E. H. SHARPLES.

**Assay of mercurochrome "220."** G. W. MARRIS (Quart. J. Pharm., 1930, 3, 455—459).—Normal mercurochrome loses about 10% of its weight when dried at 110° and 15 mm. A series of commercial samples gave values for bromine (peroxide method) ranging from 18 to 21.3%, and for acetic acid 1—3%. A limit of 1% is suggested for the latter, and the mercury content should approximate to 23.5% (on the determinations; cf. White, A., 1921, i, 71).

R. CHILD.

**Tincture of capsicum.** C. F. POE, A. P. WYSS, and B. S. SLATOR (J. Amer. Pharm. Assoc., 1930, 19, 1188—1190).—Analyses of the tinctures prepared with different concentrations of alcohol and with different samples of capsicum are discussed from the point of view of the detection of adulteration.

E. H. SHARPLES.

**Capsicum tincture, B.P.** W. J. BEARDSLEY and F. J. BOLTON (Quart. J. Pharm., 1930, 3, 427—432).—To avoid the deposition of fatty matter from the tincture at low temperature it is suggested that the capsicum (7.5 g.) be macerated with 100 c.c. of 60% alcohol, percolated, and the marc pressed; to the resulting liquid is added one half its volume of 90% alcohol. R. CHILD.

**Further studies on the salvarsans and related compounds.** C. N. MYERS (J. Amer. Pharm. Assoc., 1930, 19, 1051—1056).—The lack of efficiency and greater need for uniformity of certain types of arsenicals, particularly neosaharsan, is emphasised. Examination of some types of the latter product has shown that some are essentially monosubstituted, other disubstituted; another group was approximately half and half, and another contained 81.6% of a sulpharsphenamine-like impurity. Similarly, many commercial samples of salvarsan contain comparatively large amounts of inert, non-arsenic containing material and some require large excesses of alkali to produce the disodium salt; this factor, coupled with the action of water that has absorbed much carbon dioxide, may be the cause of disastrous results clinically. The use of warm or hot water for the addition of the alkali leads to decomposition with formation of arsenoxide. E. H. SHARPLES.

**Leaf oils of Washington conifers. II. *Juniperus scopulorum*. III. *Pinus monticola*. IV. *Tsuga heterophylla*.** A. J. LEHMAN and E. V. LYNN (J. Amer.

Pharm. Assoc., 1930, 19, 1071—1075, 1185—1187, 1304—1307; cf. B., 1930, 1003).—II. Steam-distillation of the fresh leaves of *J. scopulorum* yielded 0.49% of a yellowish-green oil ( $d_{20}^{25}$  0.966 [?],  $[\alpha]_D^{25}$  +41.18°,  $n_D^{20}$  1.4856, acid value 1.52, saponif. value 11.29, ester [as bornyl acetate] 3.41%) having a balsamic odour and containing  $\alpha$ -pinene, camphene (?),  $\beta$ -pinene, terpinene, terpineol (partly as butyrate or acetate), azulene, a phenol, and unidentified oxygenated compounds.

III. Steam-distillation of the leaves of *P. monticola* yields 0.053% of oil [ $d_{20}^{25}$  0.8695,  $n_D^{20}$  1.4724,  $[\alpha]_D^{25}$  —17.31°, acid value 4.24, saponif. value 26.78, esters (as bornyl acetate) 7.88%] containing  $\alpha$ - and  $\beta$ -pinene,  $\beta$ -phellandrene, probably camphene (total terpenes about 80%), borneol (as butyrate?), azulene, sesquiterpenes (6%), and a compound giving a lactone on oxidation.

IV. The oils distilled from the air-dried leaves (0.12%) and fresh leaves and twigs (0.09%) of *T. heterophylla* have, respectively:  $d_{20}^{25}$  0.9499, 0.8741;  $n_D^{20}$  1.4935, 1.4804;  $[\alpha]_D^{25}$  —, —17.33°; acid value 45.74, 3.11; saponif. value 78.51, 16.51; ester as bornyl acetate 11.49%, 4.69%; alcohol as ester 9.01%, 3.68%. A mixture of the two oils contained 65% of terpenes ( $\alpha$ - and  $\beta$ -pinene,  $\beta$ -phellandrene, camphene), cadinene, benzoic acid (free and as ester) and a sesquiterpene, *picene*, which has been found also in the oil from *Picea sitchensis*.

E. H. SHARPLES.

**Essential oil from the fruit of *Ferula Badra Kema*.** B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem. Pharm. Inst., Moscow, 1930, No. 22, 72—79).—The oil of *Ferula galbaniflua* had  $d_{20}^{25}$  0.8736,  $\alpha_D$  +16.16°,  $n_D^{20}$  1.4765, acid value 1.2, ester value 19.12, ester value after acetylation 31.78; it consists chiefly of hydrocarbons. *d*- $\beta$ -Pinene, *d*-limonene, ketones (1%), and an alcohol,  $d_{20}^{25}$  0.9801,  $\alpha_D$  +1.2°,  $n_D^{20}$  1.4935, were present; camphene was not found.

CHEMICAL ABSTRACTS.

**Essential oil of *Laserpitium hispidum*, MB.** B. N. RUTOVSKI and N. N. MAKAROVA-SEMLIANSKA (Trans. Sci. Chem. Pharm. Inst., Moscow, 1930, No. 22, 85—94).—The oil (0.18—0.77%) had  $d_{20}^{25}$  0.8442—0.8781,  $[\alpha]_D$  +17.05° to —3.8°,  $n_D^{20}$  1.4682—1.4786, acid value 0.92—5.27, ester value 18.66—142.16, ester value after acetylation 17.14—151.8, alcohol content 2.43—9.28% (free), 5.13—39.12% (combined). Geraniol (40—42%) is the most valuable constituent of the oil from the fruits; citronello. was not found.

CHEMICAL ABSTRACTS.

**Oil and camphor content of the leaves of *Laurus camphora*.** B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem. Pharm. Inst., Moscow, 1930, No. 22, 126—129).—The finely-powdered leaves are distilled with steam and the distillate is extracted with ether, the camphor content being calculated from the m.p., f.p., and  $[\alpha]_D$  of the residue. Results are tabulated.

CHEMICAL ABSTRACTS.

**[Characterisation of] ethereal oils.** K. BODENDORF (Arch. Pharm., 1930, 268, 486—491).—The reactions of anethole, isoeugenol, geraniol, eugenol, geranyl acetate, citral, and citronellal with 0.3*N*-perbenzoic

acid in chloroform at 20° have been followed. In the first three cases the reaction is completed within a few hours, but in the others it proceeds much more slowly. In view of the simplicity of the method, an attempt has been made to use the oxygen number (cf. Prileschaeff, A., 1911, i, 255, 604) for the characterisation of ethereal oils. The oil is dissolved in 25 c.c. of *N*-perbenzoic acid and the excess of the latter determined after 18 and 24 hrs. A satisfactory end-point was attained with seven oils, but not with oil of cinnamon.

H. E. F. NOTTON.

**Dispersion of the optical rotation of essential oils.** L. A. TSCHUGAEV and Y. Z. CHESNO (Trans. Sci. Chem. Pharm. Inst., Moscow, 1928, No. 19, 181—185).—The dispersion coefficient  $af/\alpha_c$  ( $c=656$ ;  $f\mu\mu=486\mu\mu$ ) is for a large number of substances about 1.95, but shows considerable deviations. A number of ketones give high values (2.05—3.50). The constant serves for the characterisation of individual compounds and naturally occurring mixtures. Data for 31 oils are tabulated.

CHEMICAL ABSTRACTS.

**Caution in working with ether.** NOLTE. See III. Tobacco. KOENIG; HUTCHESON and COPLEY.—See XVI.

See also A., Feb., 187, **Determination of mercury in mercuric cyanide** (CATELAIN). 190, **Continuous extraction apparatus** (SELF and CORFIELD). 195, **Automatic distillation of ether, alcohol, chloroform, etc.** (LAPP). 218, **Organic compounds of selenium** (KEIMATSU and YOKOTA). 235, **Synthesis of contrast media for pyelography** (SUGII and others). **Derivatives of 4-phenyl-2-methylquinoline** (BRAHMACHARI and BHATTACHARYA). 239, **1-Hydroxy- and 1-amino-derivatives of theobromine** (ROJAHNS and FEGELER). 242, **Alkaloid from *Anona reticulata* (SANTOS)**. **Alkaloids of *Berberis Thumbergii*** (KONDO and TOMITA). ***Strychnos* alkaloids** (LEUCHS). 243, **Oxonitin** (SPÄTH and GALINOVSKY). **M.p. of pure yohimbine** (WARNAT). **Alkaloids of *Sinomeninium* and *Cocculus*** (KONDO and others). **Identification of alkaloids** (FULTON). **Detection of alkaloids in plants: cytosine** (KLEIN and FARKASS). 246, **Microsublimation** (CHALMETA). **Determination of quinine alkaloids** (PRIDEAUX and WINFIELD). 252, **Trypsin preparations for prevention of adhesions** (WALTON). 259, **Methylglyoxaline derivatives** (SUPNIEWSKI). 260, **Antipyretic action of 3-aminohydrocarbo-styryl, its isomerides and derivatives** (WATANABE). **Isotonic soap for injection** (PICOU). **Local anæsthetics from piperidine** (ROSE and others). 266, **Purification of tuberculin** (MASCHMANN and KÜSTER). 267, **Purification and concentration of diphtheria toxin and anatoxin** (SCHMIDT and others). **Purification of Shiga and typhoid phages** (HOSOYA and others). **Bactericidal effect of ether and chloroform vapours** (YASUDA). **Terpenes and essential oils as disinfectants** (DE; SUBRAHMANYAN). 268, **Hormone of pituitary gland** (VAN DYKE and WALLEN-LAWRENCE). 269, **Hormone of anterior pituitary lobe** (BIEBL). **Female sex hormone: assay and preparation** (D'AMOUR and GUSTAVSON). 274, **Alkaloids from**

***Sida cordifolia*** (GHOSH and DUTT). **Essential oil of bark of *Magnolia obovata*** (SUGII and SHINDO).

PATENTS.

**Curing of tobacco.** W. A. PIKE, H. M. PIKE, and W. T. HENRY (U.S.P. 1,768,142, 24.6.30. Appl., 7.11.27).—The newly-cut tobacco leaves are subjected to hot fumes containing creosote, lignin, and acetic acid, preferably obtained from the hard woods, so that the fumes completely penetrate into the sap before it is evaporated by the heat. E. II. SHARPLES.

**Cyto-vaccine of tubercle bacilli.** J. S. KOMINZ (U.S.P. 1,766,131, 24.6.30. Appl., 29.10.23).—A suspension of live tubercle bacilli in a sterile normal saline solution is subjected to a treatment to kill the bacilli, the mixture is injected into a healthy mammal, and the fluctuating mass which is developed is evacuated to obtain a liquid containing the defensive anti-bodies and partly digested tubercle bacilli. This liquid is treated mechanically and/or chemically to break down capsular structures to render its absorption complete into another mammal in which it may be injected. It may be treated with ether and triturated and, after elimination of ether, the residual dry mass triturated again and then mixed with a solution of sodium chloride to produce an emulsion. E. II. SHARPLES.

**Manufacture of [mercury] products having a sterilising action on bacteria and/or fungi.** T. W. F. CLARK (B.P. 338,963, 29.8.29).—Phenolphthalein or fluorescein or their homologues or halogen derivatives are treated in solution and at raised temperature with mercury oxycyanide or with a mixture of alkali cyanide and mercury oxide. C. RANKEN.

**Basic product from oleic acid** (B.P. 317,325).—See III. **Castor oil soap** (U.S.P. 1,767,041).—See XII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Some properties of fine-grain developers for motion picture film.** H. C. CARLTON and J. I. CRABTREE (Kodak Res. Lab., Abridged Sci. Publ., 1929, 13, 191—204).—Borax developer gives more emulsion speed for longer development times; a maximum gamma of 1.4 is obtainable. The effects of varying the concentration of the constituents of the developer are described and tabulated. Borax developer becomes exhausted more rapidly than other developers, but can be revived by adding half the original quantities of elon, quinol, and borax together with a little sodium sulphite. The developer cannot be used for more than 160 ft. of film per gal. The sludge of silver which forms does not interfere with successful development. Increasing sulphite content decreases graininess, but too much is deleterious. 1 G. per litre of hypo produces very fine-grained images. Extensive tests indicate that borax developer does not produce finer-grained images than does the standard formula (D. 16), but is preferable where a low degree of development is required. Eastman motion-picture panchromatic film (type 2) was used throughout.

J. LEWKOWITSCH.



Variation of the threshold speed of an emulsion according to the developer and conditions of development. III. E. R. BULLOCK (Kodak Res. Lab., Abridged Sci. Publ., 1929, 13, 7—10).—The threshold speed of an emulsion passes through a maximum with increasing times of development. The "maximum threshold speed" varies with the composition of the developer. The time of development, for a given emulsion and developer, required to obtain the maximum threshold speed is called the "optimum development time." The treatment of an emulsion with dilute silver nitrate solution between exposure and development (cf. Wightman and Quirk, B., 1927, 268) was investigated, and it was found that (1) the maximum threshold speed and optimum development time are increased; (2) drying and keeping of the emulsion after treatment causes continuously increasing latent fog; (3) short treatment (e.g., 0.02%  $\text{AgNO}_3$  for 5 min.) has a distinct anti-fogging effect; (4) if a sulphite-free alkaline developer or ferrous oxalate is used, immediate fogging occurs, but may be obviated by washing in water, or bathing in very dilute potassium bromide solution, before development. This latter treatment does not alter effects (1) and (3). J. LEWKOWITSCH.

Chemistry of selenium toning. A. SEYEWETZ (Brit. J. Phot., 1930, 77, 718—719).—Silver images toned to completion with (a) sodium selenosulphate in excess of sodium sulphite and (b) sodium selenosulphide showed an almost quantitative conversion into silver selenide. The mechanism of the chemical reactions is suggested. J. W. GLASSETT.

See also A., Feb., 180, New photochemical effect on "celloidin" paper (MEWES).

#### PATENTS.

Production of photographic silver-salt emulsions. G. SCHWARZ (B.P. 339,769, 22.11.29).—An inner compartment containing a solution of a colloid is separated by collodion or chromated gelatin diaphragms, on one side from a silver salt solution containing the anode, and on the other from a solution of a halide compound containing the cathode. By electrolysis a silver halide emulsion free from the salts of double decomposition is formed in the inner compartment. Details of the apparatus are given. J. W. GLASSETT.

Dye-carrying layer for photographic films and the like. S. E. SHEPPARD and J. G. McNALLY (U.S.P. 1,768,795, 1.7.30. Appl., 1.3.29).—The rear side of the emulsion support is coated with a layer of polyvinyl alcohol containing an anti-halation dye which will act as a desensitiser in solution. J. W. GLASSETT.

Manufacture of sensitised bases for photographic purposes. H. D. MURRAY, D. A. SPENCER, and COLOUR PHOTOGRAPHS (BRIT. & FOREIGN), LTD. (B.P. 337,868, 24.8.29).—A water-impermeable support, e.g., nitro-cellulose, cellulose acetate, or "celastoid," is superficially de-esterified to render the surface water-absorbent, and is then impregnated with a light-sensitive diazon-

ium salt or a silver or iron compound. Suitable de-esterifying agents are suggested for each type of support. J. W. GLASSETT.

Printing of colour photographs. AMIRA TRUST (B.P. 340,044, 26.9.29. Ger., 1.10.28).—Auxiliary dyes, which are readily removed after exposure, are added to the coloured positive films in order to equalise the exposures required by each particular film under its corresponding colour-separation negative.

J. W. GLASSETT.

Production of photographs in colour. W. W. TRIGGS. From MULTICOLOR FILMS, INC. (B.P. 339,323, 4.6.29).—Two colour-separation negatives, taken under green-blue and orange-red filters, are printed simultaneously in register upon opposite sides of a double-coated film, which is developed, fixed, washed, and passed through an iron toning bath so that one side only, i.e., the orange-red filter positive, is toned blue. The film is then totally immersed in a solution of a basic red dye (a mixture of basic magenta and auramine, which is not mordanted by the iron bath) and finally passed through a uranium mordanting and toning bath, which fixes the orange-red image. The film is then fixed, washed, and dried. Suitable iron toning and uranium mordanting baths are described.

J. W. GLASSETT.

Photography of X-ray images. K. SCHINZEL (B.P. 339,996, 9.7.29. Austr., 9.7.28).

[White-light prism device for] colour photography and colour cinematography. S. PRISNER (B.P. 341,078, 7.10.29).

#### XXII.—EXPLOSIVES; MATCHES.

See A., Feb., 203, Nitration of cellulose (BERL and RUEFF). 211, Preparation of tetryl (DESVERGNES).

#### XXIII.—SANITATION; WATER PURIFICATION.

Densities of mixtures of air and various fumigants. R. C. ROARK and O. A. NELSON (J. Econ. Entom., 1930, 23, 985—987).—The density of air saturated with a number of common fumigants is recorded.

A. G. POLLARD.

Chlorine efficiency [in water purification]. ANON. (Public Works, 1930, 61, 26—28).—Data have been collected and certain recommendations have been made in a report of the Committee on Water Purification and Treatment of the Conference of State Sanitary Engineers.

CHEMICAL ABSTRACTS.

See also A., Feb., 186, Determination of carbon dioxide in sea-water (NICLOUX). 259, Toxicity of ethylene chlorohydrin (PRATT). 267, Terpenes and essential oils as disinfectants (DE; SUBRAHMANYAN).

#### PATENTS.

Purification of liquids (B.P. 339,673). Portable ozoniser (U.S.P. 1,768,891).—See XI.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAR. 27 and APRIL 3, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Adsorption intensity and its technical importance.** J. TRAUBE (Z. angew. Chem., 1931, 44, 73—75).—It has long been known that thermal, mechanical, or electrical energy is an expression of the multiple of capacity and intensity factors. This is equally true of energy quantities in dissolution and adsorption processes. Methods which have been investigated by the author and his pupils for the measurement of adsorption intensity are as follows. The heat of adsorption of 1 g. by a sufficient quantity of the adsorbent may be measured. This often depends on the adsorption intensity, but in other cases also on the capacity, as expressed by adsorbing surface and molecular volume of the adsorbed substance. Oils examined in this way fall into two classes, viz., those of low and of high adsorption heats. Another method depends on the displacement principle, using different liquids. The adsorption intensity of different vapours to activated carbon may be measured comparatively by passing a constant current of air over saturated carbon and determining the rate of removal. The question of "wettability" is important in flotation technique and lubrication. Lubrication efficiency depends on both viscosity and surface tension, and here also the adsorption intensity depends on the polarity of the oil. C. IRWIN.

**Heating of liquids in wooden vessels.** W. GRAULICH (Farben-Chem., 1930, 1, 16—20).—An example is given of the calculation of the thermal balances in the heating of water in a wooden vessel from 15° to 95° by steam at 2 atm. and at 8 atm. Heating surfaces and radiation losses are calculated and the significance in the latter connexion of surfaces of hot liquid open to the atmosphere is pointed out. Approximate formulæ for heat-transmission coefficients are given and steam velocities in pipes for a given pressure and throughput are tabulated. The advantage of using steam at the higher pressure is shown to be negligible, provided that large enough steam pipes are used. C. IRWIN.

**Determination of viscosity.** SCHLENKER. **Measurement of flow of gas.** KING and WILLIAMS.—See II. **Silver and chemical plant.** McDONALD.—See X. **Determination of moisture in chemicals.** PRITZKER and JUNGKUNZ.—See XX. **Phosphates in boiler water.** SCARRITT.—See XXIII.

#### PATENTS,

**Furnaces.** BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 340,780, 2.1.30).—The furnace is adapted to heating fluids by means of gases at not too high a temperature. Combustion takes place under the

best conditions, viz., with preheated undiluted air, but after combustion some of the flue gases are returned and the mixture is passed over the fluid-heating device. Only that proportion of the flue gases that is not returned is sent through the air preheater. B. M. VENABLES.

**Furnaces.** A. SMALLWOOD and J. FALLON (B.P. 340,710, 13.11.29. Addn. to B.P. 312,391; B., 1929, 625).—A recuperator for use underneath the furnace claimed in the prior patent is described. It comprises vertical tubes through which the air passes upwards and the gases pass horizontally around. B. M. VENABLES.

**Rotary-hearth furnace.** O. A. COLBY, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING Co. (U.S.P. 1,770,970, 22.7.30. Appl., 7.12.28).—A rotary annular furnace of which the hearth is formed of tilting trays is provided with means to jar the trays when they are tilted to discharge the goods. B. M. VENABLES.

**Heat-treating furnace.** P. W. MCCOY and J. A. MASURY, Assr. to SURFACE COMBUSTION Co., INC. (U.S.P. 1,770,081, 8.7.30. Appl., 21.12.28).—The material to be treated, e.g., small metallic articles, is placed on a shoot inclined across the furnace at an angle rather less than the angle of repose; it is supported on cushioned rests and the articles are caused to travel by rapping the shoot and are discharged down a vertical passage within the furnace. B. M. VENABLES.

**Tunnel kilns.** WOODALL-DUCKHAM (1920), and A. McD. DUCKHAM (B.P. 340,271, 19.7.29).—In a tunnel kiln (preferably annular) having continuous motion of the vehicles and therefore permanently open ends, ports are provided some distance from the inlet and outlet ends which lead to longitudinal flues in the walls and/or roof of the kiln leading to the chimney and combustion zone, respectively. Control dampers are provided and a booster fan may be connected across a partition in the flue which carries the air from the outlet end of the kiln. B. M. VENABLES.

**Heating apparatus for water or other liquids.** GEN. ELECTRIC Co., LTD., and O. W. HUMPHREYS (B.P. 342,073, 11.12.29).—In heating apparatus in which cold liquid is fed under pressure through a valve-controlled inlet pipe into a storage tank where it is heated, an expansion chamber, arranged outside the tank, communicates with the outlet pipe and is adapted to receive and retain any liquid displaced from the tank owing to expansion after the inlet pipe has been closed. J. S. G. THOMAS.

**Heating of decomposable liquids.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 341,383, 25.10.29).—Another form of rotating electric heater which produces

\* The remainder of this set of Abstracts will appear in next week's issue.

little disturbance of the liquid is described. (Cf. B.P. 333,241; B., 1930, 970.) B. M. VENABLES.

**Heat exchangers.** SOC. ANON. DES USINES J. GALLAY, and F. GALLAY (B.P. 341,247, 28.12.29).—A heat exchanger such as a honeycomb radiator is constructed of pairs of corrugated strips between which are other strips having bridge-like projections in the air spaces, inclined at angles which increase in the direction of flow of the air. B. M. VENABLES.

**Tubular heat exchangers.** W. SILLER (B.P. 340,893, 31.3.30).—An exchanger of the bundle-of-tubes type suitable for high pressures has the tubes attached to the tube plate and the latter to the casing by welding. The thickness of the tube plates must not differ by more than 2 mm. from that of the tubes, and the distance between adjacent tubes is similarly limited; it is preferable to eliminate the "islands" between tubes by giving the latter a non-circular, *e.g.*, hexagonal, section. B. M. VENABLES.

**Finned or gilled tubes for heat exchangers.** J. E. GORTNER (B.P. 340,422, 23.1.30).—A tube of flattened section (opposite long sides being straight) is provided with a continuous, roughly spiral gill comprising an L-shaped strip, the flange of which is cut by T-shaped slits at regular intervals, so that it may be bent round the short sides of the tube. The web, which lies in contact with the tube, may be formed by bending the strip back on itself or round a reinforcing wire. B. M. VENABLES.

**Heat-exchanging apparatus.** HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 340,765, 20.12.29).—Fins of heat-exchanger tubes which are formed from discs or spirally wound strip have their outer edges slit and twisted. B. M. VENABLES.

**Heat-interchanging apparatus.** D. G. BRANDT, Assr. to DOHERTY RES. CO. (U.S.P. 1,770,375, 15.7.30. Appl., 6.2.28).—An exchanger composed of a number of W-shaped tubular elements assembled together to form a number of superposed circular elements is described. B. M. VENABLES.

**Heat-transmitting apparatus.** E. MORTERUD (U.S.P. 1,770,320, 8.7.30. Appl., 16.6.27. Norw., 25.6.26).—An evaporator or condenser is constructed of a number of vertical double-concentric or Field tubes with the closed ends uppermost, so that the steam or vapour passes downwards while condensing; to maintain the velocity the annular space is caused to decrease in size downwards, preferably by tapering the inner tubes. At least one of the Field tubes is connected in series with the remainder, to discharge permanent gases from the intermediate condensate chamber. In addition to this discharge the bulk of the vapours may be circulated back from the condensate chamber to mix with the supply of new steam. B. M. VENABLES.

**Drying plant.** N. PANZIREFF (B.P. 341,192, 19.11.29).—The goods are conveyed on a vertical zig-zag conveyor through a casing to which heated air is admitted. The first few and last few of the conveyor runs are divided off by partitions between each, forming a sort of seal for the air and causing it to travel counter-current to the goods at exit and entry, but in the inter-

mediate larger zone the relation is alternately con- and counter-current. B. M. VENABLES.

**Drying of material.** F. P. RENNEBURG, Assr. to E. RENNEBURG & SONS CO. (U.S.P. 1,771,141, 22.7.30. Appl., 21.2.25).—The drying is effected in two stages, through which the material passes in series and the medium in parallel; though separate streams of the drying medium are used, they may be derived from and discharge to a common furnace and stack. The apparatus described comprises a double-concentric rotating cylinder through which the material passes in the same direction in both stages, the intermediate return being effected by a longitudinal conveyor, which also affords opportunity for escape of vapours. B. M. VENABLES.

**Drying of chemical and similar products.** "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND. (B.P. 341,140, 22.10.29. Ger., 25.10.28).—The apparatus comprises a twin-concentric cylinder dryer in the outer zone of which hot gases pass concurrent and out of contact with the material, *e.g.*, lithopone, being dried; in the inner zone another supply of drying gases, preferably of an inert nature, pass countercurrent to and in contact with the goods. Preferably the inner zone starts at the feed end as a single circular section of small diameter, expands rapidly to the maximum diameter, and then becomes subdivided into a convenient number, *e.g.*, seven, of smaller cylinders. The countercurrent gases on leaving are preferably scrubbed free from dust by the moist material entering or by water. B. M. VENABLES.

**Drying apparatus [for plastic material].** J. J. and J. B. BERRIGAN, Assrs. to K. P. MALON (U.S.P. 1,770,727, 15.7.30. Appl., 9.5.28).—Plastic material, such as clayey substances or sewage press-cake, is dried and reduced to powder by passing it in thin layers over highly heated surfaces (red hot in the early stages), and subjecting it to a reciprocating rubbing action by other parallel surfaces; a drying medium such as air may also be injected. An apparatus described comprises a trunk piston for feeding the material, from which depend a number of sheets alternating with fixed heavier partitions, the whole bundle being heated by fire gases in a suitable casing. B. M. VENABLES.

**Drying of moist substances.** GUTEHOFFNUNGS-HÜTTE OBERHAUSEN A.-G. (B.P. 340,057, 8.10.29. Ger., 8.10.28).—The substance is inserted as a resistance in an electric circuit carrying currents of very high frequency, *e.g.*, "Tesla" currents; even "non-conductors" will pass sufficient current at suitable voltage and frequency for considerable heating to take place. The drying may be assisted by known means, *e.g.*, removal of expelled moisture by a current of drying gas. B. M. VENABLES.

**Drying or conditioning of articles.** BRIT. UNITED SHOE MACHINERY CO., LTD., and C. J. SMITH (B.P. 340,321, 28.9.29).—A dryer of the type comprising a permeable conveyor is provided with means for producing a drying atmosphere at 60° and 50% humidity, the drying medium (air) being supplied by and exhausted from similar ducts on opposite sides of the conveyor. B. M. VENABLES.

**Doll-head bearings for drying cylinders and such**

like. J. W. W. EVANS (B.P. 341,260, 10.1.30).—A form of steamtight truunion is described. B. M. VENABLES.

**Cooling towers.** K. W. BRANCZIK (B.P. 340,127, 16.12.29).—A method of construction of a Venturi-shaped tower is described. A vertical section exhibits straight lines only. B. M. VENABLES.

**Furnace-cooling systems.** B. J. MULLÉN (B.P. 339,972, 17.9.29. U.S., 19.12.28).—A furnace, *e.g.*, a blast furnace, having water-cooled parts from which leakage might take place into the furnace, has the pressure of the gas in the furnace maintained normally above the pressure of the water; any reduction in flow of the latter or admission of gas through a leak into the water system is indicated by a device comprising a float suspended in a gastight vessel the lower part of which is connected to a water-pipe, preferably the outlet pipe of the cooler. B. M. VENABLES.

**Dehydrating apparatus.** S. L. AMES and H. LAUGHLIN, ASSRS. to CHEM-ELECTRIC ENG. CORP. (U.S.P. 1,770,120, 8.7.30. Appl., 18.7.23).—A spray-dryer for material which is liable to stick on the walls is constructed in conical form and downward movement is given to both the material and gases, the dried material being continuously and quickly removed by a conveyor at the bottom, and the gas through a ring of ports above the bottom. A ring of windows with adjustable apertures is also provided at an intermediate height. B. M. VENABLES.

**Thermostats.** J. H. GRAYSON (B.P. 340,781, 2.1.30).—The device described in B.P. 327,775 (B., 1930, 591) is modified by the addition of a pair of thin resilient levers in substitution for, or in addition to, the thin disc, according to whether a gradual regulation or snap action is desired. B. M. VENABLES.

**Grinding mill.** C. O. BARTLEY (U.S.P. 1,772,026, 5.8.30. Appl., 14.2.27. Renewed 22.10.29).—A method of attaching the lining to a cylindrical mill is described, in which longitudinal channels are formed outwardly on the interior of the shell and the lining sections have flanges which are wedged into the channels. B. M. VENABLES.

**High-speed screen mill.** J. CRITES, ASSR. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,771,163, 22.7.30. Appl., 6.6.29).—Feeding means for a disintegrator are described. B. M. VENABLES.

**Pulveriser.** G. H. KAEMMERLING, ASSR. to ERIE CITY IRON WORKS (U.S.P. 1,772,048, 5.8.30. Appl., 16.7.29).—The apparatus comprises a number of disintegrating chambers and a fan chamber arranged in succession lengthways of the shaft. The feed and transfer passages are near the shaft and the last passage, *i.e.*, that to the fan, is further obstructed by a sleeve. B. M. VENABLES.

**Pulverising machine.** W. J. ARMSTRONG, ASSR. to JEFFREY MANUFG. CO. (U.S.P. 1,770,382, 15.7.30. Appl., 28.4.27. Renewed 3.10.29).—The crusher plate of a disintegrator is supported in such a way that it will yield to the impact of uncrushable pieces. B. M. VENABLES.

**Manufacture of materials in granular form.** IMPERIAL CHEM. INDUSTRIES, LTD. From GRASSELLI

CHEM. CO. (B.P. 341,349, 4.4.30).—A substance which is solid at ordinary temperatures, *e.g.*, sodium bisulphate, is melted or rendered pasty and sprayed into a substantially stationary supply of air or other gas which is surrounded by swiftly moving currents of the same gas; the latter may be formed by injecting air tangentially to the walls of the chamber. B. M. VENABLES.

**Apparatus for handling and conveying loose solids by liquid flushing.** ASH CO. (LONDON), LTD. From J. ROME (B.P. 341,108, 2.10.29).—The solids are allowed to accumulate on two sets of superposed (staggered) inclined shelves and are sluiced away at intervals through a grid into a channel where the speed of fluid is maintained high. B. M. VENABLES.

**Classification of materials by elutriation.** L. ANDREWS (B.P. 340,027, 19.9.29).—Classification is effected by the combined action of gravity and vertical motion in a cylindro-conical vessel, the intermediate sized particles being caused to join the oversize that first drops out by internal conical baffles, and all sandy material being delivered together through a spigot at the bottom of the conical part of the vessel. The top of the vessel is closed and the overflow is drawn off through three siphon pipes, or only one if the proportion of water in the overflow is not important. The siphons comprise (1) a main outlet; (2) an auxiliary outlet leading back to a feeding device at a low level, the delivery of this siphon being controlled by a valve and/or admission of air at the top; and (3) a control siphon of small bore taking a sample from (1) and delivering back to the feed pump. Siphons (2) and (3) have rather longer legs than has (1) and in the latter (3) is inserted a bellows or other pressure-sensitive device which, being subjected to reduced internal pressure with increase in density of the sample of overflow pulp, controls the delivery of the return siphon (2) so that the proportion of water in outlet (1) is held constant irrespective of variations in the net feed. A stable condition of free-flowing pulp through the oversize spigot is maintained by teetering water supplied centrally near the point of the cone. The principles of operation are fully explained. B. M. VENABLES.

**Classification or separation of mixed materials by elutriation.** L. ANDREWS (B.P. 340,242, 19.9.29).—A classifier of the type in which the pulp flows horizontally, the coarser particles from which drop into hopper-shaped receptacles provided with spigots, is provided with an underflow pipe of comparatively small diameter connecting all the spigots (or all of a group); the end remote from the common outlet is connected to the same source of supply of liquid or pulp as the main feed, so that a slight upward current of water and fine solids may be produced through the spigots. The common discharge end of the pipe may be connected to a device effecting a further classification of the sands. B. M. VENABLES.

**Machine for disintegrating and sifting powdery masses.** S. SCHWALB (B.P. 340,812, 22.1.30).—The apparatus comprises a waltzing muller rubbing on the surface of a round screen below which is a receptacle such as a basin. The muller and its driving gear remain in one position, but the basin and screen are removable,

and the latter is lightly pressed against the muller by means of counterweights. B. M. VENABLES.

**Rotary mixing machine.** C. FORVILLE (B.P. 340,462, 25.4.30. Belg., 26.4.29).—Two cup-shaped vessels, concave to each other, are fixed to the same rotating shaft, the space between the rims being closed or opened by means of a cylindrical strip operated without stopping rotation by means of a striking gear. The feed is admitted through an annular port through one cup surrounding the shaft. B. M. VENABLES.

**Mixing of liquids or liquids with gases. Furthering chemical reactions between liquids and gases.** L. MELLERSH-JACKSON. From E. COHNITZ (B.P. 340,268 and 340,503, 22.6.29).—The fluids are forced through a large-surfaced body comprising a pipe filled with small gravel, glass beads, pumice stone, etc. Excessive flow along the wall of the pipe is prevented by circumferential corrugations. B. M. VENABLES.

**Carrying out mixing operations on liquid, pulverulent, granular, or pulpy masses.** G. H. SCHIEFERSTEIN (B.P. 314,097, 22.6.29. Ger., 22.6.28).—Mixing is effected by the reciprocation of a vessel which is divided by partitions parallel to the direction of motion, with the object of damping out objectionable turbulence and causing the pulp or liquid to be more rapidly accelerated. The partitions may be broadened at the base to prevent jamming. B. M. VENABLES.

**Preparation of filter elements particularly applicable for testing milk.** A. M. VAN DOORN (B.P. 340,879, 17.3.30).—Filter medium is glued to and/or sandwiched between strips of paper formed with holes to suit the filter machine. B. M. VENABLES.

**Filtering of suspended sediments (sludge).** C. PONTOPPIDAN (B.P. 341,176, 11.11.29).—The filter comprises a hollow permeable body of cylindrical or other shape that slides into and out of a pressure tank containing the prefilter through a yielding gland, so that a layer of cake formed on it will be dragged out of the tank, and, continuing its journey, may pass into another tank for washing or drying by fluids forced through it. A continuous type is described, the permeable body being in the form of a hollow conveyor belt. B. M. VENABLES.

**Filter aid.** V. VOORHEES, Assr. to STANDARD OIL Co. (U.S.P. 1,770,052, 8.7.30. Appl., 22.6.29).—The prefilter is cooled to below 0° and finely-divided ice is added as a filter aid, the process being suitable for the separation of paraffin wax from oil. B. M. VENABLES.

**Vacuum distillation.** P. SUBKOW, Assr. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,771,385, 22.7.30. Appl., 6.9.27).—A bubble tower is used for rectification under high vacuum by dividing it into stages (preferably between each bubble plate) by impervious diaphragms and connecting a separate vapour pump across each diaphragm. If the pull of the pumps is greater than the hydrostatic back-pressure of the bubble trays the vacuum will increase upwardly of the tower. B. M. VENABLES.

**Bubble towers.** (A) P. J. SWEENEY, (B) E. E. BARTELS, Assrs. to STANDARD OIL Co. (U.S.P.

1,770,221 and 1,770,725, [A] 8.7.30, [B] 15.7.30. Appl., [A] 13.5.27, [B] 28.9.27).—A form of trapped downflow is described. The liquid is taken from a point above the surface of a bubble plate at a distance from the axis of the tower, passes through an inverted siphon trap, and is delivered on the axis of the tower in a stream, dropping freely through the vapour space to the plate next below. In (B) the downflow is provided with a hole at the bottom of the trap, large enough to discharge any deposited solid matter, but too small to unseal the trap. B. M. VENABLES.

**Machines for production of emulsions.** F. C. RANDS and T. F. N. ALEXANDER (B.P. 341,193, 19.11.29).—A rotating disc runs between two fixed ones, the former being provided with ports and the latter with recesses; in the ports are inclined blades of alternate hand in successive outward rings, so that the material after entering near the axis passes from one side of the disc to the other several times and exhausts through a passage at the periphery. B. M. VENABLES.

**Centrifugal separators.** W. ZEJEZNIK (B.P. 341,298, 6.2.30).—The material, e.g., sugar, passes in succession over a number of conical separating drums, on each of which it has opportunity of parting with moisture and fine material. B. M. VENABLES.

**Centrifugal treatment of substances.** L. P. SHARPLES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,761,593, 3.6.30. Appl., 12.3.27).—One form of apparatus described comprises an imperforate bowl increasing in diameter towards the open end and divided into three compartments by transverse annular partitions, the central one of which is the main sedimentation chamber and has its outer wall lined with filter medium supported on wire gauze or the like, which permits filtrate to percolate through it to the end compartments. These latter serve for decanting, and later draining, part or all of the clear liquid. In the usual case of heavier solids, most of the clear liquid is preferably decanted from the main sedimentation compartment, but if the solids are lighter than the liquid all the latter must be passed through the filter medium. In any case, the solid is collected by sedimentation only until it has accumulated up to the capacity of the bowl; draining is then effected simply by moving the decantation pipe in the end compartment of larger diameter outwards as far as possible. Washing may be effected by inward or outward flow before or after draining, and a scraper is provided to remove solids. Various modifications in construction and use are claimed. B. M. VENABLES.

**Separating tanks [for emulsions or suspensions].** J. SCHULTE (B.P. 340,320, 27.9.29).—A settling tank is provided with a number of inclined, parallel, submerged plates, and the fluid material is fed through supply means disposed at points intermediate the ends of the plates, the position of which means may be adjusted vertically. B. M. VENABLES.

**Apparatus for the automatic discharge of liquids in accordance with their sp. gr.** IMPERIAL CHEM. INDUSTRIES, LTD., and J. S. B. FLEMING (B.P. 340,270, 18.7.29).—The apparatus comprises a balanced float

controlling a diverter which delivers the liquid into one of two conduits according to the sp. gr.

B. M. VENABLES.

**Separation of the constituents of gaseous mixtures [e.g., air].** C. C. VAN NUYS and J. L. SCHLITT, Assrs. to AIR REDUCTION Co., INC. (U.S.P. 1,771,197, 22.7.30. Appl., 23.10.26).—In the separation of air a primary rectifier is worked so as to produce pure gaseous nitrogen, the oxygen-rich liquid being run to an auxiliary rectifier at about the same pressure. Surplus vapour from an intermediate point of the first rectifier is transferred to the auxiliary column, partly direct for use as vaporising medium, and the other part after heating by interchange with incoming air, re-compression, and re-liquefaction, as reflux liquid. The rectified liquid from the auxiliary column is substantially pure oxygen and the vapour comprises nitrogen, all the argon, and a little oxygen, and is run to waste or utilised in any desired manner.

B. M. VENABLES.

**Separating dust, grit, and the like, and removing sulphurous and other gases, from flue or furnace gases.** O. B. JACOBSEN (B.P. 341,058, 6.9.29 and 21.2.30).—The gases are passed through a centrifugal separator of the turbo-blade type, and while issuing from the lower side of the group of blades are sprayed with water or other liquid. Water or steam may also be injected at an earlier stage.

B. M. VENABLES.

**Apparatus for treating flue gases.** P. J. ROBINSON (B.P. 341,109, 11.7.29).—Another form of apparatus, applicable to steamships, for carrying out the process described in B.P. 338,492 (B., 1931, 95) is described.

B. M. VENABLES.

**Treatment of gases and vapours and mixtures of the same with liquids.** S. G. WATSON, D. M. HENSHAW, and E.M.S. INDUSTRIAL PROCESSES, LTD. (B.P. 340,631, 8.10.29).—The apparatus, which is particularly suitable for the treatment of distillation products of coal etc., comprises a vessel in which the gas passes under and over baffles which are kept wet by sprays of liquid withdrawn from the bottom of the vessel. A settler for solid matter is provided in the return conduit to the circulating pump.

B. M. VENABLES.

**Liquid and gas contact apparatus.** F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,771,364, 22.7.30. Appl., 4.4.28).—A gas washer having fixed screens and a rotating disintegrator of the pin type is described.

B. M. VENABLES.

**Purification of smoke.** M. JACOB and J. CUTHBERTSON (B.P. 340,252, 23.9.29).—A device is described in which smoke may be collected or destroyed by chalk, lime, or coke after addition of steam or atomised water to the gases.

B. M. VENABLES.

**Gas washer.** H. A. STRAIN (U.S.P. 1,766,267, 24.6.30. Appl., 6.10.27).—Water or other liquid is sprayed by a rotating device at the top of a tower and falls in sheets upon and from horizontal ring splashers of which the cross-section is  $\Lambda$ ; the rings are superposed and decrease in diameter down the tower, so that the gas flows upwards and inwards through vertical sheets of water and finally through the fine spray produced by the distributing device.

B. M. VENABLES.

**Apparatus for the heat-treatment of gases and vapours.** A. S. RAMAGE (B.P. 340,277, 17.9.29).—A form of heater comprising horizontal tubes and vertical electric heaters disposed between vertical rows of tubes is described.

B. M. VENABLES.

**Gas- or fuel-storing material.** F. G. KEYES, Assr. to FRIGIDAIRE CORP. (U.S.P. 1,770,526, 15.7.30. Appl., 15.1.25).—A hard absorbent material for storing gas or liquids, e.g., ammonia, is composed of a halide of a metal of group II with, in addition, zinc oxychloride, alundum cement, and, if desired, sodium silicate. Methods of preparation are described.

B. M. VENABLES.

**Compression refrigerating apparatus.** SULZER FRÈRES SOC. ANON. (B.P. 342,188, 12.3.30. Switz., 4.7.29).

**Thermal insulator** (U.S.P. 1,770,663). **Cyclone apparatus** (U.S.P. 1,773,840). **Detecting foreign matter in fluids** (B.P. 341,086).—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Plastometer : an instrument for measuring the plastic properties of coal.** J. D. DAVIS (Ind. Eng. Chem. [Anal.], 1931, 3, 43—45).—An instrument is described which is designed to enable measurements to be made of the plasticity of coal within the plastic range and of the temperature limits of the plastic range. It consists of a cylindrical steel retort, 5 in. by  $1\frac{1}{2}$  in., mounted on a tubular axle and placed in the middle of a cylindrical furnace; the retort is capable of rotation at constant speed. Through the axle passes a shaft fitted with five blades which just clear the inside of the retort, whilst the outer end of the shaft carries a graduated brass disc and a pulley, to which are attached a chain and spiral spring. The plastic stage of the coal is indicated by the development of tension in the spring.

H. F. GILLBE.

**Petrographic study of bituminous coal briquettes.** H. BODE (Brennstoff-Chem., 1930, 11, 476—478; 1931, 12, 7—9).—Briquettes of a blend of "fat" and "lean" coals possessed much less resistance to abrasion than those made under the same conditions from a blend of "fat" and "forge" coals. Both coal blends had approximately the same sieve analysis. Petrographic examination showed that the lower strength of the former briquettes was due to the greater vitrain content of the lean coal; under the pressure used in manufacturing the briquettes, the more brittle vitrain particles readily broke down, leaving hollow spaces within the briquette, and thereby bringing about a decrease in strength. The presence of fusain in the coal affected the strength of the briquettes adversely, unless a correspondingly higher proportion of pitch was used. The pitch was absorbed into the pores of the larger particles of fusain; the very fine fusain (under 0.12 mm.) was harmless, and absorbed no more pitch than the same sized vitrain or durain.

A. B. MANNING.

**Nature of coal bitumens.** G. STADNIKOV and R. WÄHNER (Brennstoff-Chem., 1931, 12, 23—25. Cf. Stadnikov and Weizmann, B., 1929, 1002).—A number of Russian coals have been extracted successively with

(a) benzene-alcohol under the ordinary pressure (yielding bitumen-*A*), (b) benzene under increased pressure (bitumen-*B*), and (c) benzene-alcohol under the ordinary pressure after an intervening treatment with hydrochloric acid (bitumen-*C*). The yields of bitumen-*C* were in many cases greater than those of bitumen-*B*. The m.p., acid, saponification, and iodine values of the products have been determined. Bitumen-*B* appears to be a decomposition product of organic acids of high mol. wt., which exist in the coal in the form of salts insoluble in benzene-alcohol. Bitumen-*C* is derived from salts of similar acids, which, however, are stable at 250–260°, and are therefore not extracted by benzene under pressure. A. B. MANNING.

**Fleissner coal-drying process for lignite.** H. KLEIN (Braunkohle, 1930, 29, 1–10, 21–30; Chem. Zentr., 1930, ii, 172).—The amount of water evolved in the steaming period increases rapidly with rising steam temperature, in the release period it rises to a maximum, whilst in the aeration period it is always diminished. Most of the water is evolved in a short steaming period. The liberation of liquid water results from a change in the capillary surface of the colloidal coal substance. The increasing hydrophobia of the capillary surfaces is due to gradual conversion of humic acid into humin, with liberation of carbon dioxide. Associated phenomena, and the genetic relation of bituminous with brown coal, are discussed. A. A. ELDRIDGE.

**Benzene obtained by the hydrogenation of brown coal under high pressure but without the addition of tar.** J. VARGA and I. MAKRAY (Brennstoff-Chem., 1931, 12, 21–22).—The hydrogenation was carried out in a rotating autoclave, each charge consisting of 2200 g. of brown coal and 330 g. of iron oxide. The initial hydrogen pressure was 110 atm.; the autoclave was heated to 470° and allowed to remain at that temperature for 1–2 min. About 50% of the coal was converted into benzene-soluble products. The fraction boiling to 230° amounted to about 400 c.c. from each hydrogenation. A number of such fractions were combined and on analysis gave: bases 3.0%, phenol 1.58%, cresol and higher phenols 19.42%, unsaturated compounds 6.6%, benzene 0.7%, toluene 2.0%, xylene 2.65%, other aromatic hydrocarbons 6.13%, paraffins and hydroaromatic compounds 53.8%, loss 4.1%. The bases were principally of tertiary character. The product contained no naphthalene. A. B. MANNING.

**Sulphuric acid treatment of lignite distillates.** S. RUHEMANN (Z. angew. Chem., 1931, 44, 75–82).—The action of sulphuric acid of concentration from 60% to 100% on (a) benzene recovered from the gases produced in the carbonisation of lignite and (b) the distillates of producer-gas tar was studied. The acid and basic constituents were first removed and an attempt was made to separate oxygen-containing constituents with hydroferrocyanic acid. The oils were then fractionated and subjected to acid treatment. The acid of 60% concentration was found to dissolve sulphur and oxygen compounds in the form of sulphonium and oxonium salts, the sulphur originating from thiophen and sulphides. Only traces of alcohols and ketones were

found. Neutral or acid esters of olefines were also dissolved by the 60% acid. With increasing concentration of sulphuric acid the polymerising action of the latter intensifies and the sulphur content of the resins formed diminishes. The benzene was much more readily polymerised than the tar distillate. A number of hydrocarbons of the type  $C_nH_{2n-8}$  were identified in the products. With 96% acid aromatic hydrocarbons were sulphonated and the residue consisted of cyclic hydrocarbons both saturated and unsaturated. Analytical methods are described in detail. The polymerisation products were depolymerised by distillation over fuller's earth. Their mol. wts. were determined cryoscopically in benzene and other physical constants were obtained, but it was not possible to establish constitutional formulæ, which apparently must be of the ring type with 3 or 4 double linkings. They bore little resemblance to the polymerisation products of limonene and terpene. In one case the fuller's earth treatment yielded a hydrocarbon  $C_{24}H_{38}$  apparently formed by the condensation of an aromatic and a hydroaromatic hydrocarbon. C. IRWIN.

**Berginisation of coals and tars.** N. A. ORLOV, V. V. TISHCHENKO, and N. D. LICHATSHEV (J. Appl. Chem., Russia, 1930, 3, 699–719).—Pobedinski, Bobrikovski, Shchekinski, Balkashit, and Barsas coal were submitted to berginisation in presence or absence of catalysts; the composition of the products is tabulated. Results of the rehydrogenation of oils so obtained are also recorded. Phenols containing sulphur as impurity are hydrogenated more readily than pure phenols in presence of a molybdenum catalyst; the product contained 67% of cycloparaffins. Xylenol, *o*-cresol, and quinol were hydrogenated in the presence of molybdenum.

CHEMICAL ABSTRACTS.

**Coke "porosity pictures."** F. ROLL (Brennstoff-Chem., 1931, 12, 1–3. Cf. Zipperer and Lorenz, B., 1930, 974).—Coke structure has been studied by applying printers' ink to a carefully ground surface of the material and using this to produce a direct contact print on paper. The structures observed have been classified; they can be divided into two groups exhibiting continuous and discontinuous porosity respectively, and these groups can be subdivided according to the shape of the pores (round or elongated), the thickness of the walls, etc. A. B. MANNING.

**Measurement of a rapidly fluctuating flow of gas.** J. G. KING and B. H. WILLIAMS (Dept. Sci. Ind. Res., Fuel Res., 1930, Tech. Paper No. 27, 18 pp.).—The use of various methods of gas-volume measurement to the blue-gas and blow-gases formed in a water-gas plant have been investigated. A Pitot tube, in the case of blue-gas, was not satisfactory, but an orifice was successfully used. In a 12-in. main the orifice diameter was  $6\frac{3}{4}$  in.; 22 ft. of straight main preceded, and 12 ft. followed, the orifice in order to eliminate turbulence. Pressure measurements, given by a recording differential meter, were found to provide low results in comparison with a calibrated gas-holder, but an inclined pressure-gauge was then developed which was satisfactory. A difference of 0.5 in. water-pressure gave a displacement of 4 in. in this gauge, and movement of



the meniscus was recorded continuously by a photographic method on a moving strip of paper. The instrument was calibrated and showed a direct relation between displacement and pressure drop over the range explored; the total gas volume was determined by integration of an area marked on the chart and interpreted in the ordinary way. For the air supplied to the plant, a main  $1\frac{1}{2}$  in. in diam., with an orifice of  $9\frac{1}{2}$  in., was used; Pitot-tube measurements were taken for comparison, at varying distances from the centre of the main. The results agreed within about 1%, but the Pitot-tube method is less satisfactory as it necessitates preliminary exploration of the system, and where the duration of the air stream is short this is often difficult. As the deflexions obtained in the orifice-plate method are also larger, they are more likely to be accurate.

R. H. GRIFFITH.

**Bitumen from bituminous sands.** K. A. CLARK (Nature, 1931, 127, 199).—The  $p_H$  of bituminous sands varied from 2.5 to 6.4. Separation of the bitumen by washing with hot water is improved as the  $p_H$  approaches 6.4; sodium hydroxide and sodium silicate have been used for this purpose. A preliminary washing of a bituminous sand with cold water reduces the amount of alkali required and helps to remove clay, which appears to hinder separation.

L. S. THEOBALD.

**Rapid determination of benzol and phenol in ammoniacal and waste liquors.** W. MÜNZ (Brennstoff-Chem., 1931, 12, 3—4).—Benzol is determined by passing about 10 litres of the liquor, in 1 hr., up through a cylindrical tube packed with activated charcoal (100—150 g.). The charcoal is then allowed to drain and the benzol is driven off by a current of superheated steam, condensed, and measured. Phenol is determined by adding ammoniacal copper sulphate to 25 c.c. of the filtered liquor, in a 250-c.c. distilling flask, until the liquid over the precipitate remains blue. Then 5 c.c. of 40% sodium hydroxide are added and the whole is heated until free from ammonia. The flask is attached to a condenser and a current of carbon dioxide is passed through the solution until neutralisation is complete. The whole is then distilled to dryness in a gentle current of carbon dioxide and the phenol content of the distillate is determined by titration with bromate-bromide solution.

A. B. MANNING.

**Rapid determination of asphaltic precipitates by vacuum filtration.** P. WOOG and J. GIVAUDON (Bull. Soc. chim., 1930, [iv], 47, 1419—1420).—Neither sintered glass crucibles nor Gooch crucibles containing filter paper, cellulose, or mixtures of silica and cellulose are suitable for filtration of the asphaltic precipitate obtained by addition of light petroleum to oils. Talc, without any binding agent, permits the total separation of the asphalt, but adsorbs colouring matter from the solution, and the results are high. Rapid and efficient filtration is possible through a layer of powdered glass (0.058 mm. mesh) in a sintered glass crucible; the precipitate and filter pad are easily removed.

H. F. GILLBE.

**Natural gas.** V. A. SOKOLOV (Nef. Choz., 1930, 18, 793—796).—Natural gas from Surakhani, Grozni, and Dagestanskies Ogni contains, respectively, air 1.0, 1.0,

1.0; carbon dioxide 18.2, —, 7.5; methane 77.5, 51.8, 89.0; ethane 2.0, 10.7, 1.9; propane 0.8, 25.1, 0.6; butane 0.5, 8.8, 0.6%. Grozni gas contains 1.9% of hydrocarbons higher than butane.

CHEMICAL ABSTRACTS.

**Shirak crude oil.** V. N. KHUDAKOV (Nef. Choz., 1930, 18, 543—550).—The oil has  $d$  0.8074—0.885, flash point 0°, and contains gasoline ( $d$  0.7307) 15%, kerosene ( $d$  0.8074) 35%, and residue ( $d$  0.9023) 50%.

CHEMICAL ABSTRACTS.

**Sakhalin crude oils.** S. S. NAMETKIN and E. M. SHAKHNAZAROVA (Nef. Choz., 1930, 19, 290—291).—Crude oils from the Ekhabli district and Kadzussa (wells Nos. 2 and 3) are described.

CHEMICAL ABSTRACTS.

**Crude oil from gushers in Maikop.** M. A. BESTUZHEV and P. A. MALASHKIN (Nef. Choz., 1930, 18, 965—970).—Oil from the layer  $c$  contains paraffin, 37—38% of a fraction having b.p. below 200°, and 15—16% of kerosene ( $d$  0.837). Cracking with recycling gave 37% of gasoline, 48% of fuel oil, and 15% of gas.

CHEMICAL ABSTRACTS.

**Bright stocks from Grozni crude oil.** A. N. SAKHANOV, L. G. ZHERDEVA, and G. V. POLYANSKAIA (Nef. Choz., 1930, 18, 800—805).—The production of a bright stock from bottom oils of Grozni paraffinic fuel oil is described, and the characteristics of the oils are recorded.

CHEMICAL ABSTRACTS.

**Naphthenic acid content of Emba crude oil and distillates.** T. VUISTAVKINA (Nef. Choz., 1930, 18, 1000).—Crude oil contained 0.43—0.74% of acids; distillates contained 0.18—1.44%, of mol. wt. 218—466. The yield from sludge was lower. The composition of salted-out sludges is recorded.

CHEMICAL ABSTRACTS.

**Action of sulphuric acid on the lightest products obtained in vapour-phase cracking of crude oil.** M. B. MARCOVICH and V. V. FIGULEVSKI (Nef. Choz., 1930, 18, 627—640).—The "amylene" fraction, containing about 43% of dissolved gases, was treated with sulphuric acid at  $-45^\circ$ , the product being fractionally distilled and saponified; 66% or 75% acid dissolves more polymerides than 85% acid. From 12.8 to 25.5% of the gas was converted into alcohols.

CHEMICAL ABSTRACTS.

**Semi-cracking [of fuel oil].** K. P. LIKHUSHIN (Rep. Sci. Tech. Council Oil Ind., Baku [1929], 1930, I [Cracking], 45—60).—The feasibility of obtaining a fuel oil which can be easily pumped through pipe-lines over long distances in cold weather has been investigated. Sakhurani paraffin-base fuel oil when cracked in a Pintsch gas producer afforded 67.5% of fuel oil,  $d$  0.8779, cold test  $-15^\circ$  (with gasoline 9.68%, gas 10%, composition recorded), or when cracked in a coil at 500° gave a fuel oil,  $d$  0.878, cold test  $-5^\circ$  to  $-7^\circ$ .

CHEMICAL ABSTRACTS.

**Cracking and destructive hydrogenation of peat tar.** B. K. KLIMOV and V. A. LANIN (J. Appl. Chem., Russia, 1930, 3, 727—740).—Cracking of low-temperature carbonisation peat tar in a rotating autoclave afforded 30% of products having b.p. below 200° and 35% of coke. Admission into a bomb of insufficient

hydrogen for complete hydrogenation results in the production of coke even in presence of aluminium oxide or hydroxide, chromic, manganic, stannic, or cupric oxide. Zinc, ferrous, and particularly nickel and cobalt oxides are better hydrogenation catalysts, coke not being formed in presence of the last two. Hydrogenation at 100 atm. and 450° yielded 27% of gasoline, or 20% after treatment of the distillate with acid and alkali. A higher temperature or longer heating increased the yield of light products, gases, and coke. A kerosene fraction (37%) contained phenols 30, bases 5.4, and unsaturated compounds 14%. Repeated hydrogenation affords 48% of gasoline (35%, *d* 0.760, after treatment with acid and alkali).

CHEMICAL ABSTRACTS.

**Condensation of hydrocarbons.** B. K. TARASOV and N. V. POPOVA (*Neft. Choz.*, 1930, 18, 992—995).—A cracked gasoline of b.p. below 200° and one to which benzene (10%) was added were treated with 90% sulphuric acid at 0°, distilled, and the fractions treated with 98% sulphuric acid. These fractions were free from unsaturated and aromatic hydrocarbons, the latter being present only in the higher-fraction polymerides. In the presence of 60% of unsaturated hydrocarbons only one third is converted into acid-soluble compounds, the remainder undergoing further change.

CHEMICAL ABSTRACTS.

**Steam in rectification of petroleum products.** A. TREGUBOV (*Azerbaid. Neft. Choz.*, 1929, No. 12, 37—51).—Calculations are given. Saturated steam produces a very small temperature interval within the rectifying column; large amounts of petroleum vapours and reflux oil are carried over. In the presence of superheated steam, distillation proceeds under conditions comparable with those obtaining in the absence of water vapour.

CHEMICAL ABSTRACTS.

**Oxidisability of mineral oils.** B. G. TUICHININ and K. I. IVANOV (*Neft. Choz.*, 1930, 18, 979—991).—Baku gas oil, washed after treatment with anhydrous sulphuric acid, is oxidised in 3 hrs. at 150° by oxygen at 15 atm., giving an oil of saponif. value 104.3; when 6% oleum is used the value is 63. The oxidisability of the oil is increased by treatment with 50% alcohol. Treatment with sulphur dioxide affords an oil of saponif. value 112.6 after oxidation. Addition of potassium, sodium, lithium, iron, or manganese salts of naphthenic acids increases the oxidisability of an acid-treated oil.

CHEMICAL ABSTRACTS.

**Effect of treating [with fuller's earth etc.] on the oxidisability and oiliness of lubricating oils.** N. I. TSCHERNOSHUKOV and A. M. GUTZAIT (*Neft. Choz.*, 1930, 18, 806—817).—Emba spindle oil was most oily when treated with 15% of fuller's earth. The degree of wetting is independent of the acidity. Treatment with 5% of silica gel is equivalent to that with 18% of fuller's earth. Experiments on the formation of asphalt or hydroxy-acids by oxidation with oxygen, and on the production of stable oils, from Baku and Emba oils are described.

CHEMICAL ABSTRACTS.

**Effect of temperature on formation of fatty acids in oxidation of paraffin wax.** I. GUTT and A. PIOTKO (*Azerbaid. Neft. Choz.*, 1930, No. 9, 108—116).—Increase in the velocity of the air blown through the

heated wax or in the time of oxidation does not affect the yield of acids, but decreases that of acids soluble in light petroleum and increases that of hydroxy-acids; the mol. wt. of the products is decreased. The optimal temperature is 150°, the yield of hydroxy-acids being controlled by the time. Oxidation of the unsaponifiable residue does not yield hydroxy-acids.

CHEMICAL ABSTRACTS.

**Refining of shale oil with silica gel and bauxite.** B. SALADINI (*Industria chimica*, 1929, 1132—1137; *Chem. Zentr.*, 1930, ii, 172).—Both silica gel and bauxite are suitable as refiners and especially as sulphur absorbents. The sulphur content was reduced by 70% with the former and by 50% with the latter. The spent absorbents can be reactivated by heating at 200—500°.

L. S. THEOBALD.

**Corrosive action of naphthenic acids in Maikop petroleum.** A. VUIBOROVA (*Azerbaid. Neft. Choz.*, 1920, No. 9, 117—121).—Corrosion of the refinery equipment is due to naphthenic acids and not to sulphur compounds.

CHEMICAL ABSTRACTS.

**Determination of aromatic, unsaturated, and naphthene hydrocarbons in light oils and motor spirits.** A. B. MANNING and F. M. E. SHEPHERD (*Dept. Sci. Ind. Res., Fuel Res.*, 1930, *Tech. Paper No. 28*, 14 pp.).—Largely a report of work by Manning (*B.*, 1929, 546) already abstracted. The following observations appear to be new. In cases where no unsaturated compounds are present in the original oil, the absorption of aromatic substances can be carried out with 98% sulphuric acid containing 2—3% of silver sulphate; this method gives a result by direct weighing. Errors in the determinations arise from (i) incomplete oxidation of unsaturated compounds or their polymerides, (ii) partial attack of saturated hydrocarbons, and (iii) uncertainty as to the degree of nitration of different aromatic hydrocarbons. Methods for overcoming or evaluating these errors are described, and numerous results are given for artificial mixtures of known composition. Further investigation of residual saturated hydrocarbons, containing both cyclic and open-chain compounds, has been attempted. Measurements depending on critical dissolution points in aniline are considered unsatisfactory, and an alternative is proposed in which naphthenes are dehydrogenated by passage over heated palladium-black, the resulting aromatic hydrocarbons being determined as before. Loss of hydrogen, however, is not complete unless the open-chain hydrocarbons are also partly attacked. R. H. GRIFFITH.

**Petrol with a benzol value up to 71% by cracking at reduced temperature.** G. EGLOFF and E. F. NELSON (*Petroleum*, 1931, 27, 59—60).—Six different oils treated at 497—520° and under 210—350 lb. pressure yielded 53—78% of petrol (calc. on raw oil) of benzene value 54—71%. The method of treatment and the results of experiments are described.

H. E. BLAYDEN.

**Determination of hydrogen sulphide in [oil] refinery still gases.** A. R. SCHARNAGEL and A. W. TRUSTY (*Ind. Eng. Chem. [Anal.]*, 1931, 3, 29).—The iodometric method is unsatisfactory because of the reaction between the iodine and unsaturated hydrocar-

bons. In the rapid and accurate procedure recommended the gas is passed through two scrubbers containing 0.1N-lead nitrate, the second being merely an indicator to ensure complete absorption of the hydrogen sulphide in the first. After a known volume of the gas has been passed, an aliquot portion of the solution is titrated with standard ammonium molybdate solution.

H. F. GILLBE.

**Potentiometric determination of acidity in insulating oils.** R. N. EVANS and J. E. DAVENPORT (Ind. Eng. Chem. [Anal.], 1931, 3, 82–85).—Methyl alcohol should not be substituted for ethyl alcohol in the A.S.T.M. procedure for the determination of acidity in oils. Results of the determination of various acids dissolved in transformer oil by the standard, potentiometric, and alkali-blue methods show that considerable discrepancies arise if acids such as stearic or abietic are present, and that the two last-named methods yield the most concordant results. In the potentiometric method two peaks are obtained in the titration curve, and the position of the second varies with the amount of quinol used; the two tend to merge into one as the quantity of quinol is reduced. The silver-silver chloride electrode may be used to replace the agar-agar boundary. The potentiometric method, although relatively slow, is recommended for routine determinations of acidity, but a preliminary approximate determination should be made.

H. F. GILLBE.

**Determination of viscosity [of oils] at low temperatures.** E. SCHLENKER (Brennstoff-Chem., 1931, 12, 25–26).—A simple viscosimeter suitable for the determination of the viscosity of lubricating and other oils below 0° is described. A pipette, the lower part of which is drawn out to a capillary, dips into a vessel containing the oil and surrounded by an outer vessel containing a cooling mixture. The upper end of the pipette is connected to a suction vessel containing water and provided with an adjustable outflow tube. The time required for the oil to fill the pipette is measured. The apparatus is preferably so adjusted that the time required is 50 sec. with an oil of 7° viscosity (Engler).

A. B. MANNING.

**Dilution and viscosity [of oils].** J. TAUSZ and A. RABL (Petroleum, 1931, 27, 41–43).—The relation between the dilution of solutions of mineral and vegetable oils and of fatty acids in toluene and their viscosity has been studied. Both chemically pure and commercial products have been used.

F. J. WILKINS.

**Protective coatings on iron pipes.** BECK. **Corrosion of metals by liquid fuel.** USPENSKI and LADUIZHNIKOVA.—See X. **Saponification value of asphalt-drying oil mixtures.** DEMAREST and RIEMAN.—See XII. **Insecticides.** MARTIN. See XVI.

#### PATENTS.

**Furnaces for production of coke by burning coal.** T. KOMATSU (B.P. 338,435, 21.1.30. Jap., 6.2.29).—A receptacle in the form of a truck with a movable bottom can be placed below a furnace in such a manner that the bottom when raised supports the fuel bed thereof. As fresh coal is fed into the furnace the fuel bed is gradually lowered into the receptacle until the latter is full of

coke. A movable separator is then introduced into the combustion chamber and serves to support the fuel bed while the coke receptacle is removed, discharged, and replaced.

A. B. MANNING.

**Apparatus for drying and low-temperature carbonisation.** J. Y. JOINSON. From I. G. FARBEN-IND. A.-G. (B.P. 341,606, 8.11.29).—The carbonaceous material to be treated is made to flow in thin layers over several inclined surfaces vertically above each other. Motion of the material is assisted by a reciprocating motion of the surfaces. Hot gases pass over the material and under the surfaces, and carry with them the products of distillation. The layers are normally 5–20 mm. thick, but means are provided for regulating the thickness.

H. E. BLAYDEN.

**Retorts for distillation of coal, shale, peat, and other carbonaceous substances.** T. TOPPING and J. S. BLACK (B.P. 337,842, 27.3.30).—A retort of the vertical type is provided with a number of cross-flues communicating with the usual annular flue situated between the retort and its casing. A series of baffles in the annular flue causes the heating gases, which enter at the bottom, to pass through the cross-flues in succession. An annular flue also surrounds the lower part of the feeding hopper and serves to pre-dry the material therein. A depending sleeve, forming a continuation of the bottom of the hopper, ensures that the material entering the retort will not obstruct the gases and vapours leaving. Provision is made for introducing steam at the bottom of the retort, and for sealing the latter while discharging the coke from a container attached below the retort.

A. B. MANNING.

**Preparation of fuel from coal.** TRENT PROCESS CORP. (B.P. 340,337, 3.10.29. U.S., 20.10.28. Cf. B.P. 262,302; B., 1927, 100).—Powdered coal is mixed with water and a light oil, whereby its ash content is greatly reduced, and the light oil is then evaporated. The residual coal is carbonised and the coke or semi-coke obtained is pulverised, moistened with water, and moulded with a heavy oil into briquettes. After baking, the briquettes are coherent and free from oil or tar. If the coal under treatment is of a strongly coking type it may be preoxidised in order to diminish or destroy such qualities.

R. H. GRIFFITH.

**Combustion of carbonaceous substances.** A. L. MOND. From SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 338,444, 28.1.30).—Combustion is effected by means of a jet of oxygen, or air enriched with oxygen, which impinges on a restricted zone of the surface of a bed of the fuel, which is thereby burned completely to carbon dioxide with the production of a zone of very high temperature, the ash of the fuel being volatilised and converted into impalpable powder. The process may be applied to the volatilisation and conversion into impalpable powder of other refractory substances, e.g., silica, alumina, calcium phosphate, etc.

A. B. MANNING.

**Low-temperature distillation of carbonaceous materials.** A. S. RAMAGE (B.P. 340,276, 17.9.29).—Carbonaceous material, such as coal, is fed to a retort where it is heated by means of reducing gases made in an

associated water-gas generator. The sensible heat of the blow-gas is used by passing it round the outside of the retort, but the water-gas is withdrawn through the column of fresh fuel. The raw coal should be heated only to a relatively low temperature, so that the tar obtained is of a primary character; for this purpose means for agitating the coal charge may usefully be included.

R. H. GRIFFITH.

**Destructive hydrogenation.** C. F. R. HARRISON, E. D. KAMM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 338,576, 16.7.29).—Coal, oils, etc. are hydrogenated in two stages. The mixture of middle oil and light oil vapours obtained in the first stage, *e.g.*, by the process described in B.P. 336,610 (B., 1931, 144), is passed to a separator wherein the middle oils are isolated in the liquid state. These are then revaporised and passed together with hydrogen over a catalyst. The vapours leaving the vapour-phase converter may be returned to the middle-oil separator, whereby unconverted middle oils are condensed and the light hydrocarbons pass on with the uncondensed portion of the products from the first stage.

A. B. MANNING.

**Destructive hydrogenation of coal, oils, and the like.** J. CROWE, K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 341,676, 17.12.29).—The residual gases containing hydrogen, methane, ethane, and nitrogen, left after removing condensable vapours from the products of destructive hydrogenation, are scrubbed under pressure in two or more stages with an oil (petrol, light or middle oil) at temperatures below 0° to remove methane, ethane, and most of the nitrogen. The pressure upon the collected washings is released in stages, with or without application of heat, so as to liberate first the dissolved nitrogen and finally the dissolved hydrocarbons, which are cracked to yield a gas rich in hydrogen. The cracked gases are circulated with the washed gas through the hydrogenating apparatus and an amount of nitrogen corresponding to that in the make-up gases may be rejected.

H. E. BLAYDEN.

**Destructive hydrogenation of carbonaceous liquids.** W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 338,150, 13.8.29).—A vertical reaction chamber and a separate chamber for the separation of the liquid and gaseous products are housed together in the same high-pressure vessel, the temperature of the separator being maintained at a lower level than that of the reaction chamber, preferably by withdrawing some of the heat of the reaction products before they pass into the separator, by heat exchange with incoming gas or liquid material.

A. B. MANNING.

**Hydrogenation of carbonaceous bodies in the presence of catalysts.** CHEM. REACTIONS, LTD. From L. VON SZESZICH (B.P. 341,560, 22.10.29).—The carbonaceous substances to be hydrogenated (*e.g.*, tars, tar oils, mineral oils, etc.) are heated in a vessel containing chemically inactive filling material, and hydrogen or hydrogenating gases under pressure are passed through the vessel. Injurious ash-containing constituents and asphalt remain in the vessel and are removed, and the material to be hydrogenated passes in an ash-free condition with the hydrogen into a separate reaction vessel

containing the catalyst. Means are provided for controlling the temperature of the gases entering the reaction vessel. It is claimed that the process prolongs the active life of the catalyst.

H. E. BLAYDEN.

**Obtaining light hydrocarbons by the action of nascent hydrogen on carbonaceous material.** J. FOHLEN (B.P. 338,544, 21.6.29).—The material is cracked under suitable conditions of temperature and pressure in the presence of nascent hydrogen produced by the interaction of two substances or groups of substances, which are introduced into the reaction chamber through independent supply pipes.

A. B. MANNING.

**Separation of solid constituents from residues obtained in the destructive hydrogenation and similar treatment of carbonaceous materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,451, 14.10.29).—The residues from destructive hydrogenation, pressure extraction, or low-temperature carbonisation processes are heated (under pressure of nitrogen, methane, or of the vapours produced) at a temperature 20–60° above that at which they were produced. The resulting coagulation of the fine particles produces a decrease in viscosity of the mass, which is filtered or centrifuged to separate the solid and liquid portions. Coking of the product may be inhibited by mixing the residue with finely-divided, porous, refractory materials (*e.g.*, Florida earth) or catalytically active iron powder.

H. E. BLAYDEN.

**Manufacture of liquid products from coal or other carbonaceous materials.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 338,192, 14.8.29. Cf. B.P. 331,199; B., 1930, 940).—The initial materials are heated with hydrogen under pressure in the presence of a catalyst produced by adsorbing a colloidal molybdenum compound on activated carbon or similar adsorptive material.

A. B. MANNING.

**Manufacture of carbon black.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,239, 17.9.29).—The catalyst for the formation of carbon from gaseous carbon compounds is employed in the form of cylinders or balls which roll freely in a rotating furnace; thus by continuous movement the sooty deposit is removed from the active surface. The furnace walls are also provided with holes which allow the carbon powder to escape, but are too small to permit the catalyst masses to fall out.

R. H. GRIFFITH.

**Manufacture of bone black.** BAUGH & SONS CO., Assees. of T. L. WHEELER and J. B. CARPENTER, JUN. (B.P. 338,476, 4.4.30. U.S., 11.4.29).—The bones are heated at 480–700° for a relatively long period, *e.g.*, 1–2 hrs., during which most of the volatile matter is driven out, and the product is heated at 800–1100° for a short period, *e.g.*, 5–15 min. The heating in both stages is carried out preferably in rotary retorts.

A. B. MANNING.

**Production of carbon by the dissociation of gases.** S. PERCIVAL. From C. HOSTMANN-STEINBERG'SCHE GES.M.B.H. (B.P. 340,235, 12.9.29).—A gas, such as acetylene, which dissociates under high pressure with deposition of carbon, is fed to a two-stage compressor

and delivered to a cracking chamber; ignition is effected in a smaller adjacent vessel. After the decomposition is complete, the compressed hydrogen which is produced is blown down, partly into a high-pressure storage reservoir, and partly through a soot collector. Final removal of deposited carbon is achieved by the scouring action of a turbulent stream of hydrogen from the storage vessel, and, if necessary, by further circulation throughout the system.

R. H. GRIFFITH.

**Activation of carbonaceous substances by means of gases.** SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIÈRES (B.P. 316,222 and 316,870, 18.7.29. Fr., [A] 25.7.28, [B] 4.8.28. Cf. B.P. 269,477; B., 1928, 290).—

(A) In apparatus for activating carbonaceous substances by means of gases diffused through the walls of the container, these walls, instead of being formed of porous diaphragms, are provided with circular or lamellate perforations, sufficiently narrow to prevent convection gas currents therein, and inclined at such an angle as to prevent penetration of the substance into them. (B) When porous diaphragms are used, any clogging or glazing of them by the ash of the carbonaceous material may be obviated by constructing the walls with a series of projecting and re-entrant angles, or undulations, in such a manner that the area of contact between the material and the wall is diminished. A. B. MANNING.

**Reactivation of contaminated adsorptive carbon.** METALLGES. A.-G. (B.P. 338,500, 15.5.29. Ger., 8.6.28. Cf. B.P. 313,154; B., 1931, 144).—The reactivating gases, *e.g.*, air and steam, preferably at 120–200°, are passed through the preheated material in such a manner that a localised reactivation zone is formed, which travels through the material in the direction of flow of the gases. The temperature of reactivation is controlled by regulating the proportion of oxygen in the gases.

A. B. MANNING.

**Treatment of plastic materials [*e.g.*, electrode carbon].** P. GRAYIER, and COMP. LORRAINE DE CHARBONS POUR L'ÉLECTRICITÉ (B.P. 319,275, 5.9.29. Fr., 19.9.28).—The pulverised material is subjected, in a suitable container, to a scavenging process to eliminate all air or occluded gases, and is then agglomerated. The scavenging fluid may be a liquid, vapour, or gas, or a mixture of these, *e.g.*, superheated steam, light petroleum, etc. When the scavenging treatment is completed the container may be evacuated in order to remove all the scavenging fluid from the material. The treatment may be effected while the material is in the mould in which it is subsequently agglomerated.

A. B. MANNING.

**Manufacture of gas.** HUMPHREYS & GLASGOW, LTD., and A. R. GRIGGS (B.P. 341,370, 6.5.30).—A complete gasification process is effected by carbonising the coal in the upper part of a generator, the resulting coke being passed into the lower part, where it is gasified by subjecting it to alternating blasting and steaming (cf. B.P. 246,970; B., 1926, 477). The heat of the blast gases, led off from the coke, is partly used to heat a recuperator, which surrounds the carbonising chamber, thereby assisting in the carbonising of the coal. A down-run is made through the coke, with steam superheated in this recuperator, which has been heated by

the blast gases; part of the blue water-gas produced during an up-run may also be burned in this chamber.

C. B. MARSON.

**Manufacture of combustible gas.** HUMPHREYS & GLASGOW, LTD., ASSECS. OF A. F. KUNBERGER (B.P. 338,804, 28.1.30. U.S., 2.3.29).—Hydrogen, produced by passing steam over heated metallic iron, is superheated and carburetted and then passed, together with the excess steam, through the upper part of an ignited fuel bed the whole of which is used simultaneously for the production of water-gas by up-running with directly applied steam. The oxidised iron is reduced periodically, and at the same time is heated by the blow gases from the water-gas generator. The process permits the variation within wide limits of both the calorific value and the sp. gr. of the mixed gas produced.

A. B. MANNING.

**Making combustible gas.** W. W. ODELL, ASSR. TO COLUMBIA ENG. & MANAGEMENT CORP. (U.S.P. 1,762,100, 3.6.30. Appl., 4.3.27).—Gaseous paraffin hydrocarbons, particularly propane and butane, which are available from natural-gas wells, are decomposed by interaction with steam to give a mixture of carbon monoxide and hydrogen. The process is carried out in a water-gas plant by admitting the hydrocarbon gases and steam simultaneously to the fuel bed. The consumption of solid fuel in the generator can be controlled by varying the ratio of steam to gaseous hydrocarbon, and the calorific value of the resulting gas can also be altered by allowing different amounts of unchanged hydrocarbon to pass unchanged. A gas which is particularly suitable for methyl alcohol synthesis can be made by ensuring complete decomposition; this is assisted by working with a high temperature in the cracking chambers.

R. H. GRIFFITH.

**Manufacture of gas from heavy oils.** C. CHILOWSKY (B.P. 338,281, 25.9.29. Fr., 18.1.29).—The oil, mixed with preheated air and steam, is sprayed into a chamber in such a manner that it is projected into a crucible etc. supported therein and maintained in an incandescent state by the partial combustion of the oil. The hot gases and vapours produced pass round the outside of the crucible and are then circulated over a catalyst, whereby the oil is more completely gasified. A part only of the air and steam necessary may be strongly compressed and used for spraying the oil, the remainder, constituting the larger part, being heated at a lower pressure and incorporated with the oil spray by its expansion through a suitable jet. A. B. MANNING.

**Production of fuel gases of high calorific value from bituminous coals.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 338,153, 14.5. and 5.10.29).—The finely-divided coal is carbonised in a stationary chamber containing heat-accumulating material, arranged preferably in the form of a lattice, the chamber being heated periodically to at least 900° by means of combustible gases or by the combustion therein of part of the coke produced. The lower part of the chamber, wherein the coke collects, may be utilised as a gas producer, the finely-divided coke being blasted with air and/or steam at such a rate that it is brought into a state of motion resembling that of a boiling liquid.

A. B. MANNING.

**Manufacture of producer gas.** M. F. CHASE, F. E. PIERCE, and J. SKOGMARK, Assrs. to COS PROCESS Co., INC. (U.S.P. 1,761,384, 3.6.30. Appl., 10.8.20. Renewed 29.3.28).—The nitrogen content of producer gas is reduced by substituting for the moist air generally supplied to the fuel bed a mixture of gases obtained by partial or complete combustion of a portion of the producer gas previously formed. This combustion is carried out with oxygen, or a gas rich in oxygen, so that the calorific value of the final producer gas is greater than 150 B.Th.U. per cub. ft.

R. H. GRIFFITH.

**Making a mixture of water-gas and producer gas.** POWER-GAS CORP., and N. E. RAMBUSH (B.P. 341,001, 7.10.29).—Mixtures of water-gas with producer gas are made in predetermined proportions, such as are required for the production of synthetic ammonia, in plant comprising a water-gas generator and a gas-producer unit, both of the usual type. Air is supplied to both units from individual blowers or from one common blower. The mains taking the cooled gases from the producer and generator lead into a common gas main and volume regulators are provided on the two mains, so that the flow of gas from the producer unit may be adjusted to suit the flow of gas from the water-gas unit. The gas mixture in the required proportions then enters turbo-exhausters, in which it is thoroughly mixed and washed, after which it is passed under pressure into a suitable gas-holder. C. B. MARSON.

**Production of a high-grade gas rich in hydrogen.** VEREIN. STAHLWERKE A.-G., Asses. of E. H. SCHULZ and F. EISENSTECKEN (B.P. 314,870, 1.7.29. Ger., 3.7.28).—A process for converting distillation gases, hydrocarbons, or gas mixtures containing hydrocarbons into a gas rich in hydrogen, which may be used for reducing oxide ores, oxides, or metals containing oxides, consists in passing the sulphur-free gases over catalysts, together with superheated steam at or above 1000°. Suitable catalysts are iron, cobalt, nickel, chromium, aluminium, manganese, copper, vanadium, tungsten, or their alloys with each other. The reaction is carried out in a channel of carborundum, or in tubes of heat-resisting material in a channel lined with refractory material. To compensate for the decrease in strength of the refractory at high temperatures, the tubes may be supported on refractories, at spaced points, the separating spaces depending on the temperatures employed and the nature of the material. The heat necessary for the reaction is generated by induced high-frequency electric current, the catalyst itself or suitably-shaped metallic masses placed in the reaction space receiving the electrical energy. To obtain large throughputs, the gases are preheated by the sensible heat of the waste gases produced in the process. In an example, 50 m.<sup>3</sup> of coke-oven gas (CO<sub>2</sub> 2.6%, heavy hydrocarbons 1.0%, O<sub>2</sub> 1.0%, CO 6.0%, CH<sub>4</sub> 22.4%, H<sub>2</sub> 53.7%, N<sub>2</sub> 12.1%) when passed, together with 12 kg. of superheated steam, over an activated chromium-aluminium catalyst at 1100° yielded a gas containing CO 20%, H<sub>2</sub> 71%, N<sub>2</sub> 9%. The gases obtained in this process are suitable also for the production of pure hydrogen, of nitrogen-hydrogen mixtures, synthetic ammonia, or of methyl alcohol. [Stat. ref.] C. B. MARSON.

**Manufacture of hydrogen and gases containing hydrogen.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,944, 8.4.29. Cf. B.P. 336,635; B., 1931, 103).—Gaseous hydrocarbons are mixed with water vapour, if desired, and introduced into a coke oven in which coal is undergoing carbonisation, conversion and carbonisation thereby taking place simultaneously. [Stat. ref.] W. J. WRIGHT.

**Gas-purification process.** D. L. JACOBSON, Assr. to KOPPERS Co. (U.S.P. 1,752,382, 1.4.30. Appl., 8.12.21).—The gas, after removal of the tar and ammonia, is washed with an alkaline solution containing an iron compound in suspension, *e.g.*, iron oxide (2–3%) suspended in a solution of sodium carbonate ( $\frac{1}{2}$ –1%). The spent wash liquor is regenerated in stages, the first involving a preliminary agitation with air, and the second a more thorough aeration in a scrubber wherein the solution and the air are brought into intimate contact. A. B. MANNING.

**Extraction of phenols from phenolic tar oils.** E. PERILHOU (B.P. 338,638, 11.9.29).—The tar oils are extracted with a solution of an alkali carbonate at raised temperatures, *e.g.*, 170°, and under pressure, the carbon dioxide formed during the reaction being constantly evacuated and used for the acidification of the phenoxide solution obtained from a previous extraction. The regenerated carbonate is used for a further extraction. The process may be made continuous by passing the oil and the carbonate solution countercurrent to one another through a steam-heated vertical tower packed with Raschig rings. A. B. MANNING.

**Manufacture of [bituminous] dispersions.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 341,443, 9.10.29).—Aqueous dispersions of asphalt, tar, pitch, etc. are prepared by the aid of dispersing agents (*e.g.*, clay, colloidal oxides, hydroxides, or silicates) which are insoluble in the dispersing medium. The dispersion is subjected to a heating action during which may be added one or more electrolytes (*e.g.*, acids, acid salts, chromates, etc.) in the requisite quantity to induce maximum impermeability of the water-free film derived from the dispersion and/or to inhibit rust formation when applied to metals. Protective colloids may also be added to the dispersions before, during, or after addition of the electrolyte. H. E. BLAYDEN.

**Manufacture of products [asphalt] for covering carriers [paper] with bituminous substances.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 312,909, 17.5.29).—Moisture-proof products, *e.g.*, asphalt paper, are prepared by treating the carrier with an emulsified bituminous substance, the viscosity of which is reduced below 750 sec. (Redwood II) at one stage during manufacture, by heating the material above the usual heating temperature, *i.e.*, above 120°, if necessary in a non-oxidising atmosphere, by selecting a material which has a sufficiently low viscosity at 80–120°, but is not sticky at normal temperatures, or by the addition of paraffin wax etc. to the material. The paper-fibre mass is preferably first treated with resin-glue and alum. A. B. MANNING.

**Freeing tars and oils from solid particles contained therein.** J. Y. JOHNSON. From I. G. FARBERIND.



A.-G. (B.P. 338,154, 14.5.29).—Tars or oils obtained by the destructive hydrogenation of coal etc. are treated with gases which in aqueous solution are acid to litmus, *e.g.*, carbon dioxide or sulphur dioxide, and are then filtered or centrifuged. The treatment causes flocculation of the suspended particles and thereby greatly facilitates their separation. A. B. MANNING.

**Tar-removing and cleansing composition.** J. H. WYLIE (B.P. 338,167, 14.8.29).—The composition consists of a mixture of benzene, turpentine, olive oil, and citronella oil in specified proportions.

A. B. MANNING.

**[Mineral oil] emulsion.** F. W. SULLIVAN, JUN., ASSR. to STANDARD OIL CO. (U.S.P. 1,773,123, 19.8.30. Appl., 4.9.28).—The soaps of the acids derived from the oxidation of hydrocarbon waxes are used for the production of lubricating emulsions, if desired in conjunction with other emulsifying agents, *e.g.*, soaps, sulphonated oils, and alcohol. E. LEWKOWITZSCH.

**Treatment [cracking] of hydrocarbons.** E. C. D'YARMETT (B.P. 341,882, 17.9.29).—See U.S.P. 1,732,805; B., 1930, 133.

**Furnaces for burning powdered fuel.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 341,864, 16.10.29. Ger., 6.11.28. Addn. to B.P. 282,109).

**Gas burners for furnaces.** F. LILGE (B.P. 341,918, 22.10.29. Addn. to B.P. 317,582).

**Systems for combustion of pulverulent fuels, and burners therefor.** GEN. ELECTRIC CO., LTD., and H. G. HAGUE (B.P. 342,294, 24.10.29).

**Vacuum distillation (U.S.P. 1,771,385). Treatment of gases etc. with liquids (B.P. 2340,631).**—See I. Phenols from tar oils (B.P. 338,638). **Apparatus for hydrogenation (B.P. 341,153). Purification of gases (B.P. 341,444 and 341,584). Absorption of carbon dioxide from gases (B.P. 341,748). Catalytically oxidising hydrogen sulphide (B.P. 341,684).**—See VII. **Pickling-bath inhibitor (U.S.P. 1,773,953). Molybdenum from petroleum hydrocarbons (U.S.P. 1,772,960).**—See X. **Metal compounds of fatty acids (B.P. 338,919). Lubricant (U.S.P. 1,773,203).**—See XII. **Insecticide etc. (U.S.P. 1,764,792).**—See XVI.

### III.—ORGANIC INTERMEDIATES.

**Detection and determination of small quantities of water in ethyl alcohol.** F. SCHUTZ and W. KLAUDITZ (Z. angew. Chem., 1931, 44, 42—44).—The alcohol (10—100 g.) is heated with 10—15% of its weight of powdered calcium carbide for 1.5—2 hrs. under reflux and the acetylene evolved is collected in 30—40 c.c. of acetone. This solution is then poured into 50 c.c. of Hlosvay's cuprous chloride reagent, the resulting cuprous acetylide collected in a glass filter crucible, washed, and dissolved in a 10% solution of ferric sulphate in 20% sulphuric acid, and the ferrous sulphate formed titrated with 0.1N-permanganate (1 c.c.  $\equiv$  0.0018 g. of water).

A. R. POWELL.

**Silver for chemical plant.** McDONALD.—See X.

#### PATENTS.

**Manufacture of additive products from acetylene and hydrogen halide.** CONSORT. F. ELEKTROCHEM. IND.

G.M.B.H. (B.P. 339,093, 19.11.29. Ger., 19.11.28).—Highly active carbon or silica gel is used as carrier for the catalyst, which may be a compound of calcium, strontium, barium, magnesium, bismuth, antimony, vanadium, zinc, aluminium, iron, or mercury; the temperature preferred is 180—240°. The preparation of vinyl chloride and bromide, with very little ethyldene dihalide, is described. C. HOLLINS.

**Manufacture of polymerisation products of diolefines.** J. Y. JOHNSON and A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,135, 20.12.29).—The polymerisation of diolefines, *e.g.*,  $\alpha$ -butadiene, by sodium proceeds uniformly and rapidly in presence of aliphatic ethers, *e.g.*, 30—80% of ether or 45% of dimethyl ether. C. HOLLINS.

**Manufacture of oxygenated organic compounds [from carbon oxides and hydrogen or from hydrogenation products of carbon oxides].** H. DREYFUS (B.P. 338,854, 24.7.29).—Carbon monoxide and/or synthetic alcohols are passed with hydrogen over borates, silicates, or phosphates of copper or of alkali or alkaline-earth metals, preferably the acid salts, at 250—400°/50—200 atm. The products are mainly ethyl and higher alcohols. C. HOLLINS.

**Manufacture of acetone [from isopropyl alcohol].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 339,491, 6.12.29).—*iso*Propyl alcohol vapour, with or without steam, is passed through a molten metal or metal salt at about 600°. Molten lead at 625° gives a 96% yield of acetone. C. HOLLINS.

**Preparation of concentrated formic acid solutions.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 339,045, 7.10.29. Holl., 31.10.28).—Aqueous (85%) formic acid is distilled, preferably in a vacuum, *e.g.*, at 12 mm., with orthophosphoric acid which has been distilled up to 150°/12 mm. C. HOLLINS.

**Manufacture of esters of phosphorous and phosphoric acids containing halogen.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 338,981, 31.8.29).—An alkylene oxide is caused to react with a phosphorus trihalide or a phosphoryl halide. Ethylene oxide, led into phosphorus trichloride at  $-5^{\circ}$  to  $-10^{\circ}$ , yields tri-( $\beta$ -chloroethyl) phosphite. Epichlorohydrin similarly reacts at 100° with phosphoryl chloride or phosphorus tribromide. A catalyst (1% of iron filings) may be added. C. HOLLINS.

**Manufacture of carboxylic acids from nitriles.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,235, 18.12.29).—The nitrile vapour, *e.g.*, gases from interaction of ammonia and acetylene after removal of pyridines, is passed up a tower, packed with Raschig rings etc., of material unattacked by acid, in counter-current to 50—80% sulphuric acid or 90% phosphoric acid, the tower being maintained at 150—220°. (Cf. B.P. 332,258; B., 1930, 980.) C. HOLLINS.

**Liquid or plastic preparations.** H. T. BÖHME A.-G. (B.P. 316,132, 24.6.29. Ger., 23.7.28).—Liquid or plastic preparations for use as wetting or dispersing agents, *e.g.*, in dyeing furs, in salves, insecticides, etc., are improved by the admixture of lower aliphatic esters (up to the cetyl esters, but exclusive of the gly-



cerides) of fatty sulphonic acids (cf. B.P. 313,160 and 315,832; B., 1930, 986, 1058). E. LEWKOWITSCH.

[Wetting etc. agents for] liquid or plastic preparations. H. T. BÖHME A.-G. (B.P. 318,610 and 341,978—9, 19.7.29. Ger., [A—C] 7.9.28).—The sulphonation products of the higher aliphatic alcohols ( $C_6$  and above) are added to improve the wetting and dispersive powers of liquid and plastic preparations, such as (A) dispersions of dyestuff powders, pigments or ceramic materials, for dyeing preparations, or liquids for use in ink manufacture; (B) dust-binding agents, liquid corrosives, or boring oils; and (C) salves, creams, and vasenol substitutes. E. LEWKOWITSCH.

Emulsifying agents and emulsions prepared therewith. H. T. BÖHME A.-G. (B.P. 313,966, 19.6.29. Ger., 20.6.28).—Emulsifying agents for oils and fats are obtained by esterifying sulphonated fatty acids (e.g., sulphoricinoleic acid with isopropyl alcohol), by treating a fatty acid or oil with an alkyl hydrogen sulphate (e.g., oleic acid with *n*-butyl hydrogen sulphate below  $16^\circ$ ) or with an alcohol and sulphuric acid (e.g., ricinoleic acid with *n*-butyl alcohol and sulphuric acid below  $11^\circ$ ), or castor oil with *n*-butyl alcohol and sulphuric acid at  $-5^\circ$  to  $5^\circ$ . The fatty acid or oil must be unsaturated or hydroxylated. C. HOLLINS.

Preparation of cyclic non-saturated ketones with more than 9 ring-members. SOC. ANON. M. NAEF & CIE. (B.P. 339,348, 6.9.29. Switz., 9.10.28. Addn. to B.P. 235,540; B., 1925, 738. See also B.P. 263,153; B., 1928, 36).—The process of the prior patents is applied to unsaturated  $\alpha\omega$ -dicarboxylic acids above  $C_{10}$ . Examples are:  $\Delta^8$ -cyclohexadecenone, m.p.  $22^\circ$ , b.p.  $193^\circ/19$  mm. (semicarbazone, m.p.  $171^\circ$ ), from yttrium  $\Delta^7$ -pentadecene-1 : 2-dicarboxylate;  $\Delta^9$ -cycloheptadecenone (civetone) from thorium  $\Delta^8$ -hexadecene-1 : 16-dicarboxylate. C. HOLLINS.

Preparation of aminobenzyl-*o*-benzoic acids [3'-aminodiphenylmethane-2-carboxylic acids]. NEWPORT CO. (B.P. 314,804, 28.6.29. U.S., 2.7.28).—A 3'-amino- or 3'-acylamino-2-benzoylbenzoic acid, with or without a 4'-substituent (especially 4'-methoxyl), is reduced, preferably with zinc and an ammoniacal copper salt solution; the 3'-amino-group may be subsequently acylated. The acylated products are suitable for conversion into anthrones by means of sulphuric acid, and thence into anthraquinones. Examples are the preparation of 4'-chloro- (m.p.  $132^\circ$ ), 4'-methoxy- (m.p.  $148$ – $149^\circ$ ; acetyl derivative, m.p.  $155^\circ$ ), 4'-ethoxy- (m.p.  $137$ – $138^\circ$ ) derivatives of 3'-aminodiphenylmethane-2-carboxylic acid. C. HOLLINS.

Manufacture of C-alkyl and C-aralkyl derivatives of aromatic compounds. Z. FÖLDI (B.P. 319,273, 4.9.29. Hung., 19.9.28).—Aromatic compounds free from amino- and carboxyl groups are treated with alkyl or aralkyl arylsulphonates. With lower alkyl esters  $200$ – $300^\circ$  is necessary and polyalkyl derivatives are formed. Aralkyl and unsaturated alkyl groups are introduced smoothly at lower temperatures and mono-substitution is the main reaction. Mono-, di-, and triethylbenzenes are obtained from benzene and ethyl *p*-toluenesulphonate at  $300^\circ$ ; allylxylenes and resinous

polymerides from boiling xylene and allyl benzenesulphonate; diphenylmethane and dibenzylbenzenes from benzene and benzyl benzenesulphonate. The preparation of 3 : 5-dinitrodiphenylmethane, m.p.  $183$ – $185^\circ$ , *o*- and *p*-hydroxydiphenylmethanes, methoxydiphenylmethanes, methyl hydroxydiphenylmethanecarboxylate, m.p.  $80^\circ$  (from methyl salicylate; free acid, m.p.  $136^\circ$ ), and mono- and di-benzylated ethyl cinnamates ( $\alpha$ -benzyleinnamic acid, m.p.  $157$ – $158^\circ$ ), is also described.

C. HOLLINS.

Manufacture of [dinaphth]azine derivatives. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,283, 30.8. and 11.11.29).— $\beta$ -Naphthylamines or their sulphonic acids, having a free 1-position, are heated, the former in a high-boiling solvent (trichlorobenzene), the latter in aqueous solution or suspension, with copper oxide or other oxidising metal oxide (manganese dioxide).  $\beta$ -Naphthylamine with cupric oxide in trichlorobenzene at  $180$ – $200^\circ$  gives  $\alpha$ -dinaphthazine, m.p.  $277$ – $278^\circ$ , in 90% yield. The 3 : 10-dimethyl- (m.p.  $328^\circ$ ), 3 : 10-dibromo- (m.p. above  $340^\circ$ ), 1 : 8-dimethoxy- (m.p.  $222^\circ$ ), 2 : 9-dimethoxy- (m.p.  $281^\circ$ ), 3 : 10-di-(anilidodisulphonyl) (m.p.  $326^\circ$ ; from  $\beta$ -naphthylamine-6-sulphonanilide), and 2 : 9-diacetamido- (m.p.  $255^\circ$ ) derivatives are similarly prepared.  $\alpha$ -Dinaphthazine-2 : 9- and -1 : 8-disulphonic, -2 : 4 : 9 : 11- and -1 : 3 : 8 : 10-tetrasulphonic, and -1 : 3 : 6 : 8 : 10 : 13-hexasulphonic acids are obtained by oxidation of weakly alkaline aqueous solutions of the corresponding  $\beta$ -naphthylaminesulphonic acids with pyrolusite. C. HOLLINS.

Gas rich in hydrogen (B.P. 314,870).—See II. Azo dye intermediates (B.P. 318,839).—See IV. Oxygenated components from pine oil (U.S.P. 1,772,546). Borneol from pine oil (U.S.P. 1,772,895).—See XIII. Ethylene glycol dinitrate (B.P. 341,456). Purification of trinitrotoluene (B.P. 341,718).—See XXII.

#### IV.—DYESTUFFS.

Arsenic-containing colour lakes. KAPPELLER.—See XIII.

#### PATENTS.

Manufacture of halogenated 3 : 4 : 8 : 9- or 4 : 5 : 8 : 9 [2 : 3 : 7 : 8- or 1 : 2 : 7 : 8]-dibenzpyrenequinones. I. G. FARBENIND. A.-G. (B.P. 338,747, 4.12.29. Ger., 4.12.28).—2 : 3 : 7 : 8- or 1 : 2 : 7 : 8-Dibenzpyrenequinone is halogenated in a sodium or potassium aluminium chloride melt; e.g., the crude product from condensation of 1 : 5-dibenzoylnaphthalene or 1 : 4-di-*p*-chlorobenzoylnaphthalene or 4-benzoylbenzanthrone in presence of sodium aluminium chloride is chlorinated or brominated without isolation. The products are orange to scarlet vat dyes.

C. HOLLINS.

Dyes [indanthrone]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 338,891, 25.5.29).—The disulphuric ester of leuco- $\beta$ -aminoanthraquinone is oxidised with a hot aqueous cupric salt (chloride or sulphate), which may be less than that theoretically required. The product is apparently indanthrone.

C. HOLLINS.

Manufacture of [direct] azo dyes [for cotton]. I. G. FARBENIND. A.-G. (B.P. 338,672, 10.10.29. Ger.,

10.10.28).—The azo compounds of B.P. 313,636 (B., 1929, 672) are diazotised in substance and coupled with coupling components. Examples are: *p*-nitrobenzoic 4-amino-3-sulphonanilide, reduced alkaline,  $\rightarrow$  1-*p*-sulphophenyl-3-methyl-5-pyrazolone (golden-yellow); 1:4-di-*p*-nitrobenzamidobenzene-2-sulphonic acid, reduced alkaline,  $\rightarrow$  acetoacetanilide (yellow) or *m*-aminobenzoyl-J-acid (orange-red). C. HOLLINS.

**Manufacture of [azo] dyes and intermediate products [of the azine series].** SOC. CHEM. IND. IN BASLE (B.P. 318,839, 9.9.29. Switz., 8.9.28).—An aminonaphthol or its sulphonic acid, especially J-acid, is esterified, then oxidised to the dinaphthazine, and hydrolysed. The product from J-acid is coupled in substance or on the fibre with a diazo component and the resulting dye may be chromed, coppered, etc. in substance or on the fibre. Examples are: *O*-*p*-toluenesulphonyl-J-acid oxidised with hypochlorite at 20–25° and hydrolysed with 5% caustic alkali to give the azine (I); 4-chloro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  (I) (blue on cotton, becoming grey-blue when coppered; prechromed, green-grey on cotton, viscose silk, wool, or silk); 4-nitro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  (I) (grey-green on cotton; coppered, green-grey; prechromed, olive-green on cotton, viscose silk, wool, or silk; prechromed and precoppered, green-grey on cotton); sulphanilic acid  $\rightarrow$  (I) (red-brown on silk or wool); *p*-nitroaniline  $\rightarrow$  (I) (brown on silk and wool); 4-chloro-2-aminodiphenyl ether  $\rightarrow$  (I) on the fibre (dark brown). C. HOLLINS.

**Manufacture of [azo] dyes [for chrome leather].** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 338,930, 22.7.29).—An aromatic *N*-nitroso-compound, especially of an azo dye derived from diphenylamine, is warmed with an arylamine, phenol, or aminophenol, particularly an amino- or hydroxy-azo dye. The products may be produced on the leather. Examples are: nitrosated sulphanilic acid  $\rightarrow$  diphenylamine with sulphanilic acid  $\rightarrow$   $\alpha$ -naphthol (red-brown); nitrosated 2:5- and 2:8-naphthylaminesulphonic acid  $\rightarrow$  diphenylamine with 4-nitroaniline-2-sulphonic acid  $\rightarrow$   $\alpha$ -naphthol (fawn); nitrosated sulphanilic acid  $\rightarrow$  diphenylamine with 4-aminoazobenzene-3:4'-disulphonic acid  $\rightarrow$   $\alpha$ -naphthol (bordeaux); nitrosated 4-chloroaniline-3-sulphonic acid  $\rightarrow$  diphenylamine with sulphanilic acid  $\rightarrow$   $\alpha$ -naphthylamine (dark brown); *N*-nitrosodiphenylamine-4-sulphonic acid with sulphanilic acid  $\rightarrow$  resorcinol (olive-brown); nitrosated sulphanilic acid  $\rightarrow$  diphenylamine with  $\alpha$ -naphthylamine hydrochloride (dark brown) or with 2:3:6-aminonaphtholsulphonic acid (dark brown); *N*-nitrosomethylanthranilic acid with J-acid (light brown). C. HOLLINS.

**Manufacture of monoazo dyes [for wool and leather].** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 339,029, 24.9.29).—Monoazo dyes, obtained by diazotising a negatively substituted *p*-nitroaniline or homologue and coupling with a diphenylamine compound, are sulphonated. Examples are: 2-chloro- or 2:6-dichloro-4-nitroaniline  $\rightarrow$  diphenylamine, sulphonated with 5–10% oleum at 10° (brown); 2-chloro-4-nitroaniline  $\rightarrow$  diphenylamine-4-sulphonic acid,

sulphonated with 15% oleum at 15–20° (chestnut-brown). C. HOLLINS.

**Wetting etc. agents** (B.P. 318,610 and 341,978–9).—See III. **Dye pastes etc.** (B.P. 318,176 and 318,178).—See VI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Determination of the uniformity of filaments [of artificial silk].** E. VIVIANI (Giorn. Chim. Ind. Appl., 1930, 12, 580–582).—The filament is drawn through a capillary tube immersed in a vessel of mercury, the alteration in the resistance of the mercury column in the capillary, determined with a Wheatstone bridge, giving a measure of the thickness of the filament. Using a capillary about 9 cm. long and 0.4 mm. wide, the galvanometer readings (1 div.  $\equiv$  0.00001 amp.) for filaments of 65, 110, 130, 160, and 190 deniers were, respectively, 7.4, 12.0, 14.8, 19.2, and 23.8 divisions. More accurate results are obtainable by means of a second arrangement, with which the mercury penetrates the interstices of the separate threads of the filament.

T. H. POPE.

**Arsenic in writing materials.** KAPPELLER.—See XIII. **Dust in air of cotton card rooms.** BRIT. COTTON INDUSTRY RES. ASSOC.—See XXIII.

## PATENTS.

**Mechanical treatment of leaf fibre while immersed in a solvent or washing or other fluid.** J. A. DUFF (B.P. 342,298, 26.10.29).

**Manufacture of imitation furs or skins from textile fabrics.** TEXTILAUSTRÜSTUNGS-GES.M.B.H. (B.P. 342,181, 7.3.30. Ger., 6.2.30).

**Apparatus for testing textile or other extensible material.** DUNLOP RUBBER CO., LTD., and R. TRUESDALE (B.P. 342,382, 7.11.29).

**Spinning bowls for spinning artificial silk.** GEN. ELECTRIC CO., LTD., and G. W. GORVIN (B.P. 342,015, 7.11.29).

**Production of [completely saturated] insulating paper.** V. BAUSCH (B.P. 342,026, 12.11.29).

**Filter aid** (U.S.P. 1,770,052).—See I. **Asphalt paper** (B.P. 312,902).—See II. **Insulator** (U.S.P. 1,770,663). **Insulating material** (B.P. 341,535 and 342,290).—See XI. **Furfural resin composition** (U.S.P. 1,771,033).—See XIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Detection of traces of active chlorine or active oxygen in fabrics.** K. SCHWARZE (Z. angew. Chem., 1931, 44, 44).—The fabric is moistened with dilute sulphuric acid and pressed in a screw press between two sheets of potassium iodide-starch paper covered with two flat glass plates. Blueing of the paper indicates that the bleaching agent has not been completely removed from the fabric. A. R. POWELL.

**Dyeing of acetate silk. Theory of photochemical decomposition of acetate silk dyes.** H. BRANDENBURGER (Kunstseide, 1930, 12, 267–272; Chem. Zentr., 1930, ii, 1777).—A discussion.

A. A. ELDRIDGE.

**Fastness of dyes to perspiration.** C. C. N. VASS (J. Soc. Dyers and Col., 1931, 47, 9—10).—The results of previous investigations (B., 1929, 430, A., 1930, 1309) are summarised. Work is proceeding on the bleeding of dyed materials by perspiration in an endeavour to formulate perspiration test liquors. A. J. HALL.

**Standardising the methods of testing the fastness of dyed materials.** P. W. CUNLIFFE (J. Soc. Dyers and Col., 1931, 47, 7—9).—An interim report is presented which summarises previous investigations carried out in establishing methods for determining the fastness of coloured materials to light and washing. The fastness of dyes to light is influenced least by other factors such as humidity and sulphur dioxide when present in wool; it is therefore likely that dyeings of graded fastness will eventually be prepared, using wool, and a provisional list of dyes which appear suitable for the preparation of these dyeings is given. A. J. HALL.

**Determination of starch on finished goods and yarns.** D. A. DERRETT-SMITH (J. Text. Inst., 1930, 21, T 583—594).—A method for the determination of starch or starch size on finished linen goods, yarns, and cotton fabrics has been elaborated. The starch is converted into dextrose by treatment of the material with 2*N*-sulphuric acid for 2½ hrs. at the boil, the solution is nearly neutralised with sodium carbonate, and a modification of the Schwalbe—Braidy copper number method (B., 1924, 211) is used for the determination of the dextrose. A linear relation is found between the amounts of dextrose present and the permanganate titre, and the experimental curve for the relation between this titre and the weights of various starches taken deviates only slightly from the theoretical curve calculated from dextrose. The factor connecting the experimental starch curve with that obtained for dextrose is 0.943, which agrees well with values found previously. When yarns or fabrics are examined it is necessary to make a blank experiment under identical conditions on the corresponding material containing no starch, but with green flax yarns a satisfactory blank cannot be obtained by using desized material, because the desizing treatment removes a considerable amount of reducing matter as well as starch. The weight of material recommended for analysis is such that the titre does not exceed 35 c.c. of 0.04*N*-potassium permanganate; for the same fabric, smaller weights tend to give higher results than do larger ones. The method is quicker and generally more accurate than that of finding loss on desizing. B. P. RIDGE.

#### PATENTS.

**Formation of bleaching agents [for flour, soaps, fats, etc.].** R. E. ELLIS. From PILOT LABORATORIES, INC. (B.P. 339,336, 29.8.29).—Complex peroxides, obtained by the interaction of a chloride of a higher fatty acid (e.g., coconut oil acids) and benzoyl, chlorobenzoyl, or bromobenzoyl chloride with alkaline hydrogen peroxide, preferably in presence of soap, are used as bleaching agents. C. HOLLINS.

**Dyeing of skins, hairs, feathers, etc.** I. G. FARBENIND. A.-G. (B.P. 339,444, 7.11.29. Ger., 7.11.28).—4-Amino-derivatives of *N*-aralkyldiphenylamines,

which may carry halogen or alkyl in the 4'-position, are oxidised on the mordanted or unmordanted material to give clear greys. The bases (e.g., 4-amino-*N*:4'-dimethyldiphenylamine, and 4'-chloro-4-amino-*N*-methyldiphenylamine) have good solubility and are unaffected by hard water. C. HOLLINS.

**Dyeing of regenerated cellulose materials.** E. I. DU PONT DE NEMOURS & Co. (B.P. 318,891, 27.8.29. U.S., 11.9.29).—Viscose silk is dyed in even shades by the application of secondary disazo dyes of the type: naphthylaminemonosulphonic acid → non-hydroxylated middle component → J-acid or *N*-substituted-J-acid. Examples are: Cleve acid → *m*-toluidine → J-acid (bordeaux), acetyl-J-acid (bordeaux), or phenyl-J-acid (red-violet); Brönner acid → aniline → J-acid (blue-red), or phenyl-J-acid (red-violet); Brönner acid → cresidine → J-acid (red-violet); naphthionic acid → α-naphthylamine → J-acid (violet); Laurent acid → cresidine or Cleve acid → J-acid (blue-violet). C. HOLLINS.

**[Production of coloured] textile fabrics [containing cellulose acetate silk].** BRIT. CELANESE, LTD. (B.P. 318,840, 9.9.29. U.S., 8.9.28).—The presence of the usual size on cellulose acetate yarns used for warp in fabric does not prevent the satisfactory printing upon them of dyes which have an affinity for this type of artificial silk. Thus for the production of a coloured fabric cellulose acetate warp yarn is sized and wound on a beam; it is then suitably printed, dried, and woven into fabric, using a weft yarn of any suitable fibre or colour. A. J. HALL.

**Coloration of artificial materials made of or containing cellulose esters.** BRIT. CELANESE, LTD. (B.P. 314,447, 27.6.29. U.S., 27.6.28).—Superficially saponified cellulose ester materials are dyed with the usual insoluble acetate silk dyes, with or without addition of direct cotton dyes. C. HOLLINS.

**Production of discharge effects on materials made of or containing cellulose esters or ethers.** BRIT. CELANESE, LTD. (B.P. 315,005, 6.7.29. U.S., 6.7.28. Addn. to B.P. 312,655; B., 1929, 678).—Other discharging oxidants, e.g., 30% hydrogen peroxide, silver peroxide, nitric acid, acid permanganate, sodium peroxide, but preferably bleaching powder followed by treatment with citric acid, are used in place of the chlorate in the prior process. C. HOLLINS.

**Manufacture of [vat] dye pastes and printing colours.** I. G. FARBENIND. A.-G. (B.P. 318,176, 28.8.29. Ger., 29.8.28).—An ester of a hydroxy-acid (other than oils and fats and esters of high-molecular acids) is added to vat dye printing pastes in amount insufficient to dissolve all the dye. Ethyl glycollate, lactate, and tartrate are mentioned. The paste may be heated with the ester. C. HOLLINS.

**Manufacture of printing pastes and printing colours.** I. G. FARBENIND. A.-G. (B.P. 318,178, 29.8.29. Ger., 29.8.28).—The condensation products of carbamide, thiocarbamide, etc. with alcohols or ketones (cf. B.P. 278,390, 280,238, 287,095, 290,192, 292,595; B., 1928, 648; 1929, 28, 691, 709, 827) are added to dye pastes for printing. C. HOLLINS.

**Sulphurised compounds of phenol.** [Mordants for basic dyes.] A. THAUSS and A. GÜNTHER, Assrs. to GEN. ANILINE WORKS, INC. (Re-issue 17,940, 27.1.31, of U.S.P. 1,450,463, 3.4.23).—See B., 1923, 491 A.

**Treating textile fabrics with liquors.** E. W. HUNT (B.P. 342,475, 24.12.29).

[Machine for] finishing of textile fabrics. J. BAILEY (B.P. 342,519, 30.1.30).

**Tar-removal from fabrics etc.** (B.P. 338,167).—See II. **Wetting etc. agents** (B.P. 318,610 and 341,978—9). **Liquid preparations etc.** (B.P. 316,132).—See III.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Zinc oxide containing lead.** G. WENZEL (Metall u. Erz, 1930, 27, 32—34; Chem. Zentr., 1930, ii, 140—141).—Legal and analytical requirements are discussed. Alkali chloride solution (15%) is a suitable solvent, but does not always lead to accurate results. Preferably the zinc oxide is treated with a quantity of cold, aqueous barium chloride corresponding with its known sulphate content, the zinc passing into solution as chloride. The residue (washed, dried, and weighed) is boiled with acetic acid and the solution is treated with alcohol. The lead in the filtrate is that present as oxide, that present as sulphate being determined by difference.

A. A. ELDRIDGE.

**Rapid volumetric determination of carbon dioxide in carbonates.** T. HECZKO (Z. angew. Chem., 1931, 44, 85—86).—A weighed quantity of the carbonate is placed in a boat, covered with vanadium pentoxide, and the boat introduced into a combustion tube passing through an electric furnace. The apparatus is closed, oxygen turned on, and the boat drawn into the heated part of the tube by a magnet acting on a nail connected to the boat by a copper wire. The gases are collected and carbon dioxide is determined by absorption. Determinations accurate to 0.1% can be completed in 3—4 min., exclusive of the weighing.

C. IRWIN.

**Oxidation of hydrogen sulphide to sulphur in presence of brown-coal coke as catalyst.** R. SCHMIDT (Braunkohlenarch., 1930, No. 26, 12—24; Chem. Zentr., 1930, ii, 171—172).—Hydrogen sulphide (28%) and air (72%), containing a small quantity of ammonia as accelerator, were passed at the rate of 1.5 litres per hr. over the coke (3.5—4 g., 3—5-mm. particles), the increase in weight being determined after each of 18 litres had passed. Comparative values were obtained for various cokes, the values varying according to the heat treatment of the sample and being increased by pretreatment at a high temperature with steam or carbon dioxide. The activity is associated with the calcium content of the coke.

A. A. ELDRIDGE.

### PATENTS.

**High concentration of nitric acid.** F. C. ZEISBERG, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,772,123, 5.8.30. Appl., 2.8.28).—The dehydrating tower is charged at the top with cold concentrated or fuming sulphuric acid and with dilute nitric acid instead of with some of the refluxed acid as described in U.S.P.

1,772,122 (cf. B.P. 292,951; B., 1929, 1014), and the spent sulphuric acid of about 57.5% concentration flows into a boiling vessel for denitration and further concentration, the vapours passing into a dephlegmating tower in which the dilute nitric acid is partly concentrated before re-entry into the vaporiser supplying acid vapour to the first tower.

L. A. COLES.

**Manufacture of ammonium sulphate.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 341,570, 25.10., 6.12., and 13.12.29).—An aqueous solution of ammonium sulphite or bisulphite, or a mixture of gaseous ammonia, sulphur dioxide, and water vapour, is treated at 80° with oxygen or air in presence of nitrogen oxides, excluding nitrous oxide.

W. J. WRIGHT.

**Preparation of alkaline-earth cyanamides.** N. CARO and A. R. FRANK (B.P. 340,435, 4.2.30. Ger., 8.2.29).—Part of the carbon monoxide in the gas mixture employed for the ammonia synthesis is separated and utilised in the reaction between the ammonia and calcium carbonate, the production of cyanamide being thereby accelerated. The hydrogen formed by interaction of carbon monoxide and water vapour is readmitted to the ammonium synthesis cycle.

W. J. WRIGHT.

**Manufacture of acid phosphate.** B. OBER, Assr. to G. OBER & SONS Co. (U.S.P. 1,761,991—2, 3.6.30. Appl., [A] 5.6.25, [B] 18.8.25).—In (A) a process is described in which ground phosphate rock and an acid are mixed, moisture is eliminated, and the product is air-treated. Suitable apparatus is described in (B), in which a series of containers for the mixture of sulphuric acid and phosphate rock is mounted on a revolving frame, a stirring device being provided to operate in any desired container. When a charge has been mixed, its container is moved into another position, where it is inverted by means of a trip device, and the solidified, porous charge is removed by means of a conveyor to a drying room.

W. J. WRIGHT.

**Concentration of phosphate-bearing material.** W. TROTTER and E. W. WILKINSON, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,761,546, 3.6.30. Appl., 1.3.29).—The material in the form of a fine pulp is agitated with a soap produced by the combination of a fatty acid and an alkylamine, and with a liquid immiscible with water, e.g., fuel oil, the float being separated.

W. J. WRIGHT.

**Manufacture of synthetic chlorides.** D. HIRSTEL, Assr. to J. BRANDIES (U.S.P. 1,771,628, 29.7.30. Appl., 9.3.25).—Hydrogen chloride from the manufacture of salt cake is absorbed by an aqueous suspension of, e.g., magnesium oxide or carbonate, and the chloride formed is recovered by filtering and concentrating the solution obtained.

L. A. COLES.

**Manufacturing powdered metallic [calcium] chloride.** E. O. BARSTOW and S. B. HEATH, Assrs. to DOW CHEM. Co. (U.S.P. 1,768,282, 24.6.30. Appl., 30.8.28).—Granular calcium chloride is introduced into an inclined, rotary, cylindrical dryer in countercurrent to furnace gases, which enter the dryer at 400—475° and leave it at about 100°. The coarse material passes from the lower end of the dryer to a conveyor, but the fine

particles are kept suspended in the current of gas and are drawn through a duct into a separator, yielding a non-caking product containing water of crystallisation and at least 73% of the anhydrous salt.

W. J. WRIGHT.

**Production of ferric oxide.** SULPHUR & SMELTING CORP., Assees. of E. W. WESCOTT (B.P. 314,075, 18.6.29. U.S., 22.6.28).—Ferric chloride in a finely-divided form or as vapour is burned with preheated oxygen or air at 830°, additional heat being supplied at such a rate as to ensure the rapid formation of the oxide in a coarsely crystalline condition.

W. J. WRIGHT.

**Preparation of lead oxides [red lead].** P. E. HAYNES, Assr. to LINDE AIR PRODUCTS Co. (U.S.P. 1,770,777, 15.7.30. Appl., 23.12.25).—Molten lead is introduced into one end of an externally heated, rotating, horizontal kiln separated by perforated partitions into about eight compartments. Preheated air is charged into the lead-inlet end of the kiln and oxygen into the last compartment. Means are provided for transferring the material through the compartments, for conveying the contents of about the fourth compartment to an external grinder before it is passed to the next compartment, for removing the material from the last compartment, and for removing gases from the inlet compartment to maintain atmospheric pressure in the kiln.

L. A. COLES.

**Preparation of [silica] gel.** E. B. MILLER and G. C. CONNOLLY, Assrs. to SILICA GEL CORP. (U.S.P. 1,772,055, 5.8.30. Appl., 12.3.27. Renewed 19.10.29).—A silica hydrogel, before or after washing, is heated during  $\frac{1}{4}$ –5 hrs. to 80–163° and is cooled during  $\frac{1}{4}$ –2 hrs. in an atmosphere saturated with water vapour so that no marked dehydration takes place; the product after subsequent heating at about 870° has  $d$  0.5.

L. A. COLES.

**Manufacture of [silica] gels.** E. B. MILLER, Assr. to SILICA GEL CORP. (U.S.P. 1,773,273, 19.8.30. Appl., 13.3.26. Renewed 1.11.29).—Equal volumes of sodium silicate solution ( $d$  1.185) and hydrochloric acid ( $d$  1.05) are mixed with vigorous agitation and the mixture is set aside until it sets to a stiff gel, which is then broken up and washed with water at 70° for 12 hrs. After cooling, the gel is washed many times with sulphuric acid the sp. gr. of which is gradually increased from 1.45 to 1.7 and then gradually decreased to 1.45 again, and finally the acid is removed by washing for 20 hrs. with water at 70°. The resulting gel is activated by heating at 870°.

A. R. POWELL.

**Treatment of aluminium oxide minerals.** T. A. MITCHELL, Assr. to L. M. HUGHES (U.S.P. 1,772,359, 5.8.30. Appl., 30.8.22).—A finely-ground mixture of bauxite, dolomite, and limestone is heated with sulphuric acid at a temperature sufficient to convert the alumina into calcium aluminate and the magnesia into magnesium sulphate which is subsequently leached out with water. The residual aluminate is digested with sodium carbonate to obtain a solution of sodium aluminate.

A. R. POWELL.

**Removal of cupric oxide [from copper or cuprous oxide surfaces].** W. T. ANDERSON, JUN., and L. F. BIRD, Assrs. to HANOVIA CHEM. & MANUFG. Co. (U.S.P.

1,773,160, 19.8.30. Appl., 28.10.27).—The material is subjected to the action of an alternating current in dilute sulphuric acid, whereby the superficial cupric oxide disintegrates and falls off; the material is then washed in water and alkali to remove acid.

A. R. POWELL.

**Precipitation of titanium compounds.** P. FARFAR, Assr. to TITANIUM PIGMENT Co., INC. (U.S.P. 1,773,727, 26.8.30. Appl., 19.4.27. Norw., 12.5.26).—A solution of titanium and ferrous sulphates produced by the decomposition of ilmenite with sulphuric acid is run slowly into a vat containing a boiling solution of approximately the same composition, but in which the titania content is already precipitated, the latter solution being kept in vigorous agitation. Treated solution is withdrawn from the bottom of the precipitation vat and transferred to a settling tank at the same rate as new solution is added to the vat.

A. R. POWELL.

**Preparation of solid carbon dioxide.** A.-G. F. KOHLENSÄURE-IND. (B.P. 341,361, 25.4.30. Ger., 27.4.29).—Liquid carbon dioxide is caused, at a pressure above that of its solidifying point, to evaporate along the side walls of a block of solid carbon dioxide, so that the block increases in size and has  $d$  1.4–1.5. Apparatus suitable for carrying out the process is described.

L. A. COLES.

**Manufacture of liquid carbon dioxide.** I. G. FARBERIND. A.-G. (B.P. 341,368, 2.5.30. Ger., 3.5.29).—Carbon monoxide or a gas mixture containing it is subjected to catalytic decomposition under heat and pressure, the gaseous products are led under the same pressure into a vessel in which the carbon dioxide formed is condensed, and the residual gas is pumped back into the decomposition vessel, the pressure being maintained by the addition of more carbon monoxide.

L. A. COLES.

**Absorption of carbon dioxide from gases and vapours.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 341,748, 13.2.30. Holl., 26.2.29).—The removal of part of the carbon dioxide from gas mixtures, such as those obtained by treatment of methane with steam, is effected by passing the gas mixture at a temperature above the condensing point of steam and without application of pressure over an alkali carbonate absorbed in pumice stone. The absorbent mass is subsequently regenerated by treatment with dry ammonia gas at 180°, or with air or other suitable inert gas or vapour at 220°, or by subjecting it to a vacuum.

W. J. WRIGHT.

**Recovery of carbon disulphide [from the residual gases in its manufacture].** C. J. STROSACKER and J. I. JONES, Assrs. to DOW CHEM. Co. (U.S.P. 1,771,384, 22.7.30. Appl., 7.12.26. Cf. U.S.P. 1,768,803; B., 1931, 247).—The gaseous residue obtained after condensing the bulk of the carbon disulphide after its manufacture by the usual process is treated with sodium sulphide to "fix" the hydrogen sulphide present, and the carbon disulphide is recovered by distillation or decantation. The sodium hydrogen sulphide solution obtained is treated with sodium hydroxide for re-use in the process.

L. A. COLES.

**Apparatus for carrying out reactions with hydrogen [under pressure].** J. Y. JOHNSON. From

I. G. FARBENIND. A.-G. (B.P. 341,153, 30.10.29 and 1.5.30).—Apparatus for treating coal, tar, oils, etc. with hydrogen under pressure and for the synthesis of ammonia is constructed of or coated with zinc alloys of m.p.  $>500^{\circ}$ , the alloys preferably containing copper and, if desired, manganese, nickel, iron, cobalt, aluminium, or silver. A higher hydrogen concentration is used with material having a high sulphur content than with that containing little or no sulphur. L. A. COLES.

**Purification of hydrogen and hydrogen-containing gases.** TECHNICAL RESEARCH WORKS, LTD., and E. J. LUSH (B.P. 341,584, 31.10. and 6.12.29).—Sulphur compounds are removed by passing the gas through a solution or emulsion of piperidine or its homologues. W. J. WRIGHT.

**Determination of hydrogen sulphide in gas mixtures containing sulphur dioxide, carbonyl sulphide, etc.** A. H. HENNINGER, Assr. to GEN. CHEM. Co. (U.S.P. 1,770,694, 15.7.30. Appl., 16.2.27).—Anhydrous copper or silver sulphate, which may be deposited upon pumice, asbestos, etc., is used for the selective absorption of the hydrogen sulphide.

L. A. COLES.

**Catalytically oxidising hydrogen sulphide.** A. G. BLOXAM. From R. KUHN (B.P. 341,684, 21.12.29).—Oxidation is performed in presence of a complex compound derived from a heavy metal and a cyclic organic compound. Examples are: ammoniacal copper oxide mixed with an alcoholic solution of 1-hydroxyanthraquinone and dissolved in pyridine; mixed alcoholic solutions of manganese acetate and alizarin; hæmin dissolved in aqueous pyridine. W. J. WRIGHT.

**Catalyst for production of sulphur [from hydrogen sulphide]. Production of sulphur [from roaster gases].** [A, B] R. C. BENNER and [A] A. P. THOMSON, Assrs. to GEN. CHEM. Co. (U.S.P. 1,773,293—4, 19.8.30. Appl., [A] 30.6.26, [B] 21.1.27).—(A) Hydrogen sulphide mixed with air or with the requisite quantity of sulphur dioxide is passed at below  $200^{\circ}$  over activated bauxite which is obtained by dehydrating above  $400^{\circ}$  native bauxite having a high content of volatile matter. The catalyst is regenerated by distilling off the sulphur. (B) Roaster gases containing 7%  $\text{SO}_2$  are passed through a glowing low-grade carbonaceous agent, and the issuing gases are mixed with a small proportion of fresh roaster gases and passed over firebrick or bauxite at  $900$ – $1000^{\circ}$  to oxidise soot and tarry matter. The gases are cooled to  $250$ – $300^{\circ}$  to condense most of the sulphur, and the remaining gases are passed over activated bauxite to decompose carbonyl sulphide and hydrogen sulphide. A. R. POWELL.

**Oxidation of sulphur dioxide.** E. B. MAXTED (B.P. 341,725, 24.1.30).—The catalyst employed consists of a vanadate of tin or bismuth. W. J. WRIGHT.

**Purification of sulphur-containing gases or vapours.** BRIT. CELANESE, LTD., W. BADER, and E. J. SHELMEKDINE (B.P. 341,444, 10.10.29).—The gases in presence of about 20% of steam are passed over a heated catalyst containing alumina, together with other oxides if desired. To prevent the reaction of carbon monoxide

with steam, the temperature of the catalyst should not exceed  $500^{\circ}$ , and if iron oxide is present the temperature should be  $250$ – $300^{\circ}$ . The hydrogen sulphide produced is removed by passing the treated gases over iron oxide. W. J. WRIGHT.

**Granular materials** (B.P. 341,349). **Separation of gaseous mixtures** (U.S.P. 1,771,197). **Gas- or fuel-storing material** (U.S.P. 1,770,526).—See I. **Combustion of substances** (B.P. 338,444). **Water-gas-producer gas mixtures** (B.P. 341,001). **Hydrogen** (B.P. 314,870 and 336,944).—See II. **Esters of phosphorus acids** (B.P. 338,981).—See III. **Chlorination of sulphide ores** (U.S.P. 1,773,235). **Treatment of manganese ores** (U.S.P. 1,770,791).—See X. **Electrolysis of water** (U.S.P. 1,771,984). **Hydrogen peroxide by electrolysis** (B.P. 341,847).—See XI. **Gold derivatives of succinimide** (B.P. 338,506).—See XX.

## VIII.—GLASS; CERAMICS.

**Determination of the degree of oxidation of iron and manganese in glass.** F. SALAQUARDA (Glastechn. Ber., 1930, 8, 265—270; Chem. Zentr., 1930, ii, 2560).—Increasing quantities of ferric oxide were added to glass ( $\text{Na}_2\text{O} : 3\text{SiO}_2$ ), the product was melted under different conditions, and the degree of oxidation of the iron and manganese was determined. In iron glasses the same equilibrium is always attained even when potassium nitrate is added; the independence of the colour of these glasses on the ferric oxide content is discussed. In manganese glasses also equilibrium between stages of oxidation is reached, the proportion of the more highly oxidised manganese being approximately proportional to the amount of manganese added. The formation of more highly oxidised manganese is dependent on the conditions of fusion, but not on the state of the manganese added. With mixtures of iron and manganese in glass the equilibrium between ferrous and ferric oxides is independent of the manganese, although the latter affords much oxygen and finally consists entirely of manganous oxide.

A. A. ELDRIDGE.

**Coloration of glass with carbon and sulphides.** A. BORK (Glastechn. Ber., 1930, 8, 275—279; Chem. Zentr., 1930, ii, 2558).—Carbon and carbonaceous substances free from sulphur do not impart a yellow colour to glass; the yellow or brown colour of glasses is usually to be attributed to ferrous sulphide.

A. A. ELDRIDGE.

**Decolorisation of glass.** G. JAECKEL (Glastechn. Ber., 1930, 8, 257—265; Chem. Zentr., 1930, ii, 2558).—Measurements of the amount and stage of oxidation of iron on the colour imparted to glass have been made. Chemical decolorisation is affected by manganese and cerium, the iron being converted into a colourless condition. Physical decolorisation (complementary colorisation) is affected by selenium, nickel, and cobalt. The use of selenium as zinc selenide is proposed. X-Irradiation of the glass caused a reappearance of colour with manganese or selenium glass, but not with nickel and cobalt glass.

A. A. ELDRIDGE.

**Decolorisation mixture for glass.** H. RITTER (Sprechaal, 1930, 63, 700—701; Chem. Zentr., 1930, ii, 2560).—A decoloriser for glass contained manganese dioxide, selenium, and arsenious oxide. A method for the analysis of the mixture is described.

A. A. ELDRIDGE.

**Transparency to X-rays of glasses containing beryllium.** C. GOTTFRIED (Glastechn. Ber., 1930, 8, 283—285; Chem. Zentr., 1930, ii, 2559).—The presence of beryllium in glass does not markedly decrease absorption of X-rays. Glasses with a high beryllium content are hygroscopic and not readily produced.

A. A. ELDRIDGE.

**Effect of repeated burning on the structure and properties of lime-bonded silica bricks.** II. Determination of the proportions of quartz, cristobalite, and tridymite. III. Determination of the reversible thermal expansions. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1930, 29, 381—384, 384—387; cf. B., 1926, 916).—II. The amount of quartz and of tridymite (by vol.) was calculated from the sum of the linear intercepts for each constituent, as measured on a thin section examined under a microscope with a micrometer eyepiece. By floating the finely-powdered material on a liquid of  $d$  2.5, the quartz was separated from the mixture of tridymite and cristobalite. The amount of cristobalite in the mixture was calculated from the observed density of the mixture (cristobalite has  $d$  2.33, tridymite 2.28). Since no appreciable difference was found in the chemical composition of the bricks with one burning and those with 11 burnings, it was assumed that the amount of "glass" remained constant and was calculated to be 8.5% by wt. The results indicate that at least 20 burnings are necessary to invert the whole of the quartz.

III. Expansion curves are given for test-pieces burned up to 12 times in a commercial kiln.

J. A. SUGDEN.

**Oil fuel and the brick industry.** I. LUBBOCK (Trans. Ceram. Soc., 1931, 30, 68—80).

#### PATENTS.

**Elimination of iron from inorganic materials [for use in ceramics].** VEREIN. STAHLWERKE A.-G. (B.P. 341,060, 10.9.29. Ger., 12.10.28).—The material is heated at 500° in a reducing atmosphere to reduce the iron oxide and is then treated at 120° with gases containing carbon monoxide to volatilise the iron as carbonyl.

A. R. POWELL.

**[Sole for] glass-annealing furnaces.** Y. BRANCART (B.P. 342,540 10.2.30. Belg., 14.2.29).

**Grinding artificial and natural stone and the like, more particularly refractory materials.** VEREIN. STAHLWERKE A.-G. (B.P. 341,957, 24.10.29. Ger., 10.11.28).

**Tunnel kilns** (B.P. 340,271). **Drying apparatus** (U.S.P. 1,770,727).—See I. **Combustion [of refractories]** (B.P. 338,444).—See II. **Wetting etc. agents** (B.P. 318,610 and 341,978—9).—See III. **Insulators** (B.P. 340,371).—See XI.

## IX.—BUILDING MATERIALS.

**Setting of Sorel cement.** A. KREIGER (Tonind.-Ztg., 1930, 54, 577—578; Chem. Zentr., 1930, ii, 116).—The total magnesium chloride in the hardened cement has been determined by boiling three times with water and titrating with silver nitrate. The uncombined magnesium chloride is determined by extraction with alcohol, which also removes the free water. The alcohol is driven off at 80° and then at 200° the water combined with the magnesium chloride is expelled without decomposing the oxychloride. A formula for the oxychloride cannot be deduced. The initial setting must be caused by the oxychloride, which brings about an expansion. Magnesium hydroxide causes a shrinkage.

L. S. THEOBALD.

**Free lime in Portland cement and constancy of volume.** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 496—497 B).—Free lime has been determined in a number of synthetic and other cements by the glycerol method and correlated with the blowing of the samples. It is concluded that the blowing of cement is connected with the presence of free lime, although little of the latter is present in a well-burned sample. The heating test of a sample of cement giving about 4% of residue on a 4900-mesh/cm.<sup>2</sup> sieve and containing about 1% of free lime was not constant, nor were the heating and water tests of a sample containing about 2.2% of free lime. It is not clear if free lime determined by the glycerol method is absolutely in the free state or exists as explosive grains, but the constancy of volume is closely connected with the free lime content. The strength of blown cement is low, being higher in the case of a more uniform cement, and there is probably a relation between the tensile strength of cement and its blowing.

E. A. RYDER.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Laboratory concentration of the Missouri iron ores of Iron Mountain and Pilot Knob.** F. D. DE VANEY and S. R. B. COOKE (Bull. U.S. Bur. Mines, Min. and Met. Ind., Tech. Sec., 1928, 11, [3], 1—38).—The Iron Mountain ore contains 40% Fe as hæmatite together with more or less finely-disseminated garnet, calcite, and quartz in a porphyritic country rock. By crushing and gravity concentration a concentrate containing only 54% Fe with a high loss of iron in a tailing assaying 17—20% Fe is the best that can be obtained. Magnetic separation of the raw ore is impracticable, but after reduction at 600° with carbon monoxide or producer gas a good separation of the iron can be effected, provided the ore is crushed finer than 14-mesh. After grinding through 100-mesh, reducing at 600°, and separating magnetically, 76% of the total iron can be recovered as a concentrate containing 67.5% Fe, 2.6% SiO<sub>2</sub>, and only traces of lime, phosphorus, and sulphur. The Pilot Knob ore is essentially an intimate mixture of hæmatite and quartz and can be concentrated only by magnetic separation of the reduced ore ground to pass at least 150-mesh.

A. R. POWELL.



**Operation and metallurgy of a 200-ton tilting furnace for the Talbot process [of making steel].** W. ALBERTS (*Stahl u. Eisen*, 1931, **51**, 117—126).—The composition and mechanical properties of numerous charges of steel made in a 200-ton tilting open-hearth furnace by the Talbot process have been determined and the results show that steel made by this process, using a pig iron: scrap ratio of 3:1, is fully up to the standard of the best basic open-hearth steel made by the Siemens-Martin process, provided that deoxidation is effected with molten, and not with solid, ferromanganese.

A. R. POWELL.

**Nitrogenisation of iron and iron alloys. I.** W. EILENDER and O. MEYER (*Arch. Eisenhüttenw.*, 1930—1, **4**, 343—352; *Stahl u. Eisen*, 1931, **51**, 130).—When electrolytic iron is heated in ammonia below 600° only two different layers are formed on the surface—an inner layer which etches light-coloured with picric acid and consists of grains throughout which nitride needles are disseminated, and an outer granular layer which becomes dark coloured on etching; at temperatures above 600° the braunite eutectoid is formed. The diffusion of nitrogen into iron resembles closely in nature and magnitude that of carbon into iron; at 550° the diffusion constant is  $2.14 \times 10^{-8}$  cm./sec. With increasing carbon content the rate of diffusion of nitrogen into iron becomes progressively slower, but aluminium, chromium, vanadium, and molybdenum have little effect on the diffusion rate. Maximum case-hardening by nitrogen is obtained with the alloy with 1% Al, and this hardening effect appears to be due to precipitation of an insoluble aluminium nitride in the ferrite ground-mass; the nitrified layer has a peculiar structure resembling that produced by the rhythmic precipitation of a colloidal substance. This type of hardening is irreversible and differs from the duralumin type of precipitation hardening in that it cannot be reproduced by quenching and tempering after the metal has once been annealed above the temperature at which coagulation of the hardening constituent occurs, as the latter is insoluble in the ferrite ground-mass. It is assumed that precipitation of aluminium nitride takes place by interaction of the aluminium atoms with the iron nitride formed by direct action of the nitrogen on the iron.

A. R. POWELL.

**Determination of metallic iron in [reduced] ores.** W. ACKERMANN (*Chem.-Ztg.*, 1931, **55**, 30—31).—The mercuric chloride method for the determination of iron in the presence of its oxides is applicable to the determination of metallic iron in reduced ores, provided that carbides, phosphides, and sulphides, which cause high results, are absent.

A. R. POWELL.

**Determination of phosphorus in steel, alloy steels, and cast iron.** A. T. ETHERIDGE (*Analyst*, 1931, **56**, 14—21).—The iron or steel (2 g.) is dissolved in 45 c.c. of nitric acid (*d* 1.2), the solution is boiled with a few drops of permanganate solution until a permanent precipitate of manganese dioxide is produced, sodium nitrite crystals are added until the precipitate dissolves, and the solution is boiled to expel nitrous fumes. After cooling, 10 c.c. of 1:1 ammonia solution are added and the solution is treated at 80° with 30 c.c. of molyb-

date reagent. The yellow precipitate is collected on a pulp filter after shaking for 10 min., washed twice with 5% nitric acid and six times with 5% potassium nitrate solution, and dissolved in 20 c.c. of 0.1*N*-sodium hydroxide, the excess of which is titrated with sulphuric acid (1 c.c. 0.1*N*-acid  $\equiv$  0.000135 g. P). Small quantities of arsenic do not interfere, but more than 0.05% should be separated by treating the first molybdate precipitate with sodium hydroxide, acidifying with hydrochloric acid, reducing with zinc, and precipitating with hydrogen sulphide; the filtrate is boiled, oxidised with nitric acid, and treated first with 0.1 g. of iron dissolved in nitric acid and then with ammonia. The precipitate is collected, washed, and dissolved, together with 1.9 g. of electrolytic iron, in 45 c.c. of 1:1 nitric acid; the solution is then treated with molybdate and the analysis finished as above. If the metal contains much silicon it may be removed by addition of a few drops of hydrofluoric acid or, especially when nickel and chromium are present, the steel may be dissolved in hydrochloric acid and, after removal of silica, the solution is evaporated with nitric acid to convert chlorides into nitrates. With vanadium steels more nitric acid is required and a longer period of precipitation.

A. R. POWELL.

#### Determination of traces of chromium in steel.

W. J. AGNEW (*Analyst*, 1931, **56**, 24—28).—The steel (1 g.) is dissolved in 15 c.c. of 1:3 sulphuric acid and 20 c.c. of water and the solution oxidised with 5 c.c. of 1:1 nitric acid, boiled free from nitrous fumes, and diluted to 200 c.c.; 40 c.c. of this solution are boiled with 3 drops of saturated permanganate solution and the excess of the latter is destroyed by addition dropwise of 1:1 hydrochloric acid. After cooling, a saturated sodium carbonate solution is added to precipitate the iron etc., the liquid is diluted to 100 c.c., and 50 c.c. are filtered through a dry paper and treated with 20 c.c. of 1:3 sulphuric acid and 5 c.c. of a 0.1% solution of diphenylcarbazide. The resulting purple colour is compared with that produced by 0.001*N*-potassium dichromate under the same conditions.

A. R. POWELL.

#### Modified Ford-Williams method [for determining manganese in steels].

L. H. JAMES (*Ind. Eng. Chem. [Anal.]*, 1931, **3**, 31—32).—The sample (2 g.) is dissolved in aqua regia and the solution is boiled with 20 c.c. of 60% perchloric acid until fumes are evolved. After cooling the solution and adding 30 c.c. of nitric acid thereto, 8 g. of sodium chlorate are added in four portions to the solution at the b.p.; overheating should be avoided. Before effervescence has subsided the precipitated manganese dioxide is collected on asbestos, washed, and dissolved in 20 c.c. of 0.1*N*-ferrous sulphate solution containing 100 c.c. of sulphuric acid per litre. The solution is then titrated with 0.1*N*-permanganate. Better results are obtained if the calculated percentage is multiplied by 1.02. The method offers no advantage over the ordinary Ford-Williams method if the steel is soluble in nitric acid.

H. F. GILLBE.

#### Rapid colorimetric method for determination of molybdenum in plain carbon and alloy steels.

T. R. CUNNINGHAM and H. L. HAMNER (*Ind. Eng. Chem. [Anal.]*, 1931, **3**, 106—107).—The sample (0.5—1 g.) is

treated with 25 c.c. of sulphuric acid (1:4) at 60° and after addition of 3 c.c. of hydrogen peroxide (30%) the solution is boiled and filtered. To the cold filtrate, diluted to 100 c.c., 10 c.c. of potassium thiocyanate solution (5%) is added, followed, after vigorous shaking, by 5–10 c.c. of stannous chloride solution (350 g. per litre). By this procedure the iron and molybdenum are reduced and a reddish-brown complex potassium molybdenum thiocyanate is formed; this compound is soluble in ether, with which it is extracted, and the colour of the extract is compared with a standard. If iron is not present in the sample it should be added in the form of an 8% ferric sulphate solution, as in its presence the colour is more stable and is more easily compared. If more than small quantities of vanadium are present in the steel 1 g. of tartaric or citric acid is added and the solution is rendered first alkaline and then acid before addition of the thiocyanate. The method is very rapid, 20–25 min. being sufficient for a complete determination, and the colour standards remain unchanged for at least a week.

H. F. GILLBE.

**Determination of zirconium in plain carbon and alloy steel.** T. R. CUNNINGHAM and R. J. PRICE (Ind. Eng. Chem. [Anal.], 1931, 3, 105–106).—The method, which is described in detail, is applicable to any type of commercial steel and a determination may usually be completed within 2 hrs. It depends essentially on separation of the zirconium as  $ZrHPO_4$  and ignition to pyrophosphate.

H. F. GILLBE.

**Determination of aluminium in ferrochromium and in chromium.** T. R. CUNNINGHAM (Ind. Eng. Chem. [Anal.], 1931, 3, 103–104).—Details are given of a method wherein after dissolution of the metal in hydrochloric acid and precipitation of the hydroxide by ammonia solution and redissolution in acid the iron is precipitated by cupferron and removed, the chromium is oxidised by boiling with perchloric acid, and the aluminium is precipitated with ammonia as hydroxide and weighed as oxide. Chromium occluded in the oxide must be determined colorimetrically, and phosphorus, if present, is separated from the oxide after fusion with sodium carbonate and then determined as phosphomolybdate.

H. F. GILLBE.

**Analysis of ferrosilicon.** G. H. GOODWIN (Analyst, 1931, 56, 21–24).—The finely-powdered metal (1 g.) is fused with 10 g. of sodium carbonate and 5 g. of sodium peroxide in a nickel crucible, the product leached with water, an excess of hydrochloric acid added, the solution evaporated to dryness, and the residue baked at 110° for 2 hrs. and extracted with 20 c.c. of 1:1 hydrochloric acid. After addition of 100–150 c.c. of hot water the silica is collected, washed, ignited, and treated as usual. The filtrate is precipitated with ammonia, the ferric hydroxide collected, washed free from sodium chloride, and redissolved in hydrochloric acid, the solution being evaporated for the recovery of the silica which escaped collection in the first operation.

A. R. POWELL.

**Absorption of water and the prevention of rust formation by the protective coatings of iron pipes.** W. BECK (Gas J., 1931, 193, 216–217).—Determination of the electrical insulating properties of protective

coatings on iron tubes exposed to moisture, as a means of estimating their anti-corrosive properties, is criticised. Tar, pitch paints, and inelastic bituminous materials may be examined by this method, which, however, only gives a general indication of the anti-corrosive properties of the material; when the method is applied to films of anti-corrosive paints and to plastic coverings, results are obtained which are not in agreement with practical experience. A colloidal theory is developed by which the mechanism of the protective action of the "Schade" bandage may be explained. C. B. MARSON.

**Passivating action [on iron] of red lead.** E. MAASS and E. LIEBREICH (Korrosion u. Metallschutz, 1930, 6, 178–181; Chem. Zentr., 1930, ii, 2568).—The protective action on iron of red lead is attributed to its alkalinity and the formation of lead soaps, and not to passivation.

A. A. ELDRIDGE.

**Evans' theory [of corrosion].** E. MAASS and E. LIEBREICH (Korrosion u. Metallschutz, 1930, 6, 49–53; Chem. Zentr., 1930, ii, 458).—There is a clear distinction between the behaviour of attacking and passivating electrolytes, the former exerting attack at the centre of the drop in the so-called drop test, whilst the latter protect the whole surface. It is supposed that there is an increase in concentration towards the centre of a drop.

A. A. ELDRIDGE.

**Principles of corrosion.** G. MASING (Z. Metallk., 1930, 22, 321–326).—The electrochemical theory of corrosion is explained, with reference to the recent work of Evans and Palmaer, and the causes of pitting, the action of protective films, and the mechanism of corrosion of alloys are discussed in the light of modern research.

A. R. POWELL.

**Corrosion phenomena. XVI. Difference effect.** A. THIEL and W. ERNST (Korrosion u. Metallschutz, 1930, 6, 97–103; Chem. Zentr., 1930, ii, 976).—By "difference effect" is understood the diminution in dissolution of a base metal caused by formation of a local element with another suitable metal. The effect is attributed to an electrical resistance phenomenon.

A. A. ELDRIDGE.

**Corrosion of metals by [liquid] fuel.** S. P. USPENSKI and N. I. LADNIZHNIKOVA (Nef. Choz., 1930, 19, 264–277).—Data showing the comparative degrees of corrosion of lead, copper, brass, and iron when exposed to liquid or volatilised fuel oils, acetone, carbon disulphide, ethyl alcohol, or methyl alcohol are tabulated.

CHEMICAL ABSTRACTS.

**Corrosion of aluminium.** H. RÖHRIG (Korrosion u. Metallschutz, 1930, 5, Beih., 37–40; Chem. Zentr., 1930, ii, 129).—Conditions leading to corrosion of aluminium are discussed.

A. A. ELDRIDGE.

**Corrosion of aluminium alloys.** E. MAASS and W. WIEDERHOLT (Korrosion u. Metallschutz, 1929, 5, 265–270; Chem. Zentr., 1930, ii, 2568).—The effect of alkali hydroxide or salt solutions and atmospheric conditions on various aluminium alloys has been examined. As for corrosion by acids, increase in the amount of the constituents insoluble in aluminium is accompanied by diminished resistance to attack.

A. A. ELDRIDGE.

**Local detection of phosphides in metal surfaces.** M. NIESSNER (*Mikrochem.*, 1930, 8, 339—344).—The polished and degreased specimen is etched with cold, faintly acid, saturated silver sulphate solution and the deposited metallic silver is gently rubbed off. The specimen is then immersed for 2 min. in a 0.03% acetone solution of dimethylaminobenzylidenerhodanine, previously decolorised with dilute (1:5) sulphuric acid, and after removal is washed with acetone and ether. The air-dried specimen, when examined microscopically, exhibits deep red to violet patches corresponding in position and extent with the phosphide originally present. Photomicrographs of various phosphor-copper alloys after treatment are reproduced. The process depends on the formation of silver phosphide or phosphate and the subsequent production of a coloured compound between the combined silver and the organic reagent.

H. F. GILLBE.

**Determination of chromium and vanadium in ores and alloys after oxidation with perchloric acid.** H. H. WILLARD and R. C. GIBSON (*Ind. Eng. Chem. [Anal.]*, 1931, 3, 88—93).—On boiling with 70% perchloric acid chromium and vanadium salts are completely oxidised to chromic and vanadic acids, and, after diluting to stop the action of the oxidising agent and to remove free chlorine, any of the usual titration methods may be used. Manganese is not oxidised unless an excess of phosphate is present, when a manganic salt is formed and the oxidation of chromium is hindered. After concentration of the acid to dense fuming, boiling for 20 min. is usually sufficient; care must be taken to prevent loss due to spurting and bumping. The presence of ammonium salts always causes slightly low results, but halides, nitrates, and acetates are decomposed and do not influence the result. Small amounts of tungsten do not interfere, but in presence of large quantities the results are low; tungsten in steels should be oxidised with nitric acid before the perchloric acid treatment. Details are given of the procedure for steels of various types and also ferrochromium, and for chromite and chromic oxide, both of which are completely oxidised by perchloric acid. Chromium may be precipitated as lead chromate from a solution which is *M* with respect to perchloric acid and 0.04*M* to lead perchlorate, and may thus be separated from vanadium, manganese, and iron.

H. F. GILLBE.

**Silver and its application to chemical plant.** D. McDONALD (*Chem. and Ind.*, 1930, 50, 168—178).—The economic history, metallurgy, and physical and chemical properties of silver are reviewed and a brief account is given of the use of the metal in the construction of chemical plant for handling foodstuffs containing organic acids and for the distillation and condensation of acetic acid.

A. R. POWELL.

**Absorption of gold in tube mills.** H. A. WHITE (*J. Chem. Met. Min. Soc. S. Afr.*, 1930, 31, 161—171).—Data are given showing the amount of gold locked up in standard tube mills on the Rand. The lock-up depends to some extent on the grade of ore milled, and at equilibrium averages 1000—2000 oz. per mill, mainly retained between the linings and the tube shell.

A. R. POWELL.

**Working up lead-refining liquors.** F. VOGEL (*Metall u. Erz*, 1930, 27, 88—91; *Chem. Zentr.*, 1930, ii, 1604).—A cyclic process for the recovery of the whole of the sodium is proposed; it is applicable to liquors from the Harris and Betts processes.

A. A. ELDRIDGE.

**Electrolytic determination of lead as lead dioxide.** W. T. SCHRENK and P. H. DELANO (*Ind. Eng. Chem. [Anal.]*, 1931, 3, 27—29).—The influence of a number of factors on the electrodeposition of lead dioxide has been examined. The initial temperature should be 90—95° and the time of electrolysis 1.5—2 hrs. with a current of 3 amp. A high temperature throughout the operation causes incomplete deposition. The solution should contain 20—30% of free nitric acid and a small quantity of sulphate, which increases the coherence of the deposit; ammonium nitrate is not essential, and in large quantities causes scaling. Potassium, sodium, ammonium, calcium, magnesium, cadmium, copper, zinc, and iron at moderate concentration are without influence on the determination, but silver, bismuth, manganese, tin, arsenic, antimony, mercury, chromate, and phosphate in more than very small quantity inhibit deposition, may be deposited with the lead, or cause scaling. The electrodes should be of sand-blasted platinum, and after deposition should be dried rapidly at 150—200°. From 5 to 150 mg. of lead may be determined to within  $\pm 0.2$  mg.

H. F. GILLBE.

**Cold-working of lead, tin, cadmium, and zinc at different temperatures.** A. MOLNAR (*Rev. Mét.*, 1930, 27, 522—534).—Full experimental data are given of work the results of which have already been published (*B.*, 1930, 424), and tables and graphs are reproduced showing the tensile strength, elastic limit, and elongation of lead between —75° and 80°, tin between —20° and 100°, cadmium between —75° and 50°, and zinc between 15° and 200° when the load is applied slowly and when it is applied rapidly.

A. R. POWELL.

**Properties of lead-bismuth, lead-tin, type metal, and fusible alloys.** J. G. THOMPSON (*Bur. Stand. J. Res.*, 1930, 5, 1085—1107).—The tensile strength, hardness (Brinell), and elongation of a number of lead alloys have been examined. The hardening influence of bismuth on lead is less than that of antimony when present in the same proportion; the effect is roughly proportional to the bismuth content up to 50%, apart from variations at 30—40% caused by the limit of solubility of bismuth in lead. Alloys containing up to 12% Bi may be rolled into thin sheets. Heat treatment is without influence on the mechanical properties of alloys containing up to 35% Bi; richer alloys are weakened by quenching from 120°, but after ageing for 24 hrs. the original strength is almost completely restored. The tensile strength and hardness curves of lead-tin alloys (up to 50% Sn) are roughly parallel, rising to a constant value at about 30% Sn and falling slightly after 40—50%; there are irregularities below 16% Sn due to the limited solubility of tin in lead. The elongation falls rapidly with increasing tin content to a sharp minimum at 15%, and thereafter rises to values above 100%. Extensive data are given relating to the mechanical and thermal properties of a variety

of type metals with and without additions of bismuth; although bismuth causes a small decrease of the hardness, tensile strength, and resistance to compression of lead-tin-antimony alloys, it improves the casting properties. Freezing curves of lead-tin-bismuth-cadmium alloys show that the m.p. of the quaternary eutectic mixture is 70°, and that the lower m.p. reported for certain of these alloys are incorrect; minor changes of composition influence the freezing range to a greater extent than the final solidification temperature. Addition of 10% of mercury lowers the m.p. of the eutectic alloy to 66°, and reduces the hardness and elongation without influencing the tensile strength. The quaternary eutectic alloy has a remarkably high elongation (140% for a 2-in. test-bar). H. F. GILLBE.

**Importance of particle size in samples of certain metallurgical materials [alloys].** J. A. SCHERRER and G. E. F. LUNDELL (Bur. Stand. J. Res., 1930, 5, 891—895).—Examples are given illustrating the variation of the composition with the particle size of various commercial alloys consisting of more than one metallurgical constituent, e.g., bearing metal, type metal, lead bronze, cast iron, and ferrotungsten; in all of these cases considerable differences in the composition of the various sized particles were found. On the other hand, zinc-base die-casting alloys, sand-cast copper-aluminium alloys, and duralumin yielded drillings the particles of which were very uniform. Various sampling recommendations for non-uniform alloys are given.

A. R. POWELL.

**When is nickel-plate rustless?** E. WERNER (Metallbörse, 1930, 20, 1321—1322, 1378—1379; Chem. Zentr., 1930, ii, 1764).—The fat solvent must be pure and applied at high pressure; defatting agents derived from aromatic hydrocarbons are preferred to those consisting of alkalis. An electrolytic cleaning process is described and advantageous conditions of nickel-plating are discussed.

A. A. ELDRIDGE.

## PATENTS.

**Reduction of iron ore.** B. M. S. KALLING and C. VON DELWIG (B.P. 340,739, 7.12.29. Swed., 10.12.28).—The ore is fed into a rotary drum-furnace through a wide tube passing about half-way across the furnace, so that it is preheated and partly reduced by the hot carbon monoxide produced in the complete reduction process in the body of the furnace. The carbon required in the reduction is introduced into the furnace through a gastight feeding device.

A. R. POWELL.

**Reduction of [iron] oxide ores.** W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,771,972, 29.7.30. Appl., 8.5.29).—A mixture of iron ore and carbonaceous reducing agent is passed through an indirectly heated reducing furnace, and the resulting sponge iron is quenched with heavy liquid hydrocarbons, whereby the greater part of the latter is converted into more volatile products of lower mol. wt. The sponge iron is separated from the residual oils, and the oils are returned to the quenching chamber. A. R. POWELL.

**Production of metal [iron] sponge.** FLODINJERN AKTIEBOLAG (B.P. 340,586 and 340,562, 30.9.29. Swed., [A] 20.10.28, [B] 27.11.28).—(A) A mixture of finely-ground iron ore and charcoal is formed into briquettes

which are packed on layers of charcoal supported on perforated iron trays in an iron casing and the whole is heated in a gas-fired furnace to 1100°, whereby the ore is reduced to sponge iron with the production of carbon monoxide, which may be utilised in firing the furnace. For the production of chromium-iron sponge the briquettes are composed of a mixture of chromite and ferrosilicon and are heated at 1200°. (B) The reduction is carried out in a cylindrical shaft which is provided with inwardly directed projections or with solid or hollow rods joining the walls of the casing in order to promote the convection and distribution of heat within the cylinder.

A. R. POWELL.

**Utilisation of converter slags for manufacture of [chromium] alloy steel.** P. G. PARIS, Assr. to BETHLEHEM STEEL CO. (U.S.P. 1,768,710, 1.7.30. Appl., 11.3.27).—Pig iron containing chromium is blown in an acid-lined converter to oxidise the silicon and chromium, and the greater part of the refined iron is transferred to a basic open-hearth furnace for conversion into steel. To the remainder of the iron and the chromiferous slag in the converter is added sufficient ferrosilicon to raise the temperature to 1650° during a further short blow; additional ferrosilicon is then added to the slag, whereby an exothermic reaction takes place and the chromium is reduced to form ferrochromium with the iron bath, which is then refined in a basic-lined electric furnace.

A. R. POWELL.

**Moulding sand or material [for casting molten metals] and its treatment.** C. C. DE WITT (U.S.P. 1,770,685—8, 15.7.30. Appl., 8.4.29).—Treatment of (A) the clay binder, (B) the sand, (C) both sand and clay, and (D) a mixture of sand and iron ore grains, with a colloidal solution of ferric hydroxide in ferric chloride having  $pH$  4.5—6.2 is claimed.

A. R. POWELL.

**Heat treatment of cast iron.** WHITE & POPPE, LTD., and J. S. THOMPSON (B.P. 341,738, 7.2.30).—Chilled white cast iron is annealed at 900—1000° for 20—30 min., allowed to cool slowly, embedded in a heat-insulating material, roughly machined to the desired shape, reheated at 850°, and finally quenched in oil to harden it. If desired, the casting may then be tempered at 200° and allowed to cool slowly.

A. R. POWELL.

**Production of high-grade [iron] castings from high-carbon alloys.** E. PIVOVARSKY (B.P. 341,436, 10.9.29).—Cast iron containing 1.25—2.25% Si, a high carbon content, and, if desired, appropriate amounts of nickel and chromium is chill-cast to cause it to solidify, at least partly, in the white condition, annealed at 900—1050° to cause the excess carbon to separate in a finely-dispersed form, cooled slowly, normalised at 20—50° above the Ac3 point, and finally quenched from 20—40° above the Ar3 point.

A. R. POWELL.

**Manufacture of powdered iron or iron alloy for magnetic cores.** HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 340,762, 18.12.29. Ger., 18.12.28).—Iron wire, containing 0.3—0.4% C, is drawn into wire 2 mm. in diam. and cut into pieces 10—20 mm. long; these are heated to bright redness in ammonia, hydrogen, or nitrogen, and subsequently pulverised by hammering, rolling, drawing, or grinding. The resulting powder is

suitable for the manufacture of cores for Pupin coils having a high permeability and a low hysteresis loss.

A. R. POWELL.

**Steel alloys.** NITRALLOY, LTD., Assees. of H. A. DE FRIES (B.P. 341,907, 23.9.29. U.S., 9.10.28).—The steels contain 0.05–1.7% C and 3.1–8 (6–8)% of one or more of the elements silicon, manganese, chromium, molybdenum, tungsten, vanadium, titanium, zirconium, nickel, or copper. Articles made of these steels are case-hardened in nitrogen or ammonia.

A. R. POWELL.

**Manufacture of silicon steel.** W. J. WOOLDRIDGE (U.S.P. 1,773,157, 19.8.30. Appl., 26.5.28).—The metal is hot-rolled until the desired gauge is nearly reached, and is then cold-rolled in a finishing mill to the required thickness, annealed at the appropriate temperature, pickled, dried, and tempered at 300°.

A. R. POWELL.

**Rail steel.** J. K. SMITH, Assr. to GRANULAR IRON Co. (U.S.P. 1,774,189, 26.8.30. Appl., 24.11.25).—Steel made from granular iron produced by the Hornsey process (cf. B.P. 277,325; B., 1928, 931) and containing 0.6–0.9% C and 0.7–1.25% Mn is claimed. The steel is free from nitrides, and is stronger, harder, more rigid, and more resistant to shock than ordinary rail steel.

A. R. POWELL.

**Manufacture of steel having good machining properties.** F. BORGGRAEFE (B.P. 340,529, 23.9.29. Ger., 31.1.29. Addn. to B.P. 318,177; B., 1931, 207).—In melting in a cupola the steel claimed in the prior patent, copper and iron pyrites are added to the charge so that the resulting metal contains 0.15% S and 0.2% Cu.

A. R. POWELL.

**[Steel for] permanent magnets.** C. SATTLER, Assr. to DEUTS. EDELSTAHLWERKE A.-G. (U.S.P. 1,773,793, 26.8.30. Appl., 19.2.30. Ger., 8.3.29).—The steel contains 0.65–1.25% C, 0.25–1% Mn, 1.3–6.5% Cr, and one or more of the following metals: 0.5–3% W, 0.5–1.5% Mo, 0.5–1% Cu, 0.5–3% Co; the sum of these additional elements should not exceed 7%, and some or all of them may be replaced by vanadium.

A. R. POWELL.

**Nitrogenising alloy steel articles.** R. SERGESON (B.P. 341,912, 18.10.29).—The articles are heated in a nitrogenising atmosphere at 450–540° for 5–40 hrs., and then at 590–660° for a similar period.

A. R. POWELL.

**Container for case-hardening and especially for nitrogenisation [of iron or steel].** P. F. M. AUBERT and A. J. P. and H. A. M. DUVAL (AUBERT & DUVAL FRÈRES) (B.P. 340,794, 9.1.30. Ger., 23.1.29).—The container for the articles to be heated in nitrogen comprises a rectangular metal box provided internally on two opposite sides with flat perforated distributor or collector chambers for the gas.

A. R. POWELL.

**Protection of metals [iron or zinc] and their alloys.** C. F. BOEHRINGER & SÖHNE G.M.B.H. (B.P. 342,235, 21.5.30. Ger., 21.5.29).—A small quantity of quinoidine or one of its salts is incorporated into paints or protective coating compositions for iron or zinc or is added to hydrochloric or sulphuric acid pickling baths for these metals or their alloys.

A. R. POWELL.

**Cleaning metal [iron] articles. [Inhibitor for pickling baths.]** H. P. CORSON and R. E. LAWRENCE, Assrs. to GRASSELLI CHEM. Co. (U.S.P. 1,773,953, 26.8.30. Appl., 9.10.29).—The use of ichthyol preparations, derived from the sulphonation of mineral oil, is claimed.

A. R. POWELL.

**Cleaning of metals [steel].** J. H. GRAVELL (B.P. 341,974, 26.10.29. U.S., 13.7.29).—The metal surface is covered with a mixture of 2.5 lb. of acid-washed bentonite in 0.3 gal. of ethyl alcohol, 0.15 gal. of butyl alcohol, 0.39 gal. of water, and 0.06 gal. of 75% phosphoric acid. After drying at 105°, the dried paste is brushed off, carrying with it any oil or grease which contaminated the metal surface.

A. R. POWELL.

**[Inhibitor for use in] cleaning metals [e.g., iron or steel by pickling].** W. L. SEMON, Assr. to B. F. GOODRICH Co. (U.S.P. 1,756,311, 29.4.30. Appl., 20.2.29).—The use of anilinobenzthiazole as inhibitor in sulphuric acid pickling baths is claimed.

A. R. POWELL.

**Inhibitor [for steel-pickling baths].** I. WILLIAMS, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,773,247, 19.8.30. Appl., 9.8.28).—The inhibitor comprises a mixture of sulphite-cellulose waste liquor and  $\alpha$ -ethyl- $\beta$ -propyl-acraldehyde.

A. R. POWELL.

**[Inhibitor for] metal-pickling [baths].** J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,756,317, 29.4.30. Appl., 18.4.28).—The reaction product of acetaldehyde and ammonium phenyl-dithiocarbamate or similar compound is claimed.

A. R. POWELL.

**[Flotation] concentration of minerals.** F. G. MOSES, R. W. HESS, and R. L. PERKINS, Assrs. to BARRETT Co. (U.S.P. 1,774,183, 26.8.30. Appl., 13.5.27).—A compound of the type  $RS_nR'$  is claimed as a flotation agent for copper sulphide ores; R and R' are the same or different alkyl or aryl groups and n is 1 or over. Dibenzyl disulphide is specifically mentioned.

A. R. POWELL.

**Concentration of ores [by froth flotation].** C. G. QUIGLEY (U.S.P. 1,773,787, 26.8.30. Appl., 8.8.28).—Calcium amyl sulphate is claimed as a frothing agent for the flotation of copper sulphide ores (cf. U.S.P. 1,771,550; B., 1931, 256).

A. R. POWELL.

**Froth-flotation agent.** I. H. DERBY and O. D. CUNNINGHAM, Assrs. to P. C. REILLY (U.S.P. 1,772,386, 5.8.30. Appl., 17.6.27).—The product obtained by combining phenol or its homologues with the reaction product of naphthalene, anthracene, or similar compounds with phosphorus pentasulphide at 210° is claimed as a flotation agent for copper ores.

A. R. POWELL.

**Metallurgical process [for treatment of copper sulphide ores].** R. D. PIKE (U.S.P. 1,769,604, 1.7.30. Appl., 23.9.25. Renewed 27.2.28).—Chalcopyrite, ground to pass 200-mesh, is digested at 90–100° with a ferrous-ferric chloride solution containing 12% Fe until the content of ferric iron is reduced to 1–1.5%. The liquor is then passed to a second digester, in which it is treated with pyrrhotite to reduce the remainder of the ferric chloride. The residues from the digesters are distilled to recover sulphur, roasted to sulphatise their

copper content, leached with dilute sulphuric acid to remove the copper, and smelted for the recovery of gold and silver. The sulphate leach liquor is treated with calcium chloride, the precipitate of calcium sulphate removed, and the resulting chloride solution added to that obtained from the digesters. The combined solution is treated with scrap iron to precipitate copper and with hydrogen sulphide to remove zinc and lead; the purified ferrous chloride solution thus obtained is divided into 2 parts, 95% being passed to electrolytic cells for the production of electrolytic iron, and the remainder being treated with lime to produce calcium chloride for use in the process. The spent electrolyte is returned to the digesters for the treatment of further quantities of ore. A. R. POWELL.

**Two-stage chlorination of [sulphide] ores.** T. A. MITCHELL, Assr. to L. M. HUGHES (U.S.P. 1,773,235, 19.8.30. Appl., 6.4.27).—Complex sulphide ores of zinc, copper, and lead are treated with chlorine in a dry or slightly moist condition. The product is then digested with water and more chlorine to complete the conversion of sulphides into chlorides, the ferric and manganese chlorides formed in the first stage acting as chlorine carriers in the second. The solution obtained is treated with calcium chloride to remove sulphates, then with a portion of roasted ore to precipitate the iron, and finally with zinc oxide and chlorine to remove the remainder of the iron and the manganese. The filtrate is cooled to allow the lead chloride to crystallise, and treated first with sponge lead to remove copper, and then with zinc dust to regenerate the sponge lead. Finally the zinc is precipitated as carbonate by boiling the solution with powdered limestone or precipitated chalk. A. R. POWELL.

**Reduction of zinc ore.** E. C. GASKILL, Assr. to ST. JOSEPH LEAD CO. (U.S.P. 1,773,779, 26.8.30. Appl., 17.12.26).—A mixture of roasted zinc ore and carbonaceous fuel is passed downwards through a shaft, in the upper part of which it is preheated to 750–800°, and in the lower part heated to 1000° by its electrical resistance to the passage of a current. In this lower zone liquid zinc is stated to be formed, and means are provided at the bottom of the shaft for collecting the metal and separating it from the solid residue of the ore. A. R. POWELL.

**Treatment of manganese ores.** G. N. LIBBY and G. D. KNIGHT, Assrs. to E. WINSHIP (U.S.P. 1,770,791, 15.7.30. Appl., 12.4.26).—Finely-powdered pyrolusite is digested with a slight deficiency of sulphur dioxide and water to produce a solution of manganese sulphate, which is freed from iron, alumina, and copper by treatment with an alkali carbonate or with magnesia. The purified solution is then evaporated to dryness with magnesium nitrate, and the mass heated at 500° to produce magnesium sulphate and manganese dioxide. The nitrous fumes evolved are passed together with air into milk of magnesia to regenerate magnesium nitrate for use again, and the calcined residue is extracted with water to obtain pure manganese dioxide for use in electric batteries. A. R. POWELL.

**Treatment of copper-containing alloys [for removal of copper].** W. KNOLL, and HÜTTENWERKE

TROTHA A.-G. (B.P. 341,889, 21.10.29. Ger., 20.10.28).—The alloys are melted with silicon and lead, whereby copper silicide is caused to separate as an upper layer which is readily separable from the lower lead alloy layer. A. R. POWELL.

**Beryllium-aluminium alloy.** R. S. ARCHER and W. L. FINK, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,774,542, 2.9.30. Appl., 19.12.28).—An alloy of aluminium with 0.025–1% Be and 3–6% Cu is claimed. The alloy hardens on ageing at the ordinary temperature after quenching from 554°. A. R. POWELL.

**Light-metal [silicon-aluminium] alloy.** BIRMINGHAM ALUMINIUM CASTING (1903) CO., LTD., and P. PRITCHARD (B.P. 342,152, 7.2.30).—An alloy of aluminium with 11 (8–13)% Si and 3 (0.5–3.5)% Ni is claimed to have superior yield point, tensile strength, and machinability to silumin. A. R. POWELL.

**Production of [aluminium]-coated metallic objects.** E. D. MARTIN (U.S.P. 1,770,177, 8.7.30. Appl., 4.9.25. Fr., 8.9.24).—Aluminium chloride vapour is passed over iron, copper, or nickel articles heated at 950–1000°, whereby the surfaces of the articles become covered with a layer of an aluminium alloy. The vapours from the treatment chamber are passed over heated aluminium to regenerate the chloride for further use. A. R. POWELL.

**Zinc-base [die-casting] alloys.** NEW JERSEY ZINC CO., Asses. of W. MCG. PEIRCE and E. A. ANDERSON (B.P. 341,409 and 341,410, 9.8.29. U.S., 12.3.29. Cf. B.P. 341,054; B., 1931, 256).—The alloy comprises zinc with 0.005–5% Ni and (A) 0.05–2% Cd, or (B) 0.05–2% Mn. The nickel may be wholly or partly replaced by one or two of the following metals: magnesium, lithium, or (A) manganese. A. R. POWELL.

**Soldering of metal articles.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 341,340, 18.3.30. Ger., 6.4.29).—The parts of internal-combustion engines are soldered in a reducing atmosphere with a copper-nickel alloy, e.g., monel metal, or preferably with an alloy of 64% Cu, 25% Ni, and 11% Zn. A. R. POWELL.

**[Nickel] alloy for manufacture of metal-cutting tools.** W. A. WISSLER, Assr. to HAYNES STELLITE CO. (U.S.P. 1,774,862, 2.9.30. Appl., 19.5.26).—The cutting edge of a lathe tool is made of a nickel alloy containing 15–40% Cr, 15–35% W, 0.5–2.5% B, and 0.75–2.5% C, the nickel content not exceeding 50%. A. R. POWELL.

**Hard metallic composition [cobalt-tungsten carbide alloy].** R. L. ADAMS, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,775,014, 2.9.30. Appl., 31.7.29).—Lamp-black is heated at 1500° in hydrogen to expel volatile impurities and then mixed with tungsten trioxide and cobalt acetate in the proportion of 136 : 1280 : 640. The mass is ground with water, dried, and heated to decompose the acetate, and the resulting mixture is heated in hydrogen at 1150° for 4 hrs. in a graphite crucible. The tungsten carbide-cobalt powder thus obtained is pressed into bars which are sintered in hydrogen by the electrical resistance method. A. R. POWELL.



**Recovery of molybdenum from petroleum hydrocarbons.** A. OBERLE (U.S.P. 1,772,960, 12.8.30. Appl., 20.5.29).—The residual petroleum coke from cracking processes is mixed with active carbon and further heated to expel volatile matter. Leaching of the residue with a suitable solvent is claimed to extract practically the whole of any molybdenum present.

A. R. POWELL.

**Refining of antimonial lead.** T. H. DONAHUE (U.S.P. 1,774,359, 26.8.30. Appl., 17.1.29).—Lead is treated at 470–480° with 1 lb. of zinc for every 1.5 lb. of arsenic and 1 lb. of copper present, and after cooling to just above the m.p. of the metal the dross is skimmed off. The resulting antimonial lead contains less than 0.1% (Cu + As). Excess zinc is removed by agitating the metal with lead chloride at 440–450°.

A. R. POWELL.

**Production of mercury.** W. GLAESER, Assr. to GLAESER RES. CORP. (U.S.P. 1,774,883, 2.9.30. Appl., 14.11.24).—Finely-ground cinnabar ore is leached at 50–80° with a solution of barium hydroxide and sodium hydrogen sulphide to dissolve the mercury as a double sulphide. The filtered solution is treated with hydrochloric acid to precipitate mercuric sulphide or with zinc dust to precipitate zinc amalgam. Mercury is recovered from the precipitate by distillation.

A. R. POWELL.

**[Silver alloy] material for contact points etc.** J. A. WILLIAMS (U.S.P. 1,774,689, 2.9.30. Appl., 25.9.25).—An alloy of 75% Ag, 23% Cu, and 2% Co is claimed to be suitable for the manufacture of non-oxidisable contacts for magneto screws.

A. R. POWELL.

**Production of tarnish-resisting silver and silver plate.** R. O. BAILEY and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,773,702, 19.8.30. Appl., 14.12.25).—The silver is polished with a paste made of lampblack mixed with a 1% solution of iodine in kerosene.

A. R. POWELL.

**Production of silver-plated articles.** M. H. WILKENS & SÖHNE A.-G., and G. HENKE (B.P. 341,704, 10.1.30).—The articles are plated in the usual way with several successive layers of silver, and between successive plating operations the deposit is rolled, hammered, or otherwise worked and annealed at a red heat.

A. R. POWELL.

**Chromium-plating process.** R. J. PIERSOL (U.S.P. 1,774,901, 2.9.30. Appl., 6.11.28).—To deposit a coating of chromium on an old or worn layer of chromium the article is made the cathode in the usual chromium-plating bath, but the external current is not switched on for some time, to allow the old plating to become etched so that the new coating to be given will adhere firmly.

A. R. POWELL.

**[Nickel-chromium] plating process.** B. R. HAUSEN, Assr. to METALS PROTECTION CORP. (U.S.P. 1,774,269, 26.8.30. Appl., 10.5.29).—Iron or steel articles are treated anodically in a chromium-plating bath until they become coated with a brown film, then transferred to a nickel-plating bath until a thin coating of nickel is produced, and finally are coated with chromium in the usual way.

A. R. POWELL.

**Apparatus for obtaining light metals [sodium] by electrolysis.** SOC. CHEM. IND. IN BASLE (B.P. 340,451, 13.3.30. Switz., 29.8.29).—The cathode used consists of a wide iron plate provided along its width on both sides of the upper edge with a hood-like channel sloping slightly upwards towards one side and terminating at this side in a small collecting chamber from which the metal produced by electrolysis may be continuously or periodically tapped off.

A. R. POWELL.

**Electrodeposition of metals.** S. O. COWPER-COLES (B.P. 341,246, 27.12.29).—In the electrodeposition of metals on a rotating mandrel, removal of the deposit is facilitated by using a highly polished brass or copper mandrel coated with a thin film of beeswax applied as a 0.05–0.5% solution in turpentine containing 1% of carbon disulphide.

A. R. POWELL.

**Charging of furnaces for heat treatment of metal and other goods.** BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 342,362, 25.2.30).

**[Cooling-water] treatment of metal rods subsequently to a rolling operation.** A. E. WHITE. From MORGAN CONSTRUCTION CO. (B.P. 342,189, 12.3.30).

**Heat-treating furnace** (U.S.P. 1,770,081). **Furnace-cooling systems** (B.P. 339,972).—See I. **Gas rich in hydrogen** (B.P. 314,870). **Bituminous dispersions** (B.P. 341,443). **Tar removal from metals** (B.P. 338,167).—See II. **Removing cupric oxide from copper surfaces** (U.S.P. 1,773,160). **Sulphur from roaster gases** (U.S.P. 1,773,293–4).—See VII. **Resistances** (B.P. 341,872). **Galvanising etc.** (B.P. 341,093). **Electrolysis of tin salts** (U.S.P. 1,770,789).—See XI.

## XI.—ELECTROTECHNICS.

**Acidity of insulating oils.** EVANS and DAVENPORT.—See II. **Determination of filament uniformity.** VIVIANI.—See V. **Nickel-plate.** WERNER. **Electrolytic determination of lead as dioxide.** SCHRENK and DELANO.—See X. **Determination of saponification value.** DEMAREST and RIEMAN.—See XII. **Paragutta for cable insulation.** KEMP.—See XIV.

### PATENTS.

**Electric furnaces.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. N. OTIS and G. W. HEGEL (B.P. 341,946, 22.10.29. U.S., 24.10.28).—An electric furnace having a removable refractory framework supported by the walls, and an electric heating resistor, mounted on the framework and securing it in position in the heating chamber, is claimed.

J. S. G. THOMAS.

**Electric furnace.** C. STEENSTRUP, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,771,942, 29.7.30. Appl., 7.7.27).—In an electric furnace heated by internal resistors in an atmosphere, e.g., of hydrogen, means are provided for introducing easily and rapidly into the furnace chamber a charge consisting of separate pieces which are to be united by fusion and a fusing metal, e.g., copper, and for pressing the separate pieces together.

J. S. G. THOMAS.

**Electric induction [crucible] furnaces.** ELECTRIC FURNACE CO., LTD., Assees. of HIRSCH, KUPFER- U.



MESSINGWERKE A.-G. (B.P. 341,325, 27.2.30. Ger., 27.2.29).—The plane of the induction coil surrounding the crucible is, wholly or partly, inclined to the axis of the coil. J. S. G. THOMAS.

**Inductor coils for electric induction furnaces.** ELECTRIC FURNACE Co., LTD., Assees. of HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 341,741, 10.2.30. Ger., 31.12.29).—A hollow inductor coil, through which cooling liquid flows, is formed of a closed seamless tube, and the tube wall at the inner circumference of the coil is folded inwardly to provide a greater cross-sectional area for the current at that part. J. S. G. THOMAS.

**Luminous electric-discharge tubes.** GEN. ELECTRIC Co., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 341,169, 341,601, 341,716, and 342,549, [A, B] 7.11.29, [C] 20.1.30, [D] 18.2.30. Ger., [A] 17.6.29, [B] 11.7.29, [C] 11.2.29, [D] 20.2.29).—(A) Light from a mercury-vapour lamp is transmitted through a blue and a violet or bright-red colour filter arranged in series to give a sky-blue glow. (B) Condensation of vaporisable metal on the wall of the tube is prevented by constructing the tube of glass which is strongly absorbent of infra-red radiation and by partly or entirely enclosing the tube within a jacket of similar glass, the intervening space being filled with a gas of low thermal conductivity, *e.g.*, argon or nitrogen, or with a gas or vapour which is strongly absorbent of infra-red radiation, *e.g.*, carbon dioxide or water vapour. (C) A porous container, *e.g.*, of aluminium, filled with powdered, decomposable, gas-emitting material, *e.g.*, carbon, and a heating coil, are arranged in a discharge tube filled with one or more common gases, *e.g.*, nitrogen, hydrogen, or carbon dioxide, in order to replenish the gas filling. (D) Into tubes containing a filling of rare gas, *e.g.*, neon, a common gas, such as those mentioned in (B), which is rapidly absorbed or decolorised by the discharge or by auxiliary means, and which colours the discharge differently from the colour of the discharge through the rare gas filling, is introduced at definite intervals to vary the colour of the discharge. Alternatively, the secondary (common) gas filling may be replaced by a gas-emitting substance, *e.g.*, sodium azide or magnesium carbonate, arranged in a side tube and heated electrically at definite intervals.

J. G. S. THOMAS.

**Stabilising the light emitted by a discharge-tube lamp [containing neon and mercury].** A. E. WHITE. From CLAUDE NEON LIGHTS, INC. (B.P. 340,203, 18.7.29).—Sufficient neon is introduced into the tube to produce a pressure of 8–30 mm. Hg within the tube at normal temperature (15.5°). J. S. G. THOMAS.

**Manufacture of oxide cathodes for electric-discharge tubes.** EGYESÜLT IZZÓLAMPÁ ÉS VILLAMOSÁGI RÉSZVÉNYTÁRSASÁG (B.P. 341,826, 16.5.30. Hung., 23.12.29).—Oxides of refractory metals which cannot be alloyed with alkaline-earth metals, more especially oxides of tungsten or molybdenum, are electrolytically deposited from solutions of the respective tungsten or molybdenum acids or salts, *e.g.*, sodium orthomolybdate, upon a metal core, which is then coated with an alkaline-earth metal deposited from vapour.

J. S. G. THOMAS.

**Manufacture of high-emission cathodes for thermionic valves.** B. LOEWE (B.P. 314,551, 29.6.29. Ger., 30.6.28).—Barium carbide produced at the cathode by decomposition of barium alkyl, preferably barium dimethyl or diethyl, is subsequently converted into barium oxide by reaction with water vapour.

J. S. G. THOMAS.

**[Anticathode for] X-ray tubes.** N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 341,773, 12.3.30. Holl., 30.3.29).—An anticathode comprising a target of refractory metal, *e.g.*, tungsten, less than 0.5 mm. (preferably 0.1–0.3 mm.) thick, secured to a backing of copper or other material of high thermal conductivity, is claimed.

J. S. G. THOMAS.

**Filaments for electric incandescence lamps.** FALK STADELMANN & Co. From H. MENDEL (B.P. 340,473, 30.7.29).—Filaments are drawn from a metal block, *e.g.*, of tungsten, in which a stable crystal structure is produced by heating zones of the metal concurrently to a high temperature, preferably by means of high-frequency current, so that crystal nuclei existing at the zones are caused to grow.

J. S. G. THOMAS.

**Photo-electric cells.** RAYTHEON PRODUCTION CORP., Assees. of P. LeB. SPENCER (B.P. 341,398, 13.9.29. U.S., 23.11.28).—A layer of magnesium deposited on the internal surface of the cell container is oxidised and coated with alkali metal by heating a mixture of a reducing agent, *e.g.*, silicon, and an oxidising compound of an alkali metal, *e.g.*, rubidium dichromate, arranged in the container.

J. S. G. THOMAS.

**Photo-electric cells.** ARCTURUS RADIO TUBE Co., Assees. of S. RUBEN (B.P. 319,216 and 341,527, [A] 17.9.29, [B] 17.10.29. U.S., [A] 17.9.28, [B] 18.10.28).—(A) A photo-electrolytic cell comprising a copper anode coated with cuprous oxide, and a cathode consisting of zinc and mercury immersed in an emulsoid electrolyte composed of a haloid compound, *e.g.*, ammonium chloride or zinc chloride, and glycerin, is claimed. (B) In a photo-electric cell of the thermionic valve type, electron emission from the cathode is controlled by an electrode comprising a copper core coated with a photo-sensitive compound of the oxygen series of group VI, *e.g.*, molybdenum sulphide, silver sulphide or selenide, or cuprous oxide.

J. S. G. THOMAS.

**Infra-red sensitive cells.** TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 342,055, 29.11.29. Ger., 1.12.28).—Cells, *e.g.*, of selenium-tellurium, enclosed in an airtight vessel, permeable to infra-red radiation, and dried by phosphorus pentoxide, are described. Such cells are thermally formed in the presence of argon, and the vessel is finally sealed under a relatively high vacuum.

J. S. G. THOMAS.

**Primary [electric] cell.** G. POLCICH (U.S.P. 1,771,190, 22.7.30. Appl., 4.11.26. Austr., 30.7.26).—A primary cell comprising a soluble electrode containing magnesium, *e.g.*, "magnalium" or "electron," a porous carbon electrode, an electrolyte (ammonium sulphate solution), and a depolariser (ammonium persulphate solution), is claimed.

J. S. G. THOMAS.

**Material for making electrolyte for primary batteries.** A. SCHMID, and METROPOLE DEVELOPMENTS;

LTD. (B.P. 340,429, 29.1.30).—A cellular, porous, absorbent, rigid carrier, *e.g.*, of kieselguhr, charged with an oxidising agent, *e.g.*, chromic acid and/or an alkaline dichromate, and an acid, *e.g.*, sulphuric acid, both of which are gradually dissolved when the carrier is placed in water, is claimed.

J. S. G. THOMAS.

**Galvanic cells.** W. W. TRIGGS. From NAT. CARBON Co. (B.P. 342,052, 28.11.29).—A galvanic cell comprising a zinc anode and a cathode containing acetylene-black and a form of manganese dioxide more active than pyrolusite as a depolariser, separated by a paste electrolyte containing solid ammonium chloride, is claimed.

J. S. G. THOMAS.

**Electric [dry] cell.** B. W. BULLEN and F. J. WALLACE, Assrs. to ROBESON PROCESS Co. (U.S.P. 1,773,445, 19.8.30. Appl., 2.4.27).—The cell claimed comprises a zinc electrode, a depolariser containing copper oxide, and electrolyte composed of caustic alkali (sodium hydroxide) gelatinised by means of concentrated paper-pulp digester waste liquor.

J. S. G. THOMAS.

**Electrolytic apparatus [for electrolysis of water].** W. G. ALLAN, Assr. to F. G. CLARK (U.S.P. 1,771,984, 29.7.30. Appl., 16.3.25).—Electrolytic apparatus comprising a gas and electrolyte separating tank mounted on top of and thermally insulated from an electrolytic cell and having compartments united by a common passageway, means for supplying electrolyte to tanks, connexions for supplying the cell with electrolyte from the passageway, and means for removing gases from the cell and delivering them to the compartments above the cell, is claimed.

J. S. G. THOMAS.

**Apparatus for electrolysis with mercury electrodes under pressure for manufacture of hydrogen peroxide.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 341,847, 20.9.29).—Suitable apparatus comprises horizontal superimposed electrolysing dishes and a liquid mercury or amalgam electrode arranged within a pressure-resistant container, and means for supplying compressed oxygen to the electrolyte in a horizontal or slightly downwardly inclined direction just above the surface of the electrode.

J. S. G. THOMAS.

**Electrolyser [for electrolysis of tin salts].** L. U. LA CORSA (U.S.P. 1,770,789, 15.7.30. Appl., 3.5.29. Italy, 9.6.28).—An electrolyser comprising a pit having a concave bottom forming a cathode, and a channel at one edge, an anode member arranged in the pit, and means rotating about the axis of the pit for scraping off metal deposited on the cathode and delivering it to the channel, is claimed.

J. S. G. THOMAS.

**Galvanising or electroplating processes.** WALDBERG SOC. ANON., and M. VON DEVEGIS (B.P. 341,093, 11.10.29).—Light articles to be plated are forced, by centrifugal and/or magnetic force, on to the wall of a drum rotating at high speed and forming the cathode. The force is periodically interrupted so that the articles are tumbled about.

J. S. G. THOMAS.

**Electrical precipitator construction.** H. A. WINTERMUTE and C. W. J. HEDBERG, Assrs. to RES. CORP. (U.S.P. 1,773,835, 26.8.30. Appl., 12.6.26).—An electrical precipitator comprising a collector electrode

system of high-resistance material defining a gas passage having zones of high and of low gas velocity, and presenting only limited areas of surface to the discharge electrodes which are arranged so that zones of high electric field intensity are located at zones of high gas velocity, is claimed.

J. S. G. THOMAS.

**[Cyclone] apparatus for removing suspended material from gases.** A. NÄTTCHER and F. SEIPP, Assrs. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,773,840, 26.8.30. Appl., 17.5.27. Ger., 30.6.26).—Channel- or pocket-forming members are arranged around the periphery of a cyclone separating chamber, and extend along the path of travel of the gas and transversely to the direction of the cyclonic motion. Portions of the members extend substantially tangentially within the chamber and preferably overlap to form collecting surfaces, slots or openings, however, being left between adjacent members through which precipitated material passes into the channels or pockets. A high *P.D.* is maintained between insulated discharge-electrode means mounted in the chamber and the collecting surfaces.

J. S. G. THOMAS.

**[Electrical] detection of foreign matter in fluids.** WALTER KIDDE & Co., INC., Asses of G. G. FREYGANG (B.P. 341,086, 10.10.29. U.S., 30.10.28).—Light scattered by foreign matter present in a fluid falls upon photo-sensitive material, *e.g.*, a photo-electric cell, and operates a relay, indicator, or alarm.

J. S. G. THOMAS.

**Electrically insulating (A) sheet material, (B) tubing.** INTERNAT. GEN. ELECTRIC Co., INC., Asses. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 341,535 and 342,290, [A] 18.10.29, [B] 19.10.29. Ger., [A, B] 19.10.28).—(A) Fabric composed of "threads" of spun paper is impregnated with a binder such as a varnish containing natural or artificial resins. (B) Tubing made from such fabric is claimed.

J. S. G. THOMAS.

**Thermal and electrical insulator.** A. A. SAMUEL (U.S.P. 1,770,663, 15.7.30. Appl., 1.2.27. Fr., 6.2.26).—Dried cellulosic or fibrous woven material is embedded in "Thiolite," a reaction product of sulphur chloride and a condensation product of a phenol with a reactive methylene compound (cf. B., 1926, 198).

J. S. G. THOMAS.

**[High-tension] electric insulators.** CORNING GLASS WORKS, Asses. of J. T. LITTLETON (B.P. 340,371, 11.11.29. U.S., 24.11.28).—Zones of high-tension insulators, *e.g.*, heads or threaded recesses, are sprayed, while hot from the mould, with a solution of a metallic salt, *e.g.*, stannous chloride, and annealed so that a continuous conducting coating is produced.

J. S. G. THOMAS.

**Dielectric material.** STANDARD TELEPHONES & CABLES, LTD., and L. E. SALAS (B.P. 340,302, 25.9.29).—As dielectric a vulcanised mixture composed of at least 25% by wt. of wax together with rubber (balata or gutta-percha) is claimed. Suitable proportions are: wax 80–50%, rubber 20–50%, sulphur 0.5–2%, accelerator 0.1–0.25%, zinc oxide 0.5–5%.

J. S. G. THOMAS.

**Electric condenser.** S. RUBEN, Assr. to RUBEN PATENTS Co. (U.S.P. 1,774,455, 26.8.30. Appl., 19.10.25).—A condenser comprising electrodes of sheet aluminium

separated by a film-forming dielectric composed of an electronegative compound of sulphur with copper and zinc is claimed. J. S. G. THOMAS.

**Electrical condenser.** P. E. EDELMAN, Assr. to E. BANNING (U.S.P. 1,770,465, 15.7.30. Appl., 23.11.27).—In the condenser described, two aluminium electrodes, one of which has a filmed coating of a magnesium alloy, are separated by a dielectric composed of fabric or fibrous sheet impregnated with a solid mixture of sodium phosphate, ammonium phosphate, ammonium dichromate, and malic acid. J. S. G. THOMAS.

**Electrically conductive article.** E. F. KIEFER, Assr. to NAT. CARBON CO., INC. (U.S.P. 1,774,381, 26.8.30. Appl., 29.3.27).—A conducting brush for electrical machines, *e.g.*, dynamos, composed of finely-powdered graphite, metal, or alloy bonded with soluble cellulose esters or ethers (especially the nitrate) is claimed. J. S. G. THOMAS.

**Manufacture of electrical resistances.** H. A. GILL. From STEATIT-MAGNESIA A.-G. (B.P. 340,433, 3.2.30).—A non-volatile carbon compound, *e.g.*, dextrin, sugar, or starch, together with, if desired, finely-divided pure carbon, is applied to an insulating material and heated at a red heat in an inert atmosphere, *e.g.*, of nitrogen, until an adherent, conducting layer of carbon is produced. J. S. G. THOMAS.

**Electrical resistances.** BRIT. & DOMINIONS FERALLOY, LTD., and J. W. BAMPFYLDE (B.P. 341,872, 21.10.29).—A resistance for use at temperatures above 700°, composed of a non-oxidising iron-aluminium alloy (11–16% Al), is claimed. J. S. G. THOMAS.

**Arc-welding electrode.** P. O. NOBLE and J. A. CAPP (U.S.P. 1,770,799, 15.7.30. Appl., 15.4.27).—The electrode is immersed in or passed through a bath of fused sodium nitrate, and/or potassium nitrate, and/or calcium nitrate, and then washed in water. J. S. G. THOMAS.

**Electrode for arc-welding.** E. W. SCHWARTZ and F. R. KAIMER, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,770,811, 15.7.30. Appl., 15.1.27).—Electrode wire is coated with flux by immersion in a solution composed of water 100, sodium silicate 5, ferric oxide 30, and lime 40 pts. by wt., at about 100°. J. S. G. THOMAS.

**Electric contact.** C. F. CARPENTER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,770,839, 15.7.30. Appl., 1.11.26).—The contacting surfaces of conductors, *e.g.*, of copper, are coated with cadmium. J. S. G. THOMAS.

**Electric-discharge [transmitting] apparatus.** BRIT. THOMSON-HOUSTON CO., LTD., Assces. of R. H. DOUGHERTY (B.P. 342,000, 1.11.29. U.S., 8.11.28).

**Vapour electric-discharge devices [with controlled discharge].** BRIT. THOMSON-HOUSTON CO., LTD., Assces. of W. F. WINTER, JUN. (B.P. 341,995, 31.10.29. U.S., 31.10.28).

**[Electrodes for] electric-discharge tubes having an electron discharge.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 340,936, 30.9.29).

**X-Ray tubes [with rotating anticathode].** N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 342,222, 3.5.30. Holl., 16.5.29).

**Electric insulators of ceramic material.** F. SINGER (B.P. 342,244, 24.6.30. Ger., 3.9.29).

**Drying of moist substances** (B.P. 340,057). **Fire extinguisher** (B.P. 340,216). **Heating of liquids** (B.P. 341,383).—See I. **Electrode carbon** (B.P. 319,275).—See II. **Removing copper oxide from copper surfaces etc.** (U.S.P. 1,773,160).—See VII. **Treatment of copper sulphide ores** (U.S.P. 1,769,604). **Reduction of zinc ore** (U.S.P. 1,773,779). **Treatment of manganese ores** (U.S.P. 1,770,791). **Contact points** (U.S.P. 1,774,689). **Magnet steel** (U.S.P. 1,773,793). **Magnetic cores** (B.P. 340,762). **Silver-plate** (B.P. 341,704). **Chromium-plate** (U.S.P. 1,774,901). **Nickel-chromium plating** (U.S.P. 1,774,269). **Electrolytic sodium** (B.P. 340,451). **Electrodeposition of metals** (B.P. 341,246).—See X.

## XII.—FATS; OILS; WAXES.

**Recognition of vegetable and animal fats.** L. AUERBACH (Chem. Umschau, 1931, 38, 11–12).—Attempts were made to detect the addition of animal fat to vegetable fats. Recovery of the sterols from their digitonides by means of xylene (*cf.* Wizöf Standard Methods) was not convenient; it was found better to convert the digitonides into acetates, saponify these with alcoholic potash, and extract the sterols with light petroleum. The m.p. of the sterols so recovered gave no reliable information, but microscopical examination of the recrystallised products was helpful. In many cases well-defined individual crystals of cholesterol and phytosterol occurred side by side; in other cases needle-shaped mixed structures occurred, and in one case only the conical mixed crystals described by Grün were observed. E. LEWKOWITSCH.

**Use of promoters in the hydrogenation of fats and vegetable oils.** V. S. SADIKOV (J. Appl. Chem., Russia, 1930, 3, 573–584).—When added to a nickel oxide catalyst cerium dioxide depresses, but lanthanum oxide promotes, the activity of the catalyst, although without increasing the yield of hydrogenated oil. The influence of various other substances in hydrogenation was studied. CHEMICAL ABSTRACTS.

**Determination of unsaponified oil in soap or fatty acids.** E. L. SMITH (Analyst, 1931, 56, 9–14).—The soap is decomposed by boiling with dilute hydrochloric acid and 1 g. of the resulting fatty acid is dissolved in 60 c.c. of 65% alcohol and 1 c.c. of 1:1 ammonia solution; the development of an opalescence or turbidity within 10 min. indicates the presence of more than 0.025% of unsaponifiable oil. The test may be made quantitative by using 2.5 g. of fatty acid and 2.5 c.c. of 1:1 ammonia solution and diluting to 100 c.c. with 99% alcohol. Four 20-c.c. portions are transferred to test-tubes and the remainder is titrated with water to incipient turbidity. The four portions are then treated with volumes of water 0.1–0.5 c.c. less than that required in the first test; from the volume of water required just to produce a turbidity in 5 min. the oil content is read off a previously prepared calibration curve drawn from results obtained with standard mixtures of oil and fatty acid.

A. R. POWELL.

**Sulphonated oils. VI. Method of determining alkali-combined sulphuric acid, based on solubility.** K. NISHIZAWA and K. WINOKUTI (Chem. Umschau, 1931, 38, 1—7).—The solubilities of the alkali salts of sulphuricinoleic acid in salt solutions, in the presence of ether, acids, etc., have been investigated, and are discussed in relation to the available methods for the determination of alkali sulphates. Imperfect separation of the solutions in the case of certain commercial sulphonated oils vitiates the usual gravimetric methods and Hart's volumetric scheme (cf. B., 1929, 947), and seems to be due to the relatively large content of the normal salt of sulphuricinoleic acid, which is somewhat soluble in brine and resists salting out. In the proposed new Japanese method (cf. A., 1930, 321) the oil is treated with hydrochloric acid (methyl-orange indicator) in order to decompose the ether-insoluble soaps of the fatty acids and to convert the normal alkali sulphuricinoleates into acid salts, which are insoluble in brine (cf. *loc. cit.*; any free sulphuricinoleic ester produced by a small excess of acid does not affect the result of the analysis, but preferably this excess is neutralised with sodium acetate). The mixture is then shaken with ether and saturated brine (and salt), and the alkaline sulphate determined as usual. The usefulness of Kern's method (B., 1920, 632 A) is confirmed, and the procedure justified theoretically, but a more concentrated sodium monophosphate solution and the use of ether is recommended. With these modifications the analysis can be applied to such oils as Appret. Avirol E., with which other methods fail. Acid salts of sulphonic esters of hydroxy-acids are readily prepared by treating the free esters with alkali acetate and salting out the acid salts formed. Hart's criticism (B., 1929, 988) of the authors' formula for determining organically combined sulphur is answered. E. LEWKOWITSCH.

**Potentiometric determination of the saponification value of mixtures of asphalt and drying oils.** J. V. DEMAREST and W. RIEMAN (Ind. Eng. Chem. [Anal.], 1931, 3, 15—17).—The saponification value of asphalt may be determined by potentiometric titration with hydrochloric acid, using the hydrogen electrode as indicator, after saponification with alcoholic potassium hydroxide solution; anisole is the most satisfactory solvent for the asphalt. Standardisation of the alkali is unnecessary, as the titration curve exhibits peaks corresponding with the neutralisation of the excess and the free alkali, respectively. After each addition of acid a period of 9 min. is required for the establishment of a steady potential. The method is equally applicable to linseed oil and to mixtures of the oil with asphalt, but with mixtures containing soya-bean oil the results are unsatisfactory. H. F. GILLBE.

**Determination of minute quantities of dissolved phosphorus in oil.** C. STICH (Pharm. Ztg., 1931, 76, 112).—Although possible sources of error have been pointed out, exact adherence to the author's method (B., 1927, 851) gives satisfactory results if the solvents used are of D.A.B. VI standard and are free from traces of aldehyde. H. E. F. NOTTON.

**Japanese beeswax. I. General properties.** H. IKUTA (Chem. Umschau, 1931, 38, 7—10).—The work,

of which an abstract has already appeared (B., 1931, 125), is described and discussed in full detail. True Japanese beeswax is derived from the bee, *Apis indica*, Fab. var. *Japonica* "Rads," 1887 (peculiar to Japan and Korea), and resembles oriental beeswax ("Ghedda wax") in its properties. E. LEWKOWITSCH.

**Fatty acids from oxidised paraffin wax.** GUTT and PLOTKO. **Viscosity of oils.** SCHLENKER. **Dilution and viscosity of oils.** TAUSZ and RABL.—See II.

#### PATENTS.

**Metal compounds of fatty acids.** P. SPENCE & SONS, LTD., and T. J. I. CRAIG (B.P. 338,919, 21.5.29).—Stearic acid (etc.) is mixed at 80° with a solution of sodium aluminate (or other "metallate") and the product, after drying, is used to increase the viscosity of lubricating oils or as an ingredient of polishes. Normal and basic sodium aluminium stearates are described. [Stat. ref.] C. HOLLINS.

**Production of emulsions [lanolines].** T. GOLDSCHMIDT A.-G. (B.P. 339,558, 21.2.30. Ger., 9.3.29).—Cold creams are made by emulsifying, in the presence of oleic  $\beta$ -diethylaminoethylamide (or other acylated alkylendiamine), such synthetic esters of polyhydric alcohols with high-molecular saturated or unsaturated fatty acids as contain unesterified hydroxyl groups, together with the usual addenda. An example is: glycerol monostearate (18%), wax (1%), petroleum jelly (6%), wool grease (4%), paraffin oil (7%), almond oil (5%), glycerol (3%), oleic  $\beta$ -diethylaminoethylamide phosphate (0.5%), water or 0.1N-citric acid (55.5%). C. HOLLINS.

**Imparting the properties of wood oil to fatty oils having a drying power.** J. SCHEIBER (B.P. 316,872 and 338,932, 23.7.29. Ger., [A] 4.8.28, [B] 21.5.29).—Drying oils, such as linseed, poppyseed, or fish oils, are treated (A) with halogen, hydrochloric acid, sulphuric acid, hypochlorous acid, etc. and the inorganic substituent is subsequently removed so as to generate a double linking in a conjugated position, or (B) with small quantities of inorganic acids or salts (sulphuric acid, sulphates), halogens (iodine, iodine chloride or bromide), metal or metalloidal halides, metals (iron, nickel, zinc, tin), metal oxides (alumina, mercuric oxide), or mixtures of these, at 200—320°, with like effect. In either case the process may be applied to emulsions of the oils, or to corresponding acids which are subsequently esterified with glycerol etc. C. HOLLINS.

**Production of [castor oil] lubricant.** R. R. ROSENBAUM (U.S.P. 1,773,203, 19.8.30. Appl., 18.6.28).—Castor oil is heated under reduced pressure (5—27 in.) at 228—343° with 10% of a suitable distilling agent, such as a petroleum hydrocarbon having a distillation temperature below the decomposition temperature of the oil, e.g., kerosene or turpentine. Albuminous and gummy matter and aldehydes are distilled over and the non-gumming residual castor oil is miscible in all proportions with medicinal or lubricant petroleum hydrocarbons. E. LEWKOWITSCH.

**Composition [from cashew nut-shell liquid] and its manufacture and use.** M. T. HARVEY, Assr. to HARVEL CORP. (U.S.P. 1,771,786, 29.7.30. Appl., 8.1.30).

—Cashew nut-shell liquid is heated at 115–205° with sulphur, yielding sticky liquids or resilient products resembling vulcanised rubber, the consistency varying with the proportion of sulphur used. The cashew nut-shell liquid may be preheated, *e.g.*, to 315°, especially in cases when high temperatures in later stages must be avoided. The dried products are insoluble in alcohol, gasoline, etc., and may be prepared with or without admixture of fillers for coatings, moulded compositions, etc. E. LEWKOWITZCH.

**Mineral oil emulsion** (U.S.P. 1,773,123).—See II. **Liquid preparations** (B.P. 316,132). **Wetting etc. agents** (B.P. 318,610 and 341,978—9). **Emulsifying agents** (B.P. 313,966).—See III. **Bleaching agents** (B.P. 339,336).—See VI. **Cocoa by-products** (B.P. 341,000).—See XIX.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Preparation of phosphorescent zinc sulphide.** J. EINIG (Chem.-Ztg., 1931, 55, 31).—A 7% solution of zinc sulphate crystals is treated at 50° with a small quantity of hydrogen sulphide to remove heavy-metal impurities with a small proportion of the zinc. After filtration the purified solution is saturated with hydrogen sulphide and the resulting crystalline zinc sulphide is collected, washed, dried, and ignited at 900° in a non-oxidising atmosphere, after mixing with 0.00001–0.001% of copper. The product is a highly sensitive phosphorescent powder, retaining its phosphorescence overnight after exposure to ordinary daylight in a room during the day. A. R. POWELL.

**Water-resistant paints.** H. WOLFF (Farben-Chem., 1930, 1, 20–22).—The water-resistance of a paint film is dependent jointly on the following four factors: porosity of film (direct transmission of water), tendency of film to swell (indirect transmission of water), "solidity" of film (true resistance to water), and adhesion. Thus by addition of treated tung oil to a linseed oil paint, porosity and swelling tendency are diminished, but adhesion is weakened. The water-resistance (on continuous and intermittent immersion) of two-coat systems comprising white lead paint, alone and with the addition of 10 and 20% of a proprietary paraffin dispersion, "Enkaustin," using all the possible rearrangements of these as undercoats and finishing coats, was determined. The system white lead undercoat-white lead + 10% of "Enkaustin" finishing coat was the most resistant. It is pointed out that the water-resistance of the undercoat must not be raised to the point where adhesion suffers, and that a protective action on the undercoat should be exercised by a water-resistant finishing coat. S. S. WOOLF.

**Measurement of abrasion resistance.** I. Paints, varnishes, and lacquers. A. E. SCHUH and E. W. KERN (Ind. Eng. Chem. [Anal.], 1931, 3, 72–76).—In the method described for determination of the abrasion resistance of materials which can be obtained in the form of thin films a carborundum-air blast is allowed to impinge on the material at a constant angle until wear has taken place through unit thickness; the weight of abrasive used is taken as a measure of the abrasion resistance. Study of the influence of particle size and

other variables indicates that the best conditions are air pressure 6 cm., rate of flow of carborundum 24 g./min., and particle size of carborundum 170–200-mesh. For films of paint and similar substances both the humidity and temperature of the air supply must be controlled, and in all cases the nozzle must be carefully designed; a straight air tube is essential for consistent results. The average deviation in a series of 116 determinations was less than 1%. Results are given for a number of different varnishes and lacquers, together with the influence of ageing and humidity. At 41% humidity a rise of temperature from 70° to 90° F. causes an increase of the abrasion resistance of varnishes and a decrease in the case of japan, lacquers, and baked varnish; all the substances examined soften as the temperature is raised. With increase of humidity amberol lacquers show a slight increase of abrasion resistance, whilst for the ester gum lacquers, japan, and baked varnish there is a minimum at about 65% humidity; in all cases the hardness diminishes as the films take up moisture. H. F. GILLBE.

**Arsenic-containing writing materials.** G. KAPPELLER (Z. Unters. Lebensm., 1930, 60, 213–215).—Violet carbon duplicating papers and typewriter ribbons from various sources have been found to contain appreciable amounts of arsenic. Reckoning the paper base to be 50% of the total weight, the arsenic content of the colouring matter varied from 2.5 to 10% (calc. as As<sub>2</sub>O<sub>3</sub>). H. J. DOWDEN.

**Evaporation process [in varnish film formation].** A. V. BLOM (Farben-Ztg., 1931, 36, 873–875).—Although the physical and chemical processes constituting the drying of films of oil varnish etc. cannot be regarded as independent of each other, an attempt is made to define the evaporation processes mathematically, and to explain them by assuming that (a) the quantity volatilised in unit time remains constant, and (b) at any given instant the velocity of film formation is dependent on the amount of volatile matter remaining. Results obtained by earlier workers and by the author are discussed from this viewpoint. S. S. WOOLF.

**Colorimetric determination of turpentine vapour in the air.** I. M. KORENMAN (Z. anal. Chem., 1930, 82, 429–438).—The method recommended by Bogatsky and Biber (B., 1929, 255) is shown to yield unsatisfactory results owing to the variable nature of the oil and to the ease with which the more volatile constituents oxidise on exposure to the air with the formation of substances which produce other colours with vanillin and hydrochloric acid. There are also many difficulties associated with the preparation of the standard solution, neither the original oil nor the more volatile fractions yielding a satisfactory comparison solution. A. R. POWELL.

**Protective coatings on iron pipes.** BECK.—See X. **Saponification value of asphalt-drying oil mixtures.** DEMAREST and RIEMAN.—See XII.

### PATENTS.

**Undercoating for accelerating the time of drying of oil paints and enamels.** P. JAEGER (U.S.P. 1,772,897. 12.8.30. Appl., 29.6.26. Ger., 23.3.26).—An oil-free ground coat, which may be applied over an

old coat of paint, consists of a solution of nitrocellulose and resins (as fillers), containing 1—1½% of driers, *e.g.*, metal oleates. The finely-divided driers remaining after evaporation of the solvent accelerate the hardening of the finishing coat applied subsequently.

E. LEWKOWITSCH.

**Separation of certain [oxygenated] components from pine oil.** I. W. HUMPHREY, Assr. to HERCULES POWDER Co. (U.S.P. 1,772,546, 12.8.30. Appl., 26.7.28).—Pine oil is heated at, *e.g.*, 190—225° for 2—8 hrs. with 0.05—1% of hydriodic acid, which breaks down the terpeneols present into water and hydrocarbons. The hydrocarbons, including those originally present in the pine oil, are removed by distillation, and fenchyl alcohol, borneol, methylchavicol, and anethole are recovered from the residue by fractionation and recrystallisation.

E. LEWKOWITSCH.

**Production of a turpentine substitute [and borneol] from pine oil.** I. W. HUMPHREY, Assr. to HERCULES POWDER Co. (U.S.P. 1,772,895, 12.8.30. Appl., 20.7.26. Cf. U.S.P. 1,772,546; preceding abstract).—Pine oil is dehydrated by heating with 0.5—25% of, *e.g.*, fuller's earth or kieselguhr, while distilling off (preferably by the use of steam at, *e.g.*, 170—225°) the hydrocarbons formed. Of the distillate, having  $d_{45}^{25}$  0.885—0.890, 65—95% distils over below 185°; it may be used as a turpentine substitute. The high-boiling residue (10—35% of the original pine oil) may be used in disinfectants, or borneol may be separated from it by fractionation and recrystallisation.

E. LEWKOWITSCH.

**Manufacture of artificial masses [from styrene etc.].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 341,420, 14.10.29).—Polymerisation of styrene or its homologues is effected in the presence of *cyclohexanol* or a homologue. The products are generally turbid, but clarity can be ensured by incorporating a plasticising agent, such as is customarily used with nitrocellulose, *e.g.*, camphor, or an organic acid or acid anhydride, *e.g.*, acetic anhydride.

D. F. TWISS.

**Preparation of coumarone-indene resins.** R. W. OSTERMAYER, Assr. to NEVILLE CHEM. Co. (U.S.P. 1,770,281—3, 8.7.30. Appl., [A] 20.6.29, [B, C] 27.6.29).—(A) Better control of the process described in U.S.P. 1,705,857 (B., 1929, 651) is obtained if the coal-tar distillates are diluted with a petroleum distillate (ranging from gasoline to wash-oil cuts) before or during the polymerisation; polymerisation can then be carried practically to completion and naphthas with coumarone-indene contents over 80% can be treated. (B) Petroleum distillates lower and (C) higher in the Baumé scale than petroleum benzine are used as diluents after polymerisation of the naphtha required for the process mentioned in (A). The resin produced in (B) is paler and of higher m.p. than that in (A), but polymerisation cannot be carried as near completion by the former process.

E. LEWKOWITSCH.

**Production of resin esters.** A. C. JOHNSTON, Assr. to HERCULES POWDER Co. (U.S.P. 1,771,044, 22.7.30. Appl., 29.6.28. Renewed 23.8.29).—Resin esters are produced by heating (under pressure or by refluxing) rosin, resin acids, or their alkali salts with glycerol dichlorohydrin in the presence of an alkali, such as

sodium hydroxide or carbonate, and in solution in alcohol or water.

E. LEWKOWITSCH.

**Furfural resin composition.** C. ELLIS (U.S.P. 1,771,033, 22.7.30. Appl., 18.1.24).—A soluble, fusible resin is prepared by heating furfuraldehyde and phenol (in proportions about 3:4 pts.) or tar acids, cresols, etc., at 135—155° in the presence of a fixed alkali, *e.g.*, the hydroxides, carbonates, or oxides of sodium, barium, or calcium; fillers, and/or hexamethylenetetramine, and a small amount of fixed alkali may be incorporated in the product. An insoluble, infusible resin is produced by heating with a larger proportion of the aldehyde and for a longer time.

E. LEWKOWITSCH.

**Decorative painting [with paints containing glass].** J. LIDBETTER (B.P. 342,271, 28.10.29).

**Drying of chemicals [e.g., lithopone]** (B.P. 341,140).—See I. Bituminous dispersions (B.P. 341,443).—See II. Wetting etc. agents (B.P. 318,610 and 341,978—9).—See III. Red lead (U.S.P. 1,770,777). **Titanium compounds** (U.S.P. 1,773,727).—See VII. **Protection of metals etc.** (B.P. 342,235).—See X. **Insulating material** (B.P. 341,535 and 342,290).—See XI. **[Coating] composition** (U.S.P. 1,771,786).—See XII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Behaviour of anti-oxidants in rubber stocks containing copper.** P. C. JONES and D. CRAIG (Ind. Eng. Chem., 1931, 23, 23—26).—Experiments on the accelerated ageing of technical rubbers of several types, with and without the addition of 0.1% or 0.2% of copper stearate, showed that secondary amines, *e.g.*, diphenyl-*p*-phenylenediamine or phenyl- $\beta$ -naphthylamine, were particularly effective in retarding deterioration caused by copper. The effectiveness of such an anti-oxidant in preventing cracking on repeated flexure is explained by preferential adsorption of the anti-oxidant at the active interfaces which normally would give rise to earliest separation.

D. F. TWISS.

**Paragutta, a raw insulating material for submarine cables.** A. R. KEMP (J. Franklin Inst., 1931, 211, 37—57).—By heating rubber or rubber latex with water in an autoclave the rubber protein is gradually destroyed; diluted latex which has been heated for 10 hrs. at 150°, after coagulation gives a washed rubber with less than 0.1% N, or about one fourth the normal content. Such rubber is very resistant to water and maintains a high dielectric constant and electrical resistivity when kept under salt solution. Paragutta is the name given to a mixture of approx. equal quantities of deresinated gutta or balata and deproteinised rubber together with up to 40% of purified montan wax or up to 20% of hydrocarbon wax. Paragutta has all the desirable thermoplastic and mechanical properties of gutta-percha, but is cheaper and has such superior insulation characteristics as to make it suitable for long-distance submarine cables.

D. F. TWISS.

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**Treatment of [rubber] latex.** ANODE RUBBER Co., LTD., Assrs. of H. C. HOWARD (B.P. 341,744, 12.2.30. U.S., 8.3.29).—The alkalinity of preserved latex is



reduced by treatment with an adsorbent such as silica gel. The adsorbent, which should be prepared for use by heating, *e.g.*, at 105° for 30 min., is preferably in particles between 0.5 and 3 mm. in diam., so that it may be removed subsequently by sieving. The recovered adsorbent can be regenerated for further use.

D. F. TWISS.

**Process for concentration of [rubber] latices.** K.D.P., LTD. (B.P. 341,677, 13.12.29. U.S., 19.12.28).—Water-soluble concentration products of latices of vegetable resins, especially rubber, are produced with the aid of potassium salts of hydrotropic compounds, *i.e.*, compounds containing a hydrophilic group, *e.g.*, hydroxyl or carboxyl, together with a hydrophobic complex such as the benzene or naphthalene nucleus or an aliphatic chain containing at least 10 carbon atoms. The hydro-tropic potassium salt should be more effective than potassium thiocyanate in retarding the setting of gelatin.

D. F. TWISS.

**Manufacture of vulcanised articles from rubber and similar materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,258, 9.1.30).—Montan wax which has been bleached by oxidation, *e.g.*, by treatment in glacial acetic acid with chromic acid, is much superior to ordinary montan wax in facilitating the ordinary mixing operations with natural or synthetic rubber and imparts exceptional resistance to water absorption, attrition, and ageing. It may be introduced to the extent of 2–10% into the rubber prior to or during the incorporation of the other compounding ingredients; if desired, it may be worked into rubber which has been previously swollen with a solvent such as benzene.

D. F. TWISS.

**Reconditioning of old vulcanised rubber.** F. J. CLEVELAND. From A. W. MORTON (B.P. 340,985, 7.8.29).

The rubber is comminuted and mixed with about 4% of sulphur, and the mixture is treated with approx. 2–5% of a mixture of benzene, carbon disulphide, and carbon tetrachloride. Compounding ingredients may then be introduced. After 12 hrs. or so the mass is finally moulded and vulcanised.

D. F. TWISS.

**Dielectric material** (B.P. 340,302).—See XI. [Rubber-like] composition (U.S.P. 1,771,786).—See XII.

## XV.—LEATHER; GLUE.

**Enumeration of bacteria in soak waters [for animal skins].** G. E. ROCKWELL (J. Amer. Leather Chem. Assoc., 1931, 26, 2–7).—The soak water is agitated from the bottom up with a plunger, and samples of the plunger liquor and soaked skin are collected immediately in a sterile jar, which is then revolved in a shake machine for 10 min. Dilutions of 1 : 10–1 : 10<sup>6</sup> are then prepared and 1 c.c. of each of a suitable range of dilutions is plated on a meat extract-agar medium adjusted to +0.5 on the Fuller scale, in a Petri dish. Three dilutions are usually necessary for each count; the range chosen depends on the character of the soak water, but should be such as to give 50–150 colonies on incubation. When the medium has set, the plates are incubated for 24 hrs. at 37° and for 24 hrs. at room temperature. Plates with 50–150 colonies are then counted and the number of bacteria per c.c. of original soak water is calculated therefrom. D. WOODROFFE.

**Quick tannage.** M. BERGMANN, W. MÜNZ, and L. SELIGSBERGER (Collegium, 1930, 520–524).—The speed of penetration of 1–5% solutions of commercial Neradol ND and of pure naphthalene-1- and -2-sulphonic acids, respectively, was determined on hide pieces. The penetration of the sulphonic acid was greatest for the last-named compound. By determinations of the swelling and the imbibed water it is deduced that the hide capillaries are enlarged by the above products and the hide is rendered thicker, less compressible, and more distended. The hide is similarly affected by other acid solutions of similar *p<sub>H</sub>* value, but these diminish the porosity of the hide. Tannage can be accelerated by substances which will disperse the tanning materials or by sulphonic acids which increase the porosity and penetrability of the pelt.

D. WOODROFFE.

**Qualitative analysis of tannins.** [Report of a Committee of the Internat. Verein der Leder-Ind.-Chemiker.] O. GERNGROSS (Collegium, 1930, 524–532).—Mixtures of quebracho and sulphite-cellulose waste extracts were tested for sulphite-cellulose by means of the Procter-Hirst, cinchonine, and fluorescence reactions. As little as 4% of sulphite-cellulose in the mixture could be detected by the Procter-Hirst reaction, but 5% was required to show a positive reaction with the other tests. A positive test was shown with sulphited quebracho by all three reactions, but by the Procter-Hirst and cinchonine reactions the test was positive only with an extract prepared by sulphiting the insoluble matter in a quebracho extract. Publications on qualitative analysis are reviewed for the last two years.

D. WOODROFFE.

**Sediment in tanning extracts.** W. VOGEL (Gerber, 1930, 56, 129–132; Chem. Zentr., 1930, ii, 2600).—The sediment in tanning extracts (*d* up to 1.107) was determined by Kubelka and Némec's method. Different portions from the same material differ as regards the amount of sediment formed and its dependence on the concentration. The determination of insoluble matter in tanning extracts is not a measure of the amount of deposit formed in practice.

A. A. ELDRIDGE.

**Leather for gas-meters.** ANDREWS.—See II.

## PATENTS.

**Tannage of chamois, buckskins, or other leather.** A. ERNST (U.S.P. 1,771,490, 29.7.30. Appl., 8.5.28).—The limed, washed, and pickled skins are treated with formaldehyde for 45 min., then an emulsion of degreas in soap and soda is added to the formaldehyde bath, and the treatment is continued till the skins are tanned.

D. WOODROFFE.

**Manufacture of gelatin products.** O. HAAVARDSSHOLM (U.S.P. 1,770,573, 15.7.30. Appl., 30.6.28. Ger. and Norw., 7.5.28).—Gelatin is granulated or pulverised, the crumbs are stored in an atmosphere of 60–70% R.H. and at 21–27°, then compressed at 20,000–30,000 lb./in.<sup>2</sup> into tablets, and these are subjected to the action of wet steam and rapidly dried.

D. WOODROFFE.

**Plastic casein composition.** J. G. DAVIDSON and E. W. REID, Assrs. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,772,131, 5.8.30. Appl., 3.1.28).—Casein is mixed with 5% of water and triethanolamine or a mixture of "ethanolamines."

D. WOODROFFE.



Tar-removal from leather (B.P. 338,167).—See II. Azo dyes for leather (B.P. 338,930 and 339,029).—See IV. Dyeing of skins etc. (B.P. 339,444).—See VI.

## XVI.—AGRICULTURE.

**Tropical soils. I. Identification and approximate determination of sesquioxide components by adsorption of alizarin.** F. HARDY (J. Agric. Sci., 1931, 21, 150—166).—Free alumina in clays and soils absorbs alizarin only after ignition, whereas free ferric oxide absorbs it only in the fresh unignited state. A method of determining the sesquioxides is based on this difference (cf. Schmelev, B., 1928, 895). Examination of numerous soils and their clay fractions is recorded. In most cases the amounts of free alumina present were small and only moderate proportions of iron occurred as the free hydrated oxide. Of the materials examined bauxite and laterite contained the largest proportions of free oxides. The clay fractions of soils contained rather less free alumina but more free ferric oxide than the whole soils. A. G. POLLARD.

**Electrical conductivity of aqueous soil suspensions as a measure of soil fertility.** A. SEN and C. H. WRIGHT (J. Agric. Sci., 1931, 21, 1—13; cf. B., 1928, 870).—The electrical conductivity of soil suspensions and the "7-days' increase" (increase in conductivity after storage of suspension for 7 days at 25°) for a number of soils are recorded. The initial conductivity of an unmanured soil under continuous cropping steadily decreases, reaching a value which remains constant for many years. Continuously manured soils show a similar decline (except for temporary increases following applications of manures), reaching a constant value slightly higher than that of corresponding unmanured soils. The "7-days' increase" also becomes steadily smaller in both manured and unmanured soils, the decrease in the latter case being notably rapid in the early years of continuous cropping. The 7-days' increase for manured soils is consistently higher than that of the unmanured, dung producing greater differences than those given by artificial fertilisers. The 7-days' increase is closely correlated with the crop yield. An impoverished soil allowed to "run wild" shows a steady rise in the initial conductivity and the 7-days' increase. Prolonged storage of air-dry soil does not appreciably affect the initial conductivity, but the 7-days' increase rises rapidly to a fairly constant value in the first few months of storage. A. G. POLLARD.

**"Sticky-point" water of soils.** B. DE C. MARCHAND (S. Afr. J. Sci., 1930, 27, 183—193).—The water content, by vol., of soil at the "sticky point" is determined by weighing a block of soil at the sticky point in water and in xylene. From values so obtained the water capacity, pore space, and clay content are calculated. Sticky-point moisture was closely correlated with the clay content of soils, but not with the "loss on ignition." The sticky-point water of soils examined was closely related with the water capacity (cf. Keen and Raczkowski, B., 1922, 70 A), but yielded values of approximately two thirds of the water capacity. At the sticky point the volume of water held in the interstitial spaces of a colloid-free soil approximates to the pore space of an "ideal" soil (26%). A. G. POLLARD.

**Preparation of permanent records of soil colour.** C. L. WHITTLES (J. Agric. Sci., 1931, 21, 189—190).—A few drops of a suspension of the soil in water are filtered on a filter-paper disc in a Gooch crucible to form a thin and even film of soil. After drying, the disc is removed and coated with celluloid varnish.

A. G. POLLARD.

**Determination of hydrogen-ion concentration by means of the Haber-Klemensiewicz glass cell, with special regard to soil investigations.** G. HATOS and G. GOLL (Magyar chem. Fol., 1930, 36, 33—39; Chem. Zentr., 1930, ii, 121).—The Haber-Klemensiewicz cell can be used for determinations of hydrogen-ion concentrations. The  $p_H$  values for 15 soils determined by this means were practically in agreement with those obtained with the quinhydrone electrode, especially in dilute aqueous suspensions; in thick suspensions fluctuations amounting to 0.2—0.3 unit occurred.

L. S. THEOBALD.

**Influence of superphosphate on the reaction, base saturation, and buffer power of acid mineral soils.** L. SCHMITT (Superphosphat, 1931, 6, 219—227).—Continuous application of superphosphate did not appreciably affect the  $p_H$ , buffer capacity, or exchangeable-base content of acid mineral soils, nor did it reduce the effectiveness of dressings of lime on these soils.

A. G. POLLARD.

**Measurement of the drillability of fertilisers.** A. L. MEHRING (Ind. Eng. Chem. [Anal.], 1931, 3, 34—38).—The drillability of a fertiliser, i.e., the resultant of all factors influencing its rate of flow, is determined mainly by the coefficient of friction, apparent density, and particle size. A theoretical equation is given relating these factors to the time of flow of unit quantity through an orifice under the influence of gravity alone, and an empirical equation is given for the time of delivery of unit quantity when pressure has to be applied to maintain a steady flow. Appliances and methods for determination of the angle of repose, and hence of the coefficient of friction, and of the apparent density are described and an account is given of the calculation of the mean particle size from the percentages which pass sieves of various meshes.

H. F. GILLBE.

**Evaluation of the available phosphoric acid content of fertiliser materials.** C. C. HOWES and C. B. JACOBS (Ind. Eng. Chem. [Anal.], 1931, 3, 70—72).—Results obtained by the standard method for insoluble phosphate in superphosphate containing ammonia fall by 50—70% if the usual 2 g. sample be replaced by 1 g. Alterations of the  $p_H$  of the citrate solution between 4.5 and 8.8 cause great differences among the results, although in absence of ammonia no such effect is observed; similar discrepancies occur on variation of the quantity of citrate solution used and the period of digestion. It appears that an entirely new method must be developed for ammoniated superphosphate fertilisers.

H. F. GILLBE.

**[Fertiliser trials.]** R. TRNKA, L. STETSKAL, and J. CHREUST (Superphosphat, 1930, 6, 228—230).—Ammonium sulphate was preferable to sodium nitrate as a source of nitrogen for oats. Superphosphate increased the total crop and, in particular, the proportion of grain. Better proportional utilisation of soluble phosphates

was observed in podsols when superphosphate was applied in larger amounts than is customary.

A. G. POLLARD.

**Relation of nitrogen fertiliser to the firmness and composition of strawberries.** J. S. SHOEMAKER and E. W. GREVE (Ohio Agric. Exp. Sta. Bull., 1930, No. 466, 20 pp.).—Spring applications of nitrogenous fertilisers to strawberry plants increased the nitrogen content and reduced the acidity of the fruit and increased the catalase activity of both fruit and foliage. The fruit was slightly softer, but its travelling quality was not appreciably affected. The total and reducing sugar contents of the fruit were increased by the fertiliser, and the percentage of all sugars increased with successive pickings. The yield of berries was not increased by spring applications of nitrogen, but marked increases resulted from autumn applications.

A. G. POLLARD.

**Potash in relation to cotton wilt.** M. N. WALKER (Florida Agric. Exp. Sta. Bull., 1930, No. 213, 10 pp.).—Infection of cotton by *Fusarium vasinfectum* is not reduced by applications of potash fertilisers.

A. G. POLLARD.

**Cabbage fertiliser experiments.** A. B. FITE (New Mex. Agric. Exp. Sta. Bull., 1930, No. 180, 28 pp.).—Highest yields were obtained by the use of cottonseed meal and with farmyard manure supplemented with minerals. Earliness of maturity was closely associated with the total yields obtained.

A. G. POLLARD.

**Action of inorganic sprays on the germination and early growth of cereals.** H. LUNDEGÅRDH, H. BURSTRÖM, and H. EKSTRAND (Kungl. Landtbruks-Akad. Hand. Tidskr., 1930, 69, 602—632; Chem. Zentr., 1930, ii, 2176).—Stimulation was not observed with certainty. High concentrations retard germination. Grain which germinates most rapidly is most sensitive to the toxic action.

A. A. ELDRIDGE.

**Chemical composition of some N. Dakota pasture and hay grasses.** T. H. HOPPER and L. L. NESBITT (N. Dakota Agric. Exp. Sta. Bull., 1930, No. 236, 39 pp.).—A discussion of published data concerning variations in composition of numerous grasses with age and climatic conditions and of the effects of grazing.

A. G. POLLARD.

**Microbiology of farmyard manure decomposition in soil.** I. Changes in the microflora, and their relation to nitrification. II. Decomposition of cellulose. H. L. JENSEN (J. Agric. Sci., 1931, 21, 38—80, 81—100).—I. During the decomposition of manure in neutral or slightly acid soils there was a rapid multiplication of bacteria and to a less extent of actinomycetes, and subsequently a sharp decline to a level approaching that of untreated soil. The initial increase in bacterial numbers was especially great where the manure contained fresh straw. The fungal population of soil was not affected by applications of manure unless fresh straw was included, when there was a considerable increase especially in acid soils. Nitrification of manure nitrogen occurred when bacterial numbers were declining, but the process was never complete. The  $\alpha$ -humus produced from manure contained 18—25% of the original nitrogen in an inert form which was not decomposed in 6—12 months. The humus fraction of manure

consisted mainly of lignin combined with protein matter. The rate and extent of the nitrification of manure was largely influenced by its C:N ratio. "Edelmist," having a lower C:N ratio than ordinary manure, was more rapidly nitrified, although its nitrogenous constituents were not more easily decomposable. During decomposition in soil the C:N ratio of manure tends to approach 11—12:1 and no loss of nitrogen occurs. The relatively low fertilising value of the organic nitrogen of farmyard manure is ascribed to the utilisation of the available nitrogen by bacteria, which decline in numbers as the supply ceases. Mineralisation of this nitrogen then begins, but diminishes in rate and practically ceases before the whole of the nitrogen is attacked.

II. Decomposition of cellulose in soil is accompanied by a rapid increase in the numbers of organisms concerned, viz., those of the genus *Vibris* in soils of  $pH$  6.5—7.0, *Spirochaeta cytophaga* in soils of  $pH$  5.7—6.2, and of fungi in more acid soils. Both bacteria and fungi decompose the lignocellulose of straw. The nitrogen requirements of cellulose-decomposing bacteria are not smaller than those of the fungi. The ratio cellulose decomposed: nitrogen assimilated was 25—54:1. Two fungi, but no bacteria, formed humus-like compounds when grown on filter paper.

A. G. POLLARD.

**Potato scab and manuring.** EICHINGER (Superphosphat, 1931, 7, 8—16).—Effects of 9 years' continuous application of various fertilisers on the development of scab in potatoes are recorded. Superphosphate, ammonium sulphate, potassium magnesium sulphate, and probably potassium sulphate reduce scab infection. Potassium chloride, if exhibiting any influence at all, favours scab development. Chalk and basic slag, according to its free lime content, favour the spread of scab and reduce the beneficial effects of ammonium sulphate in this respect. Heaviest scab infections followed the use of sodium nitrate. Calcium cyanamide has a similar but less active effect.

A. G. POLLARD.

**Manuring of fish ponds.** D. BRÜNING (Enähr. Pflanze, 1931, 27, 49—54).—A discussion of observed results and of the conduct of experiments.

A. G. POLLARD.

**Influence of manuring on drug yields and essential oil content of *Anthemis nobilis*.** L. O. DAFERT and M. BRANDL (Angew. Bot., 1930, 12, 212—215; Chem. Zentr., 1930, ii, 2157).—Cultural conditions are described. Young blossoms contain more oil than old blossoms. The plant is very sensitive to phosphorus.

A. A. ELDRIDGE.

**Influence of temperature and moisture on the nature and extent of decomposition of plant residues by micro-organisms.** S. A. WAKSMAN and F. C. GERRETSEN (Ecology, 1931, 12, 33—60).—The rate of decomposition of oat straw by soil micro-organisms increases with temperature. In 9 months at 37°, 50—60% decomposition of lignin occurs, whereas at 7° the material remains practically unchanged. Addition of available nitrogen increases the rate of decomposition of hemicellulose and cellulose, but that of lignin is not appreciably affected. Lower temperatures favour the synthesising activities of the organisms. At higher temperatures decomposition processes predominate.

The bearing of the above on the nature of humus produced under different climatic conditions is discussed.

A. G. POLLARD.

**Cultivation of the sugar beet crop : three years' investigations of the effects of spacing.** W. M. DAVIES (J. Min. Agric., 1931, 37, 973—985).—The optimum spacing of sugar beet in the West Midlands is probably 18 in. between the rows and 9 in. in the rows. Spacing has no significant effect on the sugar content of beet.

E. HOLMES.

**Winter spraying for the control of the apple capsid bug.** F. R. PETHERBRIDGE and G. L. HEY (J. Min. Agric., 1931, 37, 1078—1087).—From the summarised results of a number of trials it appears that a winter wash containing a tar distillate and a mineral oil (approx. 7.5% of each) is best for the control of apple capsid bug, aphides, and red spider.

E. HOLMES.

**Extermination of matgrass (*Nardus stricta*, L.).** M. MALOCH (Enähr. Pflanze, 1931, 27, 54—56).—Autumn applications of kainite rapidly exterminated matgrass from pastures.

A. G. POLLARD.

**Sulphur dusting for the prevention of a bacterial disease of wheat, called Black Chaff.** F. J. GREANEY (Sci. Agric., 1931, 11, 274—280).—Repeated dusting with 15 lb. of sulphur per acre largely prevented the spread and development of the disease.

A. G. POLLARD.

**Toxic effect of inorganic substances on spores of *Tilletia tritici*.** H. BURSTRÖM (Kungl. Landtbruks-Akad. Hand. Tidskr., 1929, 68, 651—667; Chem. Zentr., 1930, ii, 2176).—The toxicity falls according to the series (a) mercury, nickel, cobalt, copper, lead, palladium, zinc, cadmium, manganese, and (b) gold, chromium, ferric iron.

A. A. ELDRIDGE.

**Examination of tar and mineral oil insecticides.** H. MARTIN (J.S.C.I., 1931, 50, 91—94 T).—A method for determining the oil content of mineral oil insecticides by ether extraction of the emulsion treated with caustic soda is described. Factors which determine the suitability of the oil for spray purposes are discussed and methods are given for the determination of constants, e.g., specific gravity, viscosity, volatility, content of aromatic and unsaturated hydrocarbons, ease of oxidation, presence of sulphur compounds, deemed to be of value for this purpose. The oil used for the determination of these constants is obtained, when possible, by breaking the emulsion by the addition of saturated sodium chloride. The methods are applied to the determination of content of neutral tar oils, tar acids, and tar bases of tar-distillate washes.

**Calcium and hydrogen-ion concentration and the interfacial tension of [insecticidal] pyrethrum extracts.** R. P. HOBSON (J. Agric. Sci., 1931, 21, 101—114).—The addition of pyrethrum extract to "white spirit" considerably lowered its interfacial tension against water. The tension decreased as the alkalinity of the aqueous phase increased. Addition of an emulsifying agent (agral W.B.) further lowered the interfacial tension, particularly against acid solutions, thereby decreasing the sensitiveness of the tension

values to the  $p_H$  of the aqueous phase. Calcium salts in the aqueous phase increase the interfacial tension, whilst alkali salts have the reverse effect. The tension values against solutions containing calcium and sodium salts can be correlated with the ionic ratio  $Ca^{++} : OH^-$ .

A. G. POLLARD.

**Evaluation of pyrethrum flowers (*Chrysanthemum cinerariaefolium*).** J. T. MARTIN and F. TATTERSFIELD (J. Agric. Sci., 1931, 21, 115—135).—Existing methods for determining the toxic constituents of pyrethrum are examined and modifications suggested. New methods for evaluating unadulterated samples and for the examination of individual flowers are described. (Cf. Tattersfield and Hobson, B., 1929, 732.)

A. G. POLLARD.

**Volumetric determination of arsenic in insecticides etc.** J. PÁSKUJ (Magyar Chem. Fol., 1930, 36, 76—83, 95—101; Chem. Zentr., 1930, ii, 1905).—The organic material is destroyed with sulphuric acid and either hydrogen peroxide or nitric acid; after addition of water, hydrochloric acid, potassium bromide, and ferrous sulphate, the arsenic trichloride is distilled off and titrated with potassium bromate by Györy's method (A., 1893, ii, 554). When hydrazine sulphate is substituted for ferrous sulphate, micro-procedure may be employed.

A. A. ELDRIDGE.

**Long-time mineral feeding experiment with dairy cattle.** C. F. HUFFMAN and O. E. REED (Mich. Agric. Exp. Sta. Circ. Bull., 1930, No. 129, 11 pp.).—Mineral supplement of calcium to an average ration of timothy hay, silage, and grain for cows was unnecessary. Supplements of bone meal improved the general health and milk production of the animals, though there was no effect on the duration of the lactation period or on susceptibility to infection (notably by *B. abortus*). Prolonged feeding of rock phosphate injured the health and caused the formation of abnormal and badly worn teeth and enlargement and roughening of the jaw and leg bones. Supplements of limestone produced no adverse effects.

A. G. POLLARD.

**Feeding *Hevea* rubber-seed meal for milk production.** W. B. ELLETT, C. W. HOLDAWAY, J. F. EHEART, and L. D. LASTING (Virginia Agric. Exp. Sta. Tech. Bull., 1930, No. 41, 12 pp.).—Rubber-seed meal is an efficient medium-protein concentrate for milch cows, and is closely comparable with linseed meal for milk production. Results of digestibility trials are recorded.

A. G. POLLARD.

#### PATENTS.

**Instrument for testing the acid or alkaline condition of soil.** A. G. GIBB (B.P. 341,266, 16.1.30).—A strip of metal or other rigid material is provided near one end with two semi-circular piercings into and through which testing papers are passed prior to insertion into the soil.

A. G. POLLARD.

**Preservation and transport of seed in tropical and sub-tropical countries and under similar conditions.** ÉTABL. ET LABORATOIRES G. TRUFFAUT and LA RADIOTECHNIQUE (B.P. 341,484, 16.10.29. Fr., 10.11.28).—Seed dried in air or *in vacuo* at 35—40° and stored, in an inert gas or dry air, in sealed

containers is claimed to retain its vitality unimpaired over long periods. E. HOLMES.

**Production of mixture suitable as fertilisers.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 339,969, 16.9.29. Norw., 24.11.28. Addn. to B.P. 339,340; B., 1931, 201).—The mother-liquor from the crystallisation of the calcium nitrate in the prior process, after, if desired, precipitation and removal of the calcium as its sulphate, is neutralised or treated with ammonia, urea, cyanamide or its salts, phosphates, ammonium or alkali carbonates, etc., or mixtures of these, the substances being supplied in a physical state such that, after removal of water, stable fertilisers are obtained. Other plant foods, e.g., potassium ammonium, or urea salts, may be added, and admixture and drying may be effected *in vacuo*. L. A. COLES.

**Insecticide and fungicide compound.** J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,764,792, 17.6.30. Appl., 1.9.25).—Petroleum products containing liquid aliphatic or naphthenic hydrocarbons are subjected to a partial oxidation treatment with air in the presence of vanadium or molybdenum oxide catalysts and the product is fractionally distilled. The portion boiling above 250° is neutralised with a weak base and the purified oil is emulsified with a soap solution made preferably from sulphonated products of the partial oxidation process. A. R. POWELL.

**Sulphur emulsion and insecticide containing the same.** A. HARTZELL and F. H. LATHROP, Assrs. to W. C. O'KANE and P. MOORE (U.S.P. 1,772,511, 12.8.30. Appl., 2.1.24).—The emulsion comprises a solution of sulphur in carbon disulphide, water, an emulsifier (e.g., fish-oil soap), and, if desired, oleic acid and nicotine sulphate. L. A. COLES.

**Manufacture of [oil-in-water] emulsion [for use as insecticide etc.].** H. B. GOODWIN, Assr. to LATIMER-GOODWIN CHEM. CO. (U.S.P. 1,774,092, 26.8.30. Appl., 30.9.26).—The emulsifier comprises a gum resin (gamboge), an alkali (ammonia), and an alcohol. L. A. COLES.

**Acid phosphate** (U.S.P. 1,761,991—2).—See VII. **Cocoa by-products** (B.P. 341,000).—See XIX.

## XVII.—SUGARS; STARCHES; GUMS.

**Cultivation of sugar beet.** DAVIES.—See XVI. **Honey and sucrose in bread manufacture.** GEDDES and WINKLER.—See XIX.

### PATENT.

**Centrifugal separator** (B.P. 341,298).—See I.

## XVIII.—FERMENTATION INDUSTRIES.

**Evaluation of diastatic enzyme preparations; taka-diastase, malt diastase, and pancreatic diastase.** T. HARADA (Ind. Eng. Chem. [Anal.], 1931, 3, 1—3).—The procedure described is a modification of Lintner's method, and is based on the production of a definite quantity of a reducing sugar by the action of a diastase on a known quantity of Lintner starch. To 100 c.c. of Lintner starch solution (2%) are added 10 c.c.

of the enzyme solution, and after 30 min. the action is stopped by addition of 10 c.c. of 0.25*M*-sodium hydroxide solution; the cooled solution is titrated with Fehling's solution. By buffering the starch solution with potassium hydrogen phthalate and sodium hydroxide the results obtained are more accurate than those obtained by other methods. The method of calculating the Lintner values from the results is described, and tables are given of the Lintner values of taka-diastase and of malt and pancreatic diastases, together with the corresponding dextrose content of the solution, for a range of titration volumes.

H. F. GILLBE.

**Improvement of English cider.** H. V. TAYLOR (J. Min. Agric., 1930, 37, 1095—1100).—A review, giving the chemical characteristics of representative ciders, which stresses the question of variety. E. HOLMES.

**Supposed proportionality between the  $p_H$  and the acid taste of aqueous solutions, especially as regards wines.** P. CRISCI (Annali Chim. Appl., 1930, 20, 566—583).—The acid taste of a wine, although modified by such constituents as sugar, alcohol, glycerol, extract, salts, tannin, etc., is due mainly to undissociated acid molecules, and depends more on the  $p_H$  than on the total acidity, but varies widely with comparatively slight changes in  $p_H$ . There exists a certain degree of relationship between  $C_H$  and total acidity of wines, these increasing together. The  $p_H$  alters little on dilution of the wine with water, although the acid taste is markedly diminished. Although it determines only a slight and variable increase in the  $C_H$ , the introduction of organic acids into wines accentuates the acid taste and is to be regarded as rational practice.

T. H. POPE.

**Vitamin content of vinegar.** J. STASTNÝ (Vestn. Českoslov. Akad. Zem., 1930, 6, 611—619; Chem. Zentr., 1930, ii, 1569).—Vinegar prepared by fermentation of alcohol contains a substance (probably vitamin-D) which permits and accelerates alcoholic fermentation in vitamin-free media. The quantity is proportional to the acetic acid content. A. A. ELDRIDGE.

### PATENTS.

**Manufacture of yeast.** L. and E. ELION (B.P. 340,637, 10.10.29. Holl., 17.10.28).—Yeast with an improved fermentative power is obtained by suspending it in a solution of fermentable carbohydrates, nitrogen, and substances containing phosphorus, and by limiting its propagation in known ways other than by regulating the aeration. C. RANKEN.

**Seed production for biochemical processes.** DARCO SALES CORP. (B.P. 340,514, 20.9.29. U.S., 27.7.29. Addn. to B.P. 336,207; B., 1931, 40).—The micro-organisms are produced by culture methods, during the whole or part of which activated decolorising carbon is present in the culture medium. C. RANKEN.

**Aeration of fermenting liquids.** E. STICH (B.P. 340,644, 12.10.29).—The fermenting liquid is aerated by forcing air under pressure through the pores of diaphragm plates which consist of two layers. The pores of the layer immediately in contact with the liquid vary from 0.16—

6  $\mu$  in diam., whilst those through which the air first passes have a larger diameter. C. RANKEN.

**Ozonisation of fermented liquids.** R. COFFRE (B.P. 340,647, 14.10.29. Fr., 15.10.28).—Porous bodies or carriers, such as oak chips, are alternately steeped in the fermented liquid and ozonised after draining for several hours. C. RANKEN.

## XIX.—FOODS.

**Bound water of wheat-flour suspensions.** R. NEWTON and W. H. COOK (Canad. J. Res., 1930, 3, 560—578).—The bound-water method has been modified in order to determine the hydration of flour in suspensions in water and in dilute lactic acid solutions. In 15% aqueous suspensions, from which the natural electrolytes have been eliminated by washing, the bound water varies from 1.8 to 5.2%, according to the  $p_H$ , there being maxima at  $p_H$  3 and 10; rather higher results are obtained if more dilute (0.5*M*) sucrose solution is employed. In 15% suspensions in dilute (about 0.45%) lactic acid the flour solids are hydrated to the extent of about 40–50%, washing raising the values slightly. The hydration of wheat starch averages 30% under all conditions, whilst that of gliadin and glutenin averages 85%; in the flour the proteins are hydrated to the extent of about 100%, which after washing rises to 200%, in accordance with the general theory of emulsoids. The similarity of the results obtained with the two proteins, although the one, gliadin, disperses completely, whereas the other does not form a true colloidal solution, indicates the necessity of taking into account structure as well as hydration when considering the properties of gluten. The differences of hydration of strong and weak flours are regarded as being too small to be of importance as factors in baking quality.

H. F. GILLBE.

**Ash content of cereals and flour.** O. HALTMEIER (Z. ges. Mühlenwesen, 1930, 7, 51—52; Chem. Zentr., 1930, ii, 1790).—The ratio of the ash content of the flour to that of the grain is preferred to the absolute ash content of the flour as a measure of the degree of milling. The readily determined relative conductivity is also serviceable.

A. A. ELDRIDGE.

**Relative value of honey and sucrose in bread manufacture.** W. F. GEDDES and C. A. WINKLER (Canad. J. Res., 1930, 3, 543—559).—Substitution of honey for sucrose in ordinary bread-baking formulæ is without influence on the loaf volume, flavour, and other characteristics, and despite the absence of the hydrolysis stage the reducing sugar content and the rate of gas production are unchanged. Buffered yeast-honey and yeast-sucrose suspensions of equivalent sugar content under conditions resembling those in fermenting bread dough and at various  $p_H$  ranges exhibit the same rates of gas production. Sucrose inversion is not a limiting factor in the rate of gas production in dough, and during the inversion of sucrose by yeast the invertase stage proceeds more rapidly than the zymase action. The use of honey in the baking industry is indicated only where its flavour and greater sweetening power and hygroscopicity are advantageous.

H. F. GILLBE.

**Use of honey in "water-ices" and sherberts.** H. A. SMALLFIELD (Sci. Agric., 1931, 11, 259—264).—Addition of honey or dextrose to "water-ice" mixtures prepared with sucrose lowers their f.p. and largely prevents the surface crystallisation of sucrose.

A. G. POLLARD.

**Diastatic activity of some American honeys.** R. E. LOTHROP and H. S. PAINE (Ind. Eng. Chem., 1931, 23, 71—74).—About 300 genuine American honeys, most of them of known floral type, have been examined by Gothe's method, and the majority have been found to meet the German requirement that 1 g. of honey should hydrolyse 17.9 c.c. of 1% starch solution. Many floral types, particularly the darker coloured ones such as sage, buckwheat, and tulip-poplar, show consistently high activities, whilst the paler and more delicately-flavoured honeys such as orange and lucerne are notably low, and that from clover is intermediate in value. Gothe's method is satisfactory only for honeys of medium diastatic activity. Improved results are obtained when the solutions are buffered to  $p_H$  5.3 with *M*/15-phosphate. Heating honey at a moderate temperature or storage over a period of years causes it to lose diastatic activity and to respond to the test for hydroxymethylfurfuraldehyde.

T. McLACHLAN.

**Variations in the f.p. and conductivity of milk.** W. PLÜCKER and A. STEINRUCK (Z. Unters. Lebensm., 1930, 60, 112—123).—Detection of added water by lowering of f.p. is not dependable since at certain periods during lactation the composition of the milk may undergo considerable change. When cows were in heat some samples had f.p. 5.7 units above the range found by Weinstein, viz., 53.7—56.0 (cf. B., 1928, 910), whilst towards the end of lactation high f.p. is also to be detected, the maximum found being 58.9. The combined effect of these two factors may be equivalent to 7.5% of added water. In cases of souring milk, for reduction to normal acidity the factor 0.0006 gives more nearly accurate results than 0.0008. In cases of intentional adulteration, the percentage of added water (calc. from f.p. or conductivity measurements) was, with one exception, less than that actually present. In similar physiological conditions the range of conductivity was found to be 39.3—56.3 (that given by Mayr and Wurster being 37.7—41.7), so that a high value is alone no indication of sickness or diseased udders.

H. J. DOWDEN.

**Detection of heated milk and a new method of testing for pasteurisation by the "holding" process.** S. ROTHENFUSSER (Z. Unters. Lebensm., 1930, 60, 94—109).—The degree of heating to which milk has been subjected may be followed by the peroxidase or diastase present in the lead acetate serum. The reagent ("p-tetrasulphite") is prepared by dissolving 1 g. of p-phenylenediamine hydrochloride in 12 c.c. of water and adding 4 g. of guaiacol dissolved in 100 c.c. of 96% alcohol, the mixture being then diluted with alcohol to 150 c.c. Freshly-prepared sodium bisulphite solution (20%) is added in the proportion of 20 drops per 100 c.c. of mixture (40 drops = 2.9 c.c.). To 10 c.c. of milk or lead acetate serum are added 3—4 drops of hydrogen peroxide (3%), and, after shaking, 10 drops of the reagent.

Raw milk gives immediately a violet coloration, and highly heated or boiled milk shows no change, whilst with moderately heated milk the colour develops more or less slowly according to the degree of heating which has been applied. With acid (or soured) and alkalis milk slight modifications are necessary. Pasteurisation by the "holding" process ( $\frac{1}{2}$  hr. at 63°) is revealed by the diastase activity. A small volume (1 c.c.) of a specially prepared, stable solution of starch, containing sodium chloride as co-enzyme, is mixed with 10 c.c. of lead acetate serum and kept for 10–16 hrs. at room temperature (or less if warmed). The product is mixed with a small volume of 0.002*N*-iodine solution and the resulting colour indicates the earlier heat treatment. An immediate violet coloration denotes highly heated milk and a pale yellow fresh milk, whilst the intermediate brown-violet or red-violet shades are associated with definite temperatures. Slow development of the violet colour indicates milk pasteurised by the "holding" process, and an immediate blue colour boiled milk.

H. J. DOWDEN.

**Determination of milk-proteins. I. Chemistry of the separation of casein.** G. M. MOIR (Analyst, 1931, 56, 2–9).—Maximum casein values for milk are obtained when the  $p_H$  is adjusted to 4.5–4.7 by the addition, to 10 c.c. of milk diluted to 50 c.c., of 1.5 c.c. of 10% acetic acid followed by 4.5 c.c. of 0.25*N*-sodium acetate. Slight souring of the milk has no effect on the results obtained.

A. R. POWELL.

**Determination of copper in dairy products.** H. T. GEBHARDT and H. H. SOMMER (Ind. Eng. Chem. [Anal.], 1931, 3, 24–26).—The Elvehjem-Lindow method (A., 1929, 614) yields low results when applied to the determination of copper in milk; much of the loss occurs as the result of too long a time and too high a temperature of ignition. The maximum ashing temperature should be 565° and the operation should be completed within 3–4 hrs. After dissolution of the ash in dilute hydrochloric acid and precipitation of the copper as sulphide the solution is cooled to 10° and shaken with about one half its volume of chloroform; on settling, the copper sulphide is retained by the chloroform layer, which after washing once is evaporated to dryness. The residue is dissolved in dilute nitric acid and after neutralisation of the solution the copper is determined colorimetrically in the usual manner, care being taken to remove all nitric acid and to prevent decomposition of the phenolphthalein indicator by either acid or alkali. This modified procedure is more rapid than the original, and the danger of losses is minimised.

H. F. GILLBE.

**Commercially prepared infant foods.** G. J. and A. M. HUCKER (New York Agric. Exp. Sta., Tech. Bull., 1930, No. 584, 13 pp.).—A general description.

**Maltol and its colorimetric determination in malt-coffee.** T. MERL (Z. Unters. Lebensm., 1930, 60, 216–227).—The maltol content has been found to be a convenient means of ascertaining whether the product conforms to the statutory requirements, and a method has been worked out for its determination. Maltol is present as a pyrogenic decomposition product of amylase, and may be detected in the product and in

the roasting-chamber fumes by virtue of its phenolic reactions with uranyl nitrate and phosphomolybdic acid. A determination is possible through its reaction with iodine and caustic soda to form iodoform, which is then converted into silver iodide, but the method is not so convenient as the following colorimetric method. The sample of coffee (50 g.), mixed with sand, a few c.c. of water, and animal charcoal, is extracted for 4–5 hrs. with carbon tetrachloride, the extract being afterwards shaken with 100 c.c. of ferric chloride solution (containing 3 drops of 10% ferric chloride solution and 2 c.c. of *N*-hydrochloric acid). A violet coloration is imparted to the carbon tetrachloride layer, which is separated, filtered, and compared in a colorimeter against the colour of a standard salicylic acid solution containing ferric chloride. A formula for the evaluation is given. The method has been satisfactorily applied to a number of roasted barley products which had undergone various degrees of germination, and from the results it is concluded that normal malt-coffee should contain not less than 0.06% of maltol. By its action as an anti-oxidant, maltol prevents the oils and fats from becoming rancid and thus improves the keeping qualities of the coffee.

H. J. DOWDEN.

**Iodine content of lamb thyroids.** B. W. SIMPSON (New Zealand J. Agric., 1930, 41, 302–306).—A definite parallelism exists between the thyroid-iodine content of lambs born and reared in a particular area and the amount of iodine available in that area. Systemic advisory work on iodine-feeding could be based on such analytical data.

A. G. POLLARD.

**Determination of purine compounds in meat extracts.** A. ESCUDERO and G. WAISMAN (Semana méd., 1930, II, 1436–1438).—The proteins are removed from a 10% solution by Folin and Wu's method, the purine compounds being determined by Krueger and Smid's method by means of copper sulphate and sodium hydrogen sulphite.

CHEMICAL ABSTRACTS.

**Microchemical detection of benzoic and salicylic acids and of *p*-hydroxybenzoic esters in foods and drugs.** R. FISCHER and F. STAUDER (Mikrochem., 1930, 8, 330–336).—The free acid or ester is isolated by extraction of 1–2 g. of the material with ether after acidification with hydrochloric acid and evaporation of the extract at 50°. Fatty substances should first be digested with warm sodium bicarbonate solution. The residue is heated for 15 min. at 55–60°, when benzoic acid, if present, sublimes and may be recognised by the growth of mercuric benzoate crystals when mercuric acetate is added to the aqueous solution. On raising the temperature to 65–70° *p*-hydroxybenzoic esters sublime and may be recognised by the red coloration formed with Millon's reagent and differentiated by the m.p.: methyl (Nipagin) 97°, ethyl 116°, propyl (Nipagin) 109–112° (lit. 126°). Salicylic acid sublimes at 70–85°. The sublimation tube should be 10–11 mm. deep, in order to prevent contamination of the sublimate with ether.

H. F. GILLBE.

**Composition of fruits as used for jam manufacture in Great Britain.** T. MACARA (Analyst, 1931, 56, 35–43).—Tables are given showing the extreme



and average composition of fruits used for making jam and the range of values for the content of insoluble solids, acids, and pectin in various fruits, together with notes on the analysis of fruits and the method of using the data tabulated in interpreting the results of jam analyses. A. R. POWELL.

**Composition of tomatoes and tomato conserves and the determination of water content.** A. LEONHARD (Z. Unters. Lebensm., 1930, 60, 185—195).—The sample (10 g. if thick and 5 g. if thin) is distilled with 150 c.c. of xylene in a specially constructed apparatus, the distillation being arranged to last for 20 min. The formation of emulsions and the adherence of droplets to the receiver are obviated by distilling briskly towards the end of the operation and by shaking the distillate with 1 c.c. of potassium hydroxide solution (50%). The distillate is free from volatile constituents, is almost neutral, and leaves no perceptible residue on evaporation. Repeat experiments were in good agreement, and the results for Italian and German commercial products, tomato preserve of various degrees of thickness (puree, marc, extract), and for fresh tomato marc are recorded. H. J. DOWDEN.

**Storage of potatoes.** N. D. VYAS (Agric. J. India, 1930, 25, 408—416).—The most successful but the costliest method of storing potatoes in India is by cold storage. Storage in coarse dry sand leads to losses between 40 and 90% or more, which can be reduced to 28—31% (by wt.) by the use of cinders or charcoal.

E. HOLMES.

**Effect of pure culture inoculation on the quality and chemical composition of sauerkraut.** C. S. PEDERSON (N.Y. State Agric. Exp. Sta. Tech. Bull., 1930, No. 169, 29 pp.).—Inoculation of kraut with pure cultures of *Leuconostoc mesenteroides*, *Lactobacillus pentoceticus*, and non-gas-forming bacilli results in some changes of flavour and variations in the amount of total, volatile, and non-volatile acids produced. Generally speaking, the product is inferior. A. G. POLLARD.

**Calorific value of silage.** M. Y. AKSENOVA (Trudi Vologod. Molochno-Choz. Inst. Bull., 1929, No. 81, 63—67).—The following calorific values (kg.-cal. per kg. of dry substance) are recorded: clover hay 4610.2, clover silage 4678, oat straw 4451.3, beet pulp 4518.8, wheat middlings 4770, linseed meal 5242.9, sunflower-seed meal 5312.5. During a feeding period without silage two cows voided, respectively, 19,393.3 and 20,757.9 kg.-cal. as manure in 24 hrs.; corresponding figures with silage were 22,009.1 and 22,110.3, respectively. Coefficients of digestibility and starch equivalent values have been calculated. The physiologically useful energy of clover silage is 2426.6 kg.-cal. per kg.

CHEMICAL ABSTRACTS.

**Nutritive value and digestibility of silage from clover.** V. A. SKVORTZOV (Trudi Vologod. Molochno-Choz. Inst. Bull., 1929, No. 81, 29—51).—Addition of clover silage to the fodder of cows increased the digestibility of cellulose and nitrogen-free extractive substances, but decreased that of proteins and fat.

CHEMICAL ABSTRACTS.

**Exchange products in feeding clover silage.** M. Y. AKSENOVA (Trudi Vologod. Molochno-Choz. Inst.

Bull., 1929, No. 81, 55—60).—Addition of silage to a dairy ration decreased the coefficient of digestibility of nitrogenous substances, apparently owing to stimulation of the splitting off of metabolic exchange products since the digestibility of the crude protein is unaltered by addition of silage. CHEMICAL ABSTRACTS.

**The antiscorbutic vitamin.** J. TILLMANS (Z. Unters. Lebensm., 1930, 60, 34—44).—For the differentiation of natural and artificial foodstuffs by measurement of their reduction potentials, 2:6-dichlorophenol-indophenol has been found extremely sensitive (cf. B., 1929, 301), and the presence of these reducing substances has been correlated with that of vitamin-C. By titration with a solution of the dye it was found that the reducing material in lemon juice possesses a sensitivity towards heat, hydrogen, and alkalis which is exactly parallel to that of the vitamin, whilst the titration values of a large number of other foodstuffs agree closely with the recorded content of vitamin-C. An attempt has been made to isolate the reducing substance from fresh lemon juice in an atmosphere of nitrogen, but, after removal of 97% of the solids, only an aqueous solution, characterised by strong reducing properties, could be obtained. No crystalline substance could be isolated. When tested on animals the aqueous solution exhibited high antiscorbutic activity. The inference that the reducing substance and the vitamin are identical has not been confirmed (cf. Zilva, A., 1928, 801). H. J. DOWDEN.

**Silver for chemical plant.** McDONALD.—See X. **Saponification values of edible fats.** GROSSFELD.—See XII. **Vitamin-D in vinegar.** STASNY.—See XVIII. **Determination of water in spices etc.** PRITZKER and JUNGKUNZ.—See XX.

PATENTS.

**Treatment of grain.** W. VON GELINCK and W. FAHRENHORST (B.P. 341,616, 14.11.29. Ger., 23.11.28).—An apparatus for producing dough from an unmilled, softened grain, which is fed by means of two worms to cutting discs under a pressure insufficient to crush the grain. E. B. HUGHES.

**Treatment of edible milling products.** H. C. J. H. GELISSEN and F. V. HOOFT, ASSRS. to NOVADEL-AGENE CORP. (U.S.P. 1,773,989, 26.8.30. Appl., 5.4.28).—When bleaching flour with organic peroxides, a volatile substance is mixed with the bleaching agent to hasten the reaction and permit the use of a less finely-ground powder. In an example, the use of a 1:1 mixture of benzoic acid and benzoyl peroxide is claimed.

E. B. HUGHES.

**Manufacture of milk products.** R. HELLERUD (B.P. 341,414, 5.10.29 and 8.3.30).—A stable, artificial cream is prepared by mixing milk containing a small proportion of egg yolk with a suitable vegetable oil and homogenising in two stages under specified conditions. It is claimed that the product may be pasteurised without breaking the emulsion, and that the artificial cream has satisfactory whipping properties. E. M. HUGHES.

**Recovering products from cocoa by-products.** H. E. POTTS. FROM MONSANTO CHEM. WORKS (B.P. 341,000, 7.10.29).—Theobromine, caffeine, and cacao



butter are extracted from cacao cake or shell by ethylene dichloride, with or without pretreatment either with hot water or steam to liberate alkaloids or with an alkali or alkaline-earth oxide to separate free fatty acid and theobromine. The products are separated and purified and the alkaloid-free residue is used as fertiliser or feeding-stuff. E. B. HUGHES.

**Treatment of [withered] green tea leaf.** COLOMBO COMMERCIAL Co., LTD., and F. A. KUP (B.P. 342,009, 5.11.29).

**Filter for milk testing** (B.P. 340,879).—See I. **Bleaching agents** (B.P. 339,336).—See VI.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Effect of sunlight on ephedrine solutions.** E. E. MOORE and M. B. MOORE (Ind. Eng. Chem., 1931, 23, 21—23).—Aqueous solutions containing ephedrine oxidise readily in the presence of strong sunlight with the formation of benzylidenephedrine. A theory of the mechanism of the change is given. Amber and black are the best protectives, flint and blue glass provide slight protective power, whilst commercial antioxidants have no effect on decomposition. Ephedrine carbonate, m.p. 80—85° (decomp.), is formed when solutions of the alkaloid in mineral oil are exposed to air or treated with carbon dioxide. T. McLACHLAN.

**Characterisation of neodorm.** G. KUHLMANN (Pharm. Ztg., 1931, 76, 113).—Neodorm ( $\alpha$ -bromo- $\alpha$ -isopropylbutyramide) sublimes in characteristic crystals. It is quantitatively decomposed by boiling water and yields with boiling aqueous sodium hydroxide mainly ethyl isopropyl ketone, hydrogen bromide, and hydrogen cyanide, with some ammonia. It gives a positive iodoform reaction and with Nessler's or Millon's reagent a white precipitate, soluble in excess. Its white mercury derivative is converted by potassium iodide into a yellow substance, which becomes red when kept. After administration of neodorm, the bromine is excreted entirely in inorganic combination. H. E. F. NOTTON.

**Biological evaluation of *Filix mas* preparations.** Z. CSIPKE (Magyar Gyó. Társ. Ert., 1930, 6, 85—101; Chem. Zentr., 1930, ii, 1898).—Chemical methods of pharmacological evaluation are insufficient; a biological method employing leeches is described.

A. A. ELDRIDGE.

**Determination of the nicotine content of tobaccos.** W. PETRI (Z. Unters. Lebensm., 1930, 60, 123—136).—The results of a large number of determinations (by Pfyl and Schmitt's method) of the nicotine content of tobacco products of all classes are summarised as follows: tobacco 0.36—2.1, cigars 0.64—1.8, small cigars ("zigarrillos") 0.64—1.7, cigarettes 0.70—1.7%. Natural tobacco contains 0.3—8.0% of nicotine and, with one exception, none of the "treated" tobacco products had a value less than 0.34%, and therefore none could rightly be described as "free from nicotine" etc. It is recommended that the designations "free from nicotine" (nicotinfrei) and "low in nicotine" (nicotinarm) should be applied to tobaccos having nicotine contents of 0.08% and 0.2%, respectively.

H. J. DOWDEN.

**Determination of *p*-chlorophenol in pharmaceutical preparations.** K. FEIST and F. KLATT (Pharm. Ztg., 1931, 76, 112—113).—The methods of the D.A.B. VI for determining phenol by bromination are applicable to the determination of *p*-chlorophenol by conversion into chlorodibromophenol. In mixtures with menthol or camphor and alcohol the phenol may be determined after extraction with alkali.

H. E. F. NOTTON.

**Luminescence analysis of homœopathic triturates.** A. KUHN (Apoth.-Ztg., 1930, 45, 343—344; Chem. Zentr., 1930, ii, 1742).—Determinations were made of the homogeneity and particle size of triturates of uranium nitrate and mercurous chloride by Danckworth's luminescence method. The greater is the degree of comminution, the more marked is the fluorescence colour of the lactose compared with that of the substance under examination. With progressive trituration animal charcoal becomes darker.

A. A. ELDRIDGE.

**Rapid determination of water in drugs, spices, and chemicals.** J. PRITZKER and R. JUNGKUNZ (Pharm. Acta Helv., 1930, 5, 1—9; Chem. Zentr., 1930, ii, 101).—A simple form of distillation apparatus which requires 30 min. at most for a determination of water content and gives results in good agreement with those obtained with a drying oven or air-bath is described. It permits work with light and heavy liquids.

L. S. THEOBALD.

**Mechanical processes for extracting oil of lemon.** F. LA FACE (Boll. Uff. R. Staz. Sperim. Ind. Ess. Calabria, 1930, 5, 129—137, 149—159, 178—191, 212—218, 234—243).—The various processes are described in detail and working costs and yields are considered. The sponge oil is superior to the mechanical oil, as the latter contains a lower proportion of the odoriferous components and has an odour resembling that of grass, this being caused by the prolonged and intense abrasion to which the peel is subjected. The loss in perfume, especially the diminution in the content of citral, which the oils undergo is due principally to fermentations, which occur the more intensely in a strong air current; use of sterile water for the boiling or addition of sodium benzoate to the oil is suggested as a remedy.

T. H. POPE.

**Bergamot oil.** I. HEROLD (Deut. Parfümerieztg., 1930, 16, 392—394; Chem. Zentr., 1930, ii, 2585).—Bergamot oil contains less than 1% of terpineol. The complete fission of linalyl acetate and the production of a large fraction of alcohols by rearrangement is attributed to fermentation or distillation.

A. A. ELDRIDGE.

***Anthemis nobilis*.** DAFERT and BRANDL.—See XVI. **Detection of benzoic and salicylic acids and of *p*-hydroxybenzoic acid in drugs.** FISCHER and STAUDER. **Antiscorbutic vitamin.** TILLMANS.—See XIX.

## PATENTS.

**Manufacture of aqueous solutions of soporific drugs.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 338,985, 29.8.29. Addn. to B.P. 325,847; B., 1930, 532).—The solubilisation of barbituric acids by

means of alkylamides of lower fatty acids is extended to other hypnotics, *e.g.*, trichloroethylurethane, tribromoethyl alcohol, bromodiethylacetamide. C. HOLLINS.

**Manufacture of pyridine compounds [halogenated pyridones].** A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 339,436, 31.10.29).—3- and/or 5-Halogenated 2-hydroxypyridines are treated with alkylating agents, especially chloroacetic acid, preferably in presence of alkali (potassium hydroxide). 5-Iodo-1-methyl-2-pyridone, m.p. 73–74°, b.p. 182–185°/12 mm., 5-bromo-1-carboxymethyl-2-pyridone, m.p. 237–238°, and the 3:5-dibromo- (m.p. 240–241°), 5-iodo- (decomp. 243°), 3:5-di-iodo- (decomp. 244°), and 3-bromo-5-iodo- (m.p. 244–245°) compounds are described. C. HOLLINS.

**Manufacture of a class of compounds [gold derivatives of succinimide] for therapeutic use.** (SIR) W. J. POPE (B.P. 338,506, 19.7.29).—New compounds containing non-ionised gold are obtained by treating succinimide in presence of a base (ammonia or an amine) with a chloroaurate or chloroauric acid, gold hydroxide, or fulminating gold, preferably in a solvent (alcohol). Chloroauric acid and succinimide are warmed with aqueous ammonia and to the neutral solution ammonia is added drop by drop: the compound,  $[(\text{NH}_3)_2\text{AuSu}_2]\text{Cl}$ , 1–2% soluble in cold water, crystallises out, and may be converted by sodium chloroaurate into the insoluble chloroaurate,  $[(\text{NH}_3)_2\text{AuSu}_2]\text{AuCl}_4$ . The compounds  $[(\text{NH}_2\text{Me})_2\text{AuSu}_2]\text{Cl}$ ,  $[(\text{NH}_2\text{Et})_2\text{AuSu}_2]\text{Cl}$ ,  $[(\text{NH}_2\text{Et})_2\text{AuSu}_2]\text{AuSu}_2$ ,  $[\text{NH}_3\text{AuSu}_3]$  (from the first-mentioned chloride and succinimide),  $[(\text{NH}_2\text{Me})\text{AuSu}_3]$ , alkali and amine salts of  $\text{H}[\text{AuSu}_2]$ , and alkali salts of  $\text{H}[\text{Cl}_2\text{AuSu}_2]$ , are described ( $\text{Su} = \text{C}_4\text{H}_4\text{O}_2\text{N} \cdot$ ). C. HOLLINS.

**Treatment of smoking tobaccos.** R. E. ELLIS. From GENERAL-DIREKTION DER OESTERR. TABAK RÉGIE (B.P. 315,715, 16.7.29).—Tobacco or tobacco mixtures are denicotined by the process described in B.P. 290,204 (B., 1929, 623), and are then treated with a solution of an essential oil. E. H. SHARPLES.

**Liquid preparations etc.** (B.P. 316,132).—See III. **Cocoa by-products** (B.P. 341,000).—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Determination of the degree of exhaustion of developers.** M. L. DUNDON, G. H. BROWN, and J. G. CAPSTAFF (J. Soc. Motion Picture Eng., 1930, 14, 389).—An exposed standard strip of film is immersed in the developer for a definite short time, and then in a solution which stops development and makes the undeveloped emulsion stable when exposed to light. An exhausted borax developer requires a longer time for development and causes apparent loss of exposure; fresh developer containing bromide behaves similarly. It is suggested that the loss of exposure is caused by the solvent action of the sulphite while the development is being delayed. CHEMICAL ABSTRACTS.

**Measurement of flow of gas.** KING and WILLIAMS.—See II.

PATENT.

**Washing of photographic negatives and prints.** P. W. YUILLE, R. HARVEY, and R. BALLANTYNE (B.P. 342,387, 8.11.29).

## XXII.—EXPLOSIVES; MATCHES.

**Reclamation of solvent in [gun]powder works.** O. KREBS (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 215–218).—Ether and alcohol used as solvents in the manufacture of gunpowder may be reclaimed by drying by means of an air current, washing, condensing the vapours, and finally rectifying these by distillation. E. S. HEDGES.

**Coefficients of thermal expansion of explosives.** M. I. MANTROV (J. Chem. Ind., Moscow, 1929, 6, 1686–1688).—Data for trinitrotoluene, dinitronaphthalene, picric acid, and mixtures of picric acid with naphthalene or dinitronaphthalene are recorded.

CHEMICAL ABSTRACTS.

PATENTS.

**Manufacture of organic nitrates [ethylene glycol dinitrate] applicable in the explosives industry.** A. HOUGH (B.P. 341,456, 11.9.29).—Ethylene glycol is nitrated with an acid mixture in which the ratio of nitric to sulphuric acid is not lower than 9:10 nor higher than 5:4, and in which the water content is 4–8.5%, the mixed acid being cooled to 10°, and the temperature of nitration maintained at 5–15°.

W. J. WRIGHT.

**Purification of trinitrotoluene.** A. G. ALLEN (B.P. 341,718, 21.1.30).—Partly purified trinitrotoluene (setting point 78–79°) is mixed at a suitably high temperature with 5–20% of toluene, the mixture being allowed to cool slowly without stirring. A solid mass with a liquid core is thus obtained, and the latter is separated by draining. W. J. WRIGHT.

**Production of an organic [explosive] compound.** J. PEASE and F. NEWELL (B.P. 339,024, 20.9.29).—Hexamethylenetetramine is oxidised with hydrogen peroxide of at least 30 vols. concentration, treated with nitric acid below 16°, and finally oxidised again with hydrogen peroxide, the mixture being covered with benzene or other light liquid and kept for 1–2 hrs. C. HOLLINS.

## XXIII.—SANITATION; WATER PURIFICATION.

**Nature of dust in the air of cotton card rooms.** BRIT. COTTON INDUSTRY RESEARCH ASSOC. (J. Text. Inst., 1930, 21, T 595–604).—An investigation is described of the dust produced during carding in 17 card rooms, covering as wide a range of practical conditions as possible. Methods of sampling the air and counting the particles of dust have been critically examined, and the sampling by the Owens jet extraction apparatus (cf. B., 1923, 579 A), the computation of the

particles, and the expression of the results are described. The dust consists mainly of minute, almost invisible particles and, in addition, a few large ones, chiefly fragments of fibre. About 90% consists of organic matter, and of the remaining 10% of inorganic matter about 60% is silica, whilst iron compounds form the chief part of the remainder. As much as 1% of the dust may consist of fungus spores, especially those belonging to the *Aspergillus niger* group. When the Shirley high-speed dust cage was used in the opening machinery, the very fine dust removed contained up to 80% of mineral matter and a very small amount of nitrogenous material.

B. P. RIDGE.

**Copper and health.** W. G. SCHNEIDER (J. New Eng. Water Works' Assoc., 1930, 44, 485—494).—It is considered safe to use copper or brass pipes for a domestic water supply or to use copper sulphate for the control of algae in reservoirs, as it is shown that small amounts of copper are not harmful to health and may even be beneficial. The permissible copper content of domestic water, based on a daily consumption of 1 gal., should not exceed 20 p.p.m., but water containing such an amount is most unlikely to be used, as with a content of 5 p.p.m. its taste is so disagreeable that it is almost unpotable.

C. JEPSON.

**Proposed modification of the oxygen-consumed method for determination of sea-water pollution.** H. K. BENSON and J. F. G. HICKS, JUN. (Ind. Eng. Chem. [Anal.], 1931, 3, 30—31).—The standard permanganate method when applied to saline water frequently yields results which bear no direct relation to the biochemical oxygen demand. By adaptation of the Zimmermann-Reinhardt method, reliable determinations of pollution may be made provided that a blank is carried out on the purest water obtainable in the neighbourhood of the pollution. To the sample, after digestion, is added a solution containing (per litre) manganese sulphate (67 g.), 85% phosphoric acid (138 c.c.), and sulphuric acid (130 c.c.), and immediately thereafter 10 c.c. of acidified 0.1N-ferrous sulphate; the mixture is then titrated with 0.1N-permanganate.

H. F. GILLBE.

**Determination of phosphates in presence of silica in boiler water.** E. W. SCARRITT (Ind. Eng. Chem. [Anal.], 1931, 3, 23).—The interference caused by silica in the colorimetric determination of phosphate with molybdate and quinol may be eliminated by so adjusting the  $p_H$  of the solution that the ionisation of the silicic acid is repressed. To 40 c.c. of the sample are added 5 c.c. of sulphuric acid (about 30%), 1 c.c. of ammonium molybdate solution (92.3 g. per litre with 38 c.c. of sulphuric acid), and 1 c.c. of quinol solution (23 g. per litre with 5 c.c. of sulphuric acid). After 5 min., 15 c.c. of a solution containing 183 g. of sodium hydroxide and 16 g. of sodium sulphite per litre are added, and the blue coloration due to phosphate is compared with a standard. The coloration begins to fade after 10 min., and silica coloration is liable to appear 5—10 min. after neutralisation with the sulphite solution.

H. F. GILLBE.

**Determination of iron in water.** O. MAYER (Z. Unters. Lebensm., 1930, 60, 195—210).—For the determination of total iron, where only small amounts are present, the residue after evaporation and ignition is taken up with dilute (1:5) hydrochloric acid and a 10% thiocyanate solution, and diluted to suitable volume. A similar volume of thiocyanate-acid mixture is titrated with an iron solution of known concentration to colour equality with the sample. For iron contents above 10 mg./litre, the residue after evaporation and ignition is dissolved in sulphuric acid, and, after reduction by zinc, the solution is titrated with permanganate. For determining inorganically combined iron, 100 c.c. of the water are mixed with 1 c.c. of concentrated hydrochloric acid containing a trace of bromine, and, after 1—2 min., 2 c.c. of thiocyanate solution and then 10 c.c. of ether-amyl alcohol are added. On shaking and keeping, the alcohol layer contains about 60% of the iron. A similar volume of distilled water mixed with identical volumes of reagents is titrated with standard iron solution until the amyl alcohol layers are of equal colour. Ferric ions may be determined directly by siphoning 100 c.c. of the water into a vessel filled with carbon dioxide, and adding 1 c.c. of hydrochloric acid ( $d$  1.19) and 2 c.c. of 50% thiocyanate solution. The colour (concentrated, if necessary, by shaking with 5—10 c.c. of amyl alcohol) is compared against that of a similarly treated iron solution of known concentration. Ferrous ions cannot be determined directly, but are given by the difference between inorganic iron and ferric ions. Organically combined iron is given by the difference between total iron and the inorganic iron.

H. J. DOWDEN.

**Corrosion-resistant protective films of calcium carbonate in water pipes.** G. SCHIKORR (Z. angew. Chem., 1931, 44, 40—41).—The formation of a protective film of calcium carbonate in iron water pipes occurs only when the water contains in solution sufficient calcium carbonate and oxygen and not too great an excess of carbon dioxide. The deposition of calcium carbonate from the water is an electrochemical reaction, ferrous hydrogen carbonate being produced at anodic areas by the action of  $HCO_3^-$  ions and the calcium liberated at cathodic areas forming calcium carbonate with the calcium hydrogen carbonate in solution. This behaviour can be seen by connecting an iron plate to a platinum foil through an ammeter and hanging both metals in a solution of calcium hydrogen carbonate, when a current of about 1 milliamp. is produced and the platinum becomes coated with a film of calcium carbonate.

A. R. POWELL.

## PATENTS.

**Insecticide.** C. B. GNADINGER (U.S.P. 1,773,102; 19.8.30. Appl., 6.7.26).—Powdered flowers of *Pyrethrum* are extracted with acetone (preferably 3 gals. per 5 lb. of flowers), and the filtered solution is diluted with water.

L. A. COLES.

**Drying apparatus** (U.S.P. 1,770,727).—See I. Liquid preparations etc. (B.P. 316,132).—See III. Turpentine substitute (U.S.P. 1,772,895).—See XIII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

APRIL 10 and 17, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

#### Portable pyrometric cone-equivalent furnace.

V. CARTWRIGHT and S. M. PHELPS (J. Amer. Ceram. Soc., 1931, 14, 1—7).—The development of a portable furnace which weighs about 110 lb., uses compressed gas (natural gas or propane) as fuel, and will at the proper rate of heating attain a temperature of cone 34, is fully described and illustrated. R. J. CARTLIDGE.

#### [Tunnel] dryer problems with calculations.

J. L. CARRUTHERS (J. Amer. Ceram. Soc., 1931, 14, 8—20).—Drying operations are discussed and a method of using humidity charts to enable such problems as the prevention of condensation and the proper control of temperature, volume, and humidity of air in waste-heat and radiated-heat dryers to be solved is described. R. J. CARTLIDGE.

**Temperature distribution in internally heated cylinders.** A. B. NEWMAN (Ind. Eng. Chem., 1931, 23, 29—32).—Theoretical. The theory is developed of the flow of heat through the thick walls of a hollow cylinder heated internally. Reference is made to the practical uses to which the curves and formulæ deduced may be put. H. INGLESON.

**Modern grinding.** T. C. FARRANT (Soc. Chem. Ind., Chem. Eng. Group, 1931. Advance proof. 32 pp.).—Grinding mills may be divided into three groups: (i) slow-speed mills in which attrition and impact are the chief factors, *e.g.*, pan mills, burrstone, ball mills suitable for silica; (ii) medium-speed mills depending chiefly on compression, *e.g.*, roller mills, suitable for phosphates; and (iii) high-speed mills depending largely on impact and shearing, suitable for precipitated and light materials. Power consumption depends largely on toughness. The closed circuit with air separation has many advantages, and its scope has been extended to abrasive materials by placing the fan on the remote side of the cyclone and sealing the bottom of the latter. For wet grinding, pan mills, edge-runners, burrstones, ball mills, and to a less extent stamp mills are chiefly used. Recent developments include the use of the close circuit with classifiers, notably the Andrews classifier. In the closed circuit the fineness of the product is controlled by the circulating load, quantity of water used, speed of mill, and fineness of grinding media. For pulverised fuel groups (i) and (ii) are indicated for the central system; for the unit system all groups are used, but preferably mills in group (iii), because of the small floor space required and simplicity. Operating data are given, and various types of mills are illustrated. D. K. MOORE.

**Conception and measurement of plasticity. C.g.s. unit, and a new plastometer.** E. KARRER

(Z. tech. Physik, 1930, 11, 326—337; Chem. Zentr., 1930, ii, 3175).—Plasticity is defined as the ability to suffer and to retain changes of shape. A unit is defined and an apparatus described. A. A. ELDRIDGE.

**Recording dust-concentration meter and its application to the blast furnace.** A. W. SIMON, L. C. KRON, C. H. WATSON, and H. RAYMOND (Rev. Sci. Instr., 1931, 2, 67—83).—An exponential expression connecting the light transmitted by a gas containing suspended matter and the concentration of the latter is deduced, and the construction of a meter, involving the use of a thermopile as the light-sensitive element, is described. C. W. GIBBY.

**Sulphuric acid.**—See VII.

See also A., Mar., 318, Catalytic gas reactions. 330, Vacuum evaporation etc. Gas analysis.

### PATENTS.

**Rotary furnaces for soda-recovery plants.** J. HOLMES and H. A. KINGCOME (B.P. 342,545, 12.2.30).—Spent liquor from paper works is fed to the burner end of a rotary furnace and from the other end drops into a secondary furnace, where combustion takes place between the carbon and additional air; finally the soda drops in a molten condition into water. The rotary furnace is provided with internal scrapers at the cooler end and with air seals between the moving and fixed parts, the waste gases passing to a waste-heat boiler. B. M. VENABLES.

**Steam-heated drying cylinders.** S. F. BARCLAY, H. HOYLE, and MATHER & PLATT, LTD. (B.P. 342,513, 28.1.30).—Water of condensation is lifted out of a drying cylinder to the discharge trunnion by a device comprising alternate spirals and cylinders of decreasing diameter. Each spiral is duplicated by one of opposite hand so that the drum may be rotated in either direction. B. M. VENABLES.

**Drying machine [for textile materials etc.].** B. A. PARKES (U.S.P. 1,773,852, 26.8.30. Appl., 18.5.29).—The materials are dried by air blown through them while they are sandwiched between two perforated belt or apron conveyors. The conveyors form a circular loop within which the circulating fan runs at a higher speed. B. M. VENABLES.

**Transmitting heat to and regulating the temperature of dryers, heaters, and the like.** COKE & GAS OVENS, LTD., and T. B. SMITH (B.P. 342,291, 21.10.29).—A vessel is heated by hot fluid, *e.g.*, oil, circulating through a jacket. The separate heater for the oil has an open or vented top permitting expansion of the oil, the rise of level operating a float which controls the supply of heating fuel; thus the actual temperature

\* The remainder of this set of Abstracts will appear in next week's issue.

of the oil may be regulated by adjusting the total amount in the system (cf. B.P. 256,385; B., 1926, 915). Application to the drying of ammonium sulphate is indicated.

B. M. VENABLES.

**Drying or roasting clay, carbon, etc.** F. M. HARTFORD, Assr. to HARROP CERAMIC SERVICE CO. (U.S.P. 1,773,675, 19.8.30. Appl., 1.3.28).—A tunnel kiln is operated with transverse circulation of the drying or roasting gases and a general slower movement either con- or counter-current to the goods. The circulation is from and to a pair of longitudinal flues in the roof, which are subdivided to suit the number of circulating fans provided. The circulating currents are directed through the goods at different levels by curved recesses in the walls, which terminate at different heights.

B. M. VENABLES.

**Treating [drying of moist] material [lactose etc.].** E. T. MEAKIN (U.S.P. 1,766,030, 24.6.30. Appl., 2.8.26).—Material such as casein or lactose is dried by dropping it into a rising current of air in which it remains dancing until sufficiently dried. A number of vertical drums are arranged side by side, and each is provided with an upward current of air, the outlet for which is at the end of the series remote from the feed; the air from any earlier drum passes through controllable shutters in the upper wall (above the level of the original feed), across all the later drums, to a collecting hopper and cyclone where the material is collected.

B. M. VENABLES.

**Grinding mill.** J. KLAGSBRUNN, Assr. to P. LEGRAND (U.S.P. 1,773,906, 26.8.30. Appl., 21.11.27. Austr., 26.11.26).—A mill having a grinding disc with teeth outstanding from its surface and fixed rings with a wavy surface is described.

B. M. VENABLES.

**Centrifugal [grinding] mills.** E. RAPP, and SCHNELL-PRESSENFABR. A.-G. HEIDELBERG (B.P. 342,507, 22.1.30).—The apparatus is provided with a rotating disc and central inlet. The disc has upstanding beaters in the form of vanes which are interrupted not far from the circumference, and stationary grinding bars project into the spaces.

B. M. VENABLES.

**Revolving drum for mixing, shaking, and cracking.** J. E. POLLAK. From MACHINEFABRIEK I.H.O.R. (B.P. 342,541, 10.2.30).—The lower part (when stationary) of a revolving drum is formed as a vehicle capable of being run away on rails and duplicated to keep the drum in almost continuous operation.

B. M. VENABLES.

**Apparatus for cooling liquids [e.g., milk].** F. W. CAVE, and LAWRENCE & Co., LTD. (B.P. 342,265, 21.10.29).—A tubular cooler which may have to be made of steel or other corrodible material in order to resist pressure is covered closely with corrugated sheets of a corrosion-resisting metal over which the streams of liquid to be cooled flow. To obtain good heat-transference the casing is joined to headers at the ends surrounding the return bends of the inner tubes, and the space is either filled with brine or some good conducting liquid, or is subjected to a vacuum which draws the outer cover closely in contact with the tubes.

B. M. VENABLES.

**Measuring the concentration of solids in liquids.** H. J. KERR, Assr. to BABCOCK & WILCOX Co. (U.S.P.

1,773,735, 26.8.30. Appl., 18.2.21. Renewed 5.10.28).—A vessel is described in which the electrical conductivity of, e.g., boiler water can be measured at a reasonably constant temperature. A portion of the liquid under pressure is admitted to the top of the vessel through a small orifice, the vessel being covered but vented to atmosphere so that part of the liquid flashes into vapour and the remainder passes downward through the vessel to a siphon outlet at the bottom. In this way the bulk of the liquid is held at the atmospheric b.p., and is also constantly renewed to give a continuous sample of the contents of the boiler.

B. M. VENABLES.

**Centrifugal separators for liquids containing solids.** G. TER MEER (B.P. 342,589, 24.3.30).—A centrifuge of the strainer type is provided with permeable zones through the other walls of the basket, in addition to the usual ones through the circumferential wall. The auxiliary apertures are less likely to choke, because they are not subjected to the full centrifugal force, and are useful for discharging liquor from the later instalments of feed.

B. M. VENABLES.

**Apparatus for separating pulverised solids from a gaseous current.** FOURS ET APPAREILS STEIN (B.P. 342,594, 26.3.30. Fr., 30.4.29).—An apparatus of the change-of-direction type is described, the finer particles being allowed to pass on into the fan.

B. M. VENABLES.

**Filter casing and its operation.** E. J. SWEETLAND (U.S.P. 1,774,044, 26.8.30. Appl., 28.10.25).—A vacuum filter of any type is placed, as a whole, inside a casing and operated under compressed air.

B. M. VENABLES.

**Apparatus for crystallisation from solutions by evaporation under reduced pressure.** E. L. R. A. SCHELE and F. H. WITTENBURG (R. O. MEYER) and KUPFERHÜTTE ERTEL, BIEBER & Co. (B.P. 342,449, 4.12.29. Ger., 4.12.28).—The apparatus comprises a single shell divided into a number of compartments each having its own evacuating means and stirrer; only the last of the series has an overflow for mother-liquor with the crystals in suspension.

B. M. VENABLES.

**Desiccating apparatus [for liquids, emulsions, juices, etc.].** W. S. BOWEN (U.S.P. 1,774,350, 26.8.30. Appl., 8.5.28).—The casing of a spray-drying apparatus is formed of permeable material and is operated under slight vacuum so that cool dry air is drawn in and prevents any contact of liquid material with the walls.

B. M. VENABLES.

**Filters for air or other gases.** A. SCHIRP GES. M.B.H. (B.P. 342,455, 9.12.29. Ger., 26.10.29).—The filter elements are in the form of panels attached at one end to a vertical chain conveyor. Methods of forming seals between the panels and between them and the framework are described.

B. M. VENABLES.

**Apparatus for filtering air or other gases and for cooling or heating air, or other gases, or liquids.** J. OGDEN (B.P. 342,292, 22.10.29).—An apparatus of the type having screens that are kept wet by sprays of water or other liquid is provided with a filter submerged in the sump through which the used liquid is drawn by the circulating pump. The screens (for gas) are free to swing or are arranged in the form of a perforated continuous band.

B. M. VENABLES.

**Apparatus for precipitation of dust [from gases].** A. E. DAVIS (B.P. 342,439, 30.11.29).—A separator of the laminar flow type having parallel plates of  $\Lambda$ -shape is provided with slits at the lower edges of the plates through which the dust may drop out.

B. M. VENABLES.

**Treatment of furnace gases.** J. G. COUTANT, Assr. to W. C. DRAKE, E. B. PRIEBE, and H. BUCHERT (U.S.P. 1,773,954, 26.8.30. Appl., 8.9.28. Fr., 21.9.27).—An apparatus for the simultaneous removal of dust and recovery of waste heat is described.

B. M. VENABLES.

**Preventing condensation of moisture from flue gases.** G. G. SCHMIDT, Assr. to CARRIER ENG. CORP. (U.S.P. 1,773,870, 26.8.30. Appl., 3.4.28).—An air heater is operated efficiently in that the combustion gases are cooled below the dew point, but condensation in the flue is prevented by admixture of some of the heated air.

B. M. VENABLES.

**Manufacture of pressure gases from liquefied gases at the place of consumption. Production and consumption of compressed gases.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 342,423 and 342,527, [A] 21.11.29, [B] 3.2.30. Ger., [A] 5.1.29, [B] 4.2.29).—(A) Liquefied gas is withdrawn from a container at a moderate pressure, its pressure is raised while still liquid by a pump, and it is then vaporised. A piston attached to the ram of the pump may be operated by gas at moderate pressure withdrawn from the upper part of the storage vessel. (B) Liquefied gas (carried on a vehicle) under moderate pressure is delivered as high-pressure gas by the method described in (A), the pump being operated by the engine of the vehicle and the heat provided by the radiator water. If the gas stored is oxygen it may partly be replaced by air which is compressed, and used as the heating medium for the outgoing oxygen, being thereby itself liquefied, and then rectified.

B. M. VENABLES.

**Vaporisation of liquid gases.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 342,597, 31.3.30. Ger., 5.4.29. Addn. to B.P. 287,909; B., 1929, 501).—The process described in the prior patent is modified by having more than one container: when one is emptied as far as possible into consumers' vessels the residual gas in it is admitted to a full container to increase the pressure and number of heat-conveying molecules therein. If the containers are mounted on a motor vehicle the heat of the cooling water may be used to aid vaporisation.

B. M. VENABLES.

**Separating the constituents of gaseous mixtures.** C. C. VAN NUYS and J. L. SCHLITT, Assrs. to AIR REDUCTION Co., Inc. (U.S.P. 1,774,462, 26.8.30. Appl., 23.10.26).—In, e.g., the separation of air, liquefied air is rectified in a primary rectifier so as to produce pure oxygen. The top effluent is mixed with additional air, recompressed, reliquified at a rather lower pressure than before, mixed again with some of the original liquid air, and rectified in an auxiliary tower so as to produce pure nitrogen. The liquid product from the auxiliary is introduced into the primary rectifier.

**Refrigerant.** J. G. DAVIDSON, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,765,211, 17.6.30. Appl., 7.4.27).—A mixture of a substance of the formula

$C_nH_{2n+1}R$ , where R is hydrogen or a halogen, with at least 20% of vinyl chloride is claimed. Methyl chloride is specifically claimed as the second constituent.

A. R. POWELL.

**Refrigerators.** SOC. ANON. POUR LE CONSERVATION INDUSTRIELLE DU POISSON, and J. E. W. REEH (B.P. 342,896, 4.2.30).

**Furnace fronts.** J. HOWDEN & Co., LTD., and W. H. HOWDEN (B.P. 342,571, 8.3.30).

**Lubricating and liquid seal systems for grinding mills.** E. F. STIMSON (B.P. 342,568, 6.3.30).

**Pyrometer tubes.**—See VIII. Heat-conducting bond.—See IX. Lubricants.—See XII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Fusain.** S. W. PARR, II. C. HOPKINS, and D. R. MITCHELL (Ind. Eng. Chem. [Anal.], 1931, 3, 64—65).—Proximate analyses of 11 samples of fusain from different parts of Illinois and of the total coal substance from the same faces are given. The fusains had an average volatile matter of 20.44%, the values being 15—20% lower than those of the total coal. Sulphur values (0.75—8.18%) followed rather closely the ash values (4.45—18.90%) and the unit coal B.Th.U. values (14,635—15,100) were slightly higher than those of the total coal (14,039—14,705).

E. H. SHARPLES.

**Development of Dakota lignite. IV. Critical oxidation temperature of lignite.** W. C. EATON, G. A. BRADY, A. W. GAUGER, I. LAVINE, and C. A. MANN (Ind. Eng. Chem., 1931, 23, 87—93; cf. B., 1931, 97).—The methods of Wheeler and of Parr and Coons (B., 1925, 195) for studying the ignition temperatures of coals have been adapted for a study of North Dakota lignites from different localities. No material difference in the carbon dioxide index and critical oxidation temperature was apparent, but these properties are affected by moisture content of the sample, size of particles, rate of gas supply, and previous history of the lignite. Drying of the lignite by the Fleissner process does not materially affect the carbon dioxide index and critical oxidation temperature beyond the effect due to decreased moisture content.

H. S. GARLICK.

**Macroscopical examination of coal deliveries.** C. C. CARPENTER (Gas World, 1931, 94, 79—83).—The application of macroscopical methods as an aid in the selection of coal for gasworks' purposes is discussed. The distribution of cannel, durain, clarain, vitrain, and fusain, and of the ash-forming impurities such as slate and clay shales, calcareous strata, stony and earthy matters in the coal, and the size, colour, and texture of the resulting coke are noted. The presence of large quantities of durain makes the coke friable and incoherent, whilst clarain and vitrain enhance the coking qualities of the coal and give high gas yields; the presence of fusain, which is non-coking, is undesirable, and cannel, though increasing the gas yield, exerts deleterious influences on the coke. The presence of the various ash-forming impurities will, in general, affect adversely the quality of the coke.

C. B. MARSON.

**Chemical constitution of coal.** W. A. BONE (J. Roy. Soc. Arts, 1930, 79, 77—95).—A lecture.

**Pyritic oxidation in relation to spontaneous combustion of coal.** I. MIYAGAWA (Mem. Coll. Eng., Kyushu, 1930, 5, 295—397).—Experiments showing the importance of pyritic oxidation as a promoter of spontaneous combustion are described in detail. Pyrites and marcasite from different sources differ in their absorptive capacities for oxygen, but, in general, marcasite, which is easily crushed, oxidises the more readily. When freshly powdered pyrites is treated with dry oxygen, at the beginning the reaction is vigorous, but the rate of oxidation decreases with time; oxidation may be increased by introducing water, which acts as a catalyst. Oxidation of the pyrites produces ferrous sulphate and sulphur dioxide, the last-named being adsorbed on to the surfaces of the pyrites, thereby limiting further oxidation; if this adsorbed sulphur dioxide is removed by water, oxidation again occurs. The oxidation of pyrites is dependent on its surface area, and hence on its degree of fineness. An apparatus is described in which the rise of temperature, due to pyritic oxidation, is measured by a thermopile, and its use is recommended to determine the liability of coals to spontaneous combustion. The capacity of a coal for adsorbing sulphur dioxide is of greater importance than its ease of oxidation in determining its liability to spontaneous combustion. It is concluded that (1) pyritic oxidation is not represented by the commonly accepted equation  $2\text{FeS}_2 + 7\text{O}_2 + 2\text{H}_2\text{O} = 2\text{FeSO}_4 + 2\text{H}_2\text{SO}_4$ , but by  $\text{FeS}_2 + 3\text{O}_2 = \text{FeSO}_4 + \text{SO}_2$ ; and (2) coals containing pyrites which are stored in heaps are most liable to spontaneous combustion during wet weather.

C. B. MARSON.

**Determination of the swelling pressure of coals.** H. NEDELMANN (Brennstoff-Chem., 1931, 12, 42—43).—The coal (120 g.) is charged into a cylindrical steel crucible (80 mm. in diam.; 105 mm. high) provided with a close-fitting piston, which is connected by means of a lever arm with a hydraulic recording manometer. Between the coal and the piston is a perforated disc of refractory material. The crucible is heated by means of a Méker burner to 700—900° (900° at the bottom of the crucible). The swelling pressure is recorded, and the amount of the swelling, or subsequent shrinkage, can also be measured. Concordant and reproducible results are obtained.

A. B. MANNING.

**Dry cooling of coke.** J. RUDE (Engineering, 1930, 130, 543—544).—By the dry-cooling process the sensible heat of the coke is recovered; but it is also claimed that the quality of the coke is improved, owing to its freedom from moisture and breeze. The main objection to the general adoption of the dry-cooling principle is the costliness of the plant. Descriptions are given of the Sulzer and Collin methods. It is suggested that the cost of the process might be reduced by sacrificing part of the steam production (by discharging the coke from the cooler at a higher temperature than the usual 250—300°) and by substituting steam for inert gas as the cooling medium.

C. B. MARSON.

**The cracking process in the gas-making industry.** G. EGLOFF and J. C. MORRELL (Ind. Eng. Chem., 1930, 22, 1080—1083).—Gas from the cracking process has a calorific value of approx. 1250 B.Th.U. per cub. ft.

and is relatively rich in olefines. Tables are given showing the analyses of a number of gases from the cracking process. By the residual oil method of operation, high yields of gasoline, a marketable fuel oil residue, and an insignificant yield of coke can be obtained with a relatively small amount of gas. The operation may also be controlled so that only cracked gasoline, gas, and coke are produced. The potential value of gas from the cracking of low-temperature coal tars, tar acids, and gas tars is considered. Coke from the cracking process has an apparent *d* of 0.9—1.1, a cellular structure exposing maximum area to reaction with steam, and contains 5—15% of volatile matter with 80—90% of fixed carbon and 0.1—1.5% of ash, these physical characteristics making it an ideal raw material for the manufacture of blue gas.

H. S. GARLICK.

**Problems in the determination of unsaturated hydrocarbons in gases.** III. Some factors in bromination with potassium bromide-bromate mixture. H. S. DAVIS, G. S. CRANDALL, and W. E. HIGBEE, JUN. (Ind. Eng. Chem. [Anal.], 1931, 3, 108—110; cf. Davis and Quiggle, B., 1930, 357).—Oxygen prevents the quantitative titration of acetylenes and probably of some diolefines by the bromide-bromate method. A method, in which oxygen is excluded as far as possible from the reaction flask, is described and satisfactory results have been obtained with synthetic mixtures of gaseous olefines and acetylenes. The preparation of methyl- (b.p. —20° to —18°) and ethyl-acetylene (b.p. 6.8—8.5°), trimethylene (b.p. —28° to —27°), and other hydrocarbons is described. Aluminium, nickel, and mercuric salts aid the quantitative titration of acetylene, but prevent that of ethylene.

E. H. SHARPLES.

**Treatment of water-gas tar.** A. V. SIVOLOBOV and L. L. BOLOTINA (J. Chem. Ind., Moscow, 1930, 7, 1802—1805).—The tar is dehydrated by distilling off about 6% of its volume, when the residue on keeping separates into an aqueous and a tarry layer, the latter containing about 1.5% of water. The tar on fractional distillation yields a series of liquid hydrocarbons, b.p. 70—245° (85%), from which about 20% of naphthalene and anthracene separate on keeping. The residue contains 45% of oils, b.p. 245—350°, leaving a brittle, dark brown tar suitable for the manufacture of lacquer. The dehydrated tar thus yields 61.2% of liquid hydrocarbons (containing 35% of benzene and 8% of toluene), 32.2% of naphthalene and anthracene, and 6.6% of non-volatile residue.

R. TRUSZKOWSKI.

**New process for the preparation of hydrogen and hydrogen-nitrogen mixtures [from coke-oven gas].** W. GLUUD (Ber. Ges. Kohlentechn., 1930, 3, 211—220).—Introductory (cf. following abstracts). A new process has been worked out by the Gesellschaft für Kohlentechnik in which gas from the coking of coal, freed from hydrogen sulphide, is treated with steam at high temperature and the following change occurs:  $\text{C}_n\text{H}_m + n\text{H}_2\text{O} = n\text{CO} + (n + m/2)\text{H}_2$ . This carbon monoxide-hydrogen mixture is then treated with additional steam and the hydrogen content of the gas is still further increased:  $\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{H}_2$ . With dolomite as catalyst for the second stage the tempera-



ture necessary as well as the space velocities were well suited to large-scale operation. A study of the best types of dolomite capable of withstanding the disintegrating influence of repeated regenerations showed that samples in which the calcium and magnesium were present in equimolecular proportions were the most suitable, and that the temperature of 1050° must not be exceeded. The initial stage of the process proceeded satisfactorily in the presence of a nickel catalyst, but the sulphur content of the gas caused a loss of activity due to the production of the sulphide. By suitable choice of working temperature the catalyst was maintained in an active condition by the oxidation of the sulphide by the steam, which re-formed nickel (oxide). A special grog was used as a support for the catalyst. Many materials tested were found to cause the deposition of soot by direct decomposition of the hydrocarbons and soon became powdery and friable. Soot deposition, which is excessive in the temperature interval 500—700°, was avoided by rapid heating of the gases through this range following the addition of small amounts of air or oxygen. In 1927 a plant was erected which was designed to produce 400 cub. m. of hydrogen per hr. So successful was the large-scale operation that the carbon monoxide content of the final gas was only 0.05—0.07%.

H. INGLESON.

#### The methane-steam equilibrium and its control.

W. KLEMP and F. BRODKORB (Ber. Ges. Kohlentechn., 1930, 3, 220—229).—Theoretical. At high temperatures the reaction between methane and steam takes place according to the equation (1)  $\text{CH}_4 + \text{H}_2\text{O} = \text{CO} + 3\text{H}_2$  —48.9 kg.-cal., whilst at lower temperatures it takes the form (2)  $\text{CH}_4 + 2\text{H}_2\text{O} = \text{CO}_2 + 4\text{H}_2$  —38.5 kg.-cal. The equilibrium constants for these two reactions have been determined only for a few temperatures, but it is possible to calculate their values from those obtained in the reactions  $\text{CH}_4 = \text{C} + 2\text{H}_2$ ;  $\text{C} + \text{CO}_2 = 2\text{CO}$ ; and  $\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{H}_2$ , which are known over the range 450—1200°. The constants  $K'_p = p_{\text{CH}_4} \cdot p_{\text{H}_2\text{O}} / p_{\text{CO}} \cdot p_{\text{H}_2}$  and  $K''_p = p_{\text{CH}_4} \cdot p_{\text{H}_2\text{O}}^2 / p_{\text{CO}_2} \cdot p_{\text{H}_2}^4$  are tabulated over the same range, but it is emphasised that the values of  $K'_p$  can only be strictly accurate at very high temperatures, whilst the applicability of those of  $K''_p$  is limited to lower temperatures. Calculation of the equilibrium condition shows that at 500° the cracked gas contains  $\text{CH}_4$  33.3%,  $\text{H}_2\text{O}$  33.3%,  $\text{CO}$  8.33%, and  $\text{H}_2$  25.0%, whilst at 940° the composition is  $\text{CH}_4$  0.505%,  $\text{H}_2\text{O}$  0.505%,  $\text{CO}$  24.8%, and  $\text{H}_2$  74.1%, when reaction (1) takes place. A similar table is given in connexion with equation (2). Since in practice an excess of steam would be used, calculations have been made to find the effect of two-fold and six-fold excess of steam on the equilibrium when pure methane is used. The influence is considered of the other constituents on the equilibrium amount of methane present of a gas of the following composition: 10% ( $\text{CO} + \text{CO}_2$ ), 30% ( $\text{CH}_4 + \text{C}_n\text{H}_m$ ) 50%  $\text{H}_2$ , 10%  $\text{N}_2$ . Temperature-composition graphs are given for both reactions (1) and (2) with this mixture. At about 820° the methane decomposes to give at equilibrium a gas containing equal proportions of carbon monoxide and dioxide.

H. INGLESON.

**Catalytic conversion of gases containing methane into carbon monoxide and hydrogen.** K. KELLER

and W. KLEMP (Ber. Ges. Kohlentechn., 1930, 3, 230—261).—The reactions studied were (1)  $\text{CH}_4 + \text{H}_2\text{O} = \text{CO} + 3\text{H}_2$  and (2)  $\text{CH}_4 + \text{O} = \text{CO} + 2\text{H}_2$ . The composition of the gas used was approx. 2%  $\text{CO}_2$ , 2%  $\text{C}_n\text{H}_m$ , 0.5%  $\text{O}_2$ , 6%  $\text{CO}$ , 26%  $\text{CH}_4$ , 55%  $\text{H}_2$ , and 8%  $\text{N}_2$ . Poisoning of the catalyst by sulphur compounds present in the gas appears to be due to the formation of nickel sulphide, which, at suitable temperatures, can be decomposed by oxidation with steam and thus prevented from deactivating the catalyst completely. Since reaction (1) is endothermic, considerable difficulty was experienced in maintaining the catalyst at the high temperature required when pure steam was used. In the large-scale experiments a study was made of the best method of combining reactions (1) and (2) to prepare a hydrogen-nitrogen-carbon monoxide mixture in proportions suitable for the subsequent production of a hydrogen-nitrogen mixture in the proportions 3:1. A vertical furnace of alloy steel packed with catalyst was heated externally by gas, and the gas mixture with regulated volumes of steam and air was passed in at the base of the catalyst. With this furnace, which had a layer of catalyst 94 cm. long by 12 cm. in diam., 6 m.<sup>3</sup> of gas, 3 kg. of steam, and 3 m.<sup>3</sup> of air per hr. produced a gas containing (%)  $\text{CO}_2$  2,  $\text{CO}$  17,  $\text{H}_2$  56.5,  $\text{N}_2$  24.5,  $\text{CH}_4$  traces. The highest temperature in the tube was 1050°. In the absence of air it was only possible to convert 3.5 m.<sup>3</sup> of gas per hr. into a methane-free mixture. Increase in the amount of steam does not in all cases increase the methane conversion. In experiments on the carrying out of the reaction in two stages the catalyst was divided into two separate portions. Gas and steam were passed through the first lower-temperature portion and air was added at suitable intervals along the second length of the catalyst. In this way the methane content could be reduced to 0.03—0.09% when a 3:1 hydrogen-nitrogen mixture was required.

H. INGLESON.

#### The water-gas equilibrium and its control.

W. KLEMP and F. BRODKORB (Ber. Ges. Kohlentechn., 1930, 3, 261—274).—Theoretical. The known values for the equilibrium constant of the reaction  $\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{H}_2$  over the range 327—2090° indicate that at higher temperatures the equilibrium is in favour of carbon monoxide-steam. In order to favour the production of hydrogen as low a temperature as is compatible with suitable reaction velocity must be chosen. The use of catalysts alone does not materially assist in the complete removal of carbon monoxide from the gas at temperatures at which it is practicable to work. The theory is discussed of the disturbance of the equilibrium by use of excess of steam and by the removal of the carbon dioxide formed simultaneously with the hydrogen by absorbents such as lime. The pressure of carbon dioxide in the system  $\text{CaCO}_3$ - $\text{CaO}$ - $\text{CO}_2$  limits the temperature at which the latter method of disturbing the water-gas equilibrium can be used to an extreme of 600°. In practice lower temperatures would have to be used. This theoretical study leaves no doubt that the removal of carbon dioxide is the more practicable method of achieving a good conversion of the carbon monoxide. Thus at 550°, to reduce the steam- and carbon dioxide-free gas to the same carbon monoxide

content as that produced by the passage over lime, using one volume of steam, requires a volume of steam twenty times that of the carbon monoxide. By using a slight excess of steam in conjunction with carbon dioxide removed at this temperature the carbon monoxide can be reduced to a mere trace. The foregoing considerations are applied to the gas resulting (cf. preceding abstract) from the action of steam and air on methane.

H. INGLESON.

**Conversion of carbon monoxide by means of steam or air into hydrogen or nitrogen-hydrogen mixtures.** K. KELLER and W. KLEMP (Ber. Ges. f. Kohlentechn., 1930, 3, 274—306).—The disturbance of the water-gas equilibrium by means of substances capable of absorbing carbon dioxide has been investigated on a laboratory and also on a larger scale. In order to combine the function of an absorbent and catalyst in the contact substance various minerals containing iron were examined. The most suitable of these was found to be burnt dolomite, which was able to be regenerated without becoming friable. A working temperature of 480—500° was found to be the most suitable. The study was continued in an enlarged apparatus capable of producing 10 m.<sup>3</sup>/hr. of nitrogen-hydrogen mixture. A detailed description of the regeneration furnaces is given. The gas resulting from the treatment contained small amounts of hydrogen sulphide and carbon monoxide. The former could be removed rapidly at 250—270° at a space velocity of 100—200 litres/hr./100 c.c. over "Luxmasse." Carbon monoxide was removed by passing the desulphuretted gas over a nickel catalyst at 350—400°.

H. INGLESON.

**Large-scale plant at Zeche Viktoria, Lünen, [for preparation of hydrogen and hydrogen-nitrogen mixtures from coke-oven gas].** I—III. R. BESTEHORN (Ber. Ges. f. Kohlentechn., 1930, 3, 306—341, 341—359, 359—362; cf. preceding abstracts).—I. A description is given of the planning and erection of the plant on a scale of 400 m.<sup>3</sup> of hydrogen per hr. To prepare hydrogen, coke-oven gas was passed through the plant in accordance with the following scheme: hydrogen sulphide washer—booster—(with steam) into methane converter—waste-heat boiler—dolomite converters—gas cooler—booster—storage. A diagram is given showing the paths traversed by heating gas, air, and steam. The design of each section of the plant is discussed in detail.

II. [With W. KLEMP, J. SCHROETER, F. BRODKORB, and E. CURLAND.] Working details are given for the preparation of the hydrogen-nitrogen mixture. The coke-oven gas entering the plant contained (%) CO<sub>2</sub> 2.0, C<sub>2</sub>H<sub>4</sub> 2.0, O<sub>2</sub> 0.5, CO 6.5, CH<sub>4</sub> 25.0, H<sub>2</sub> 52.0, N<sub>2</sub> 12.0; the decomposed steam-air-treated gas CO<sub>2</sub> 3.0, CO 16.0, CH<sub>4</sub> 0.0—0.2, H<sub>2</sub> 57.0, N<sub>2</sub> 24.0; and the final gas H<sub>2</sub> 75, N<sub>2</sub> 25%, CH<sub>4</sub> traces. In producing hydrogen the same general procedure was adopted, but since no air could be admitted the maintenance of the temperature in the first stage necessitated an increase in the external heating and, according to the purity of the gas required, a reduction in the volume of gas passing. It is possible, however, to convert 60 m.<sup>3</sup> of coke-oven gas per hr. with a final methane content of 0.0—0.2%.

III. Calculations of cost of running the plant are based on the use of a plant divided into 5 similar units producing conjointly 10,000 m.<sup>3</sup> of hydrogen-nitrogen mixture (3:1) per hr., corresponding to 70 metric tons of combined nitrogen per 24 hrs., reckoning 6% loss in the ammonia conversion. The erection and plant costs total R.M. 3,250,000. The total preparation cost of 1000 m.<sup>3</sup> of mixed gas is R.M. 27.96. The cost of hydrogen (containing 1—1.5% CH<sub>4</sub>, 6% N<sub>2</sub>) would be 3.35 Pf. per m.<sup>3</sup>, whilst a gas with less than 0.1% CH<sub>4</sub> and 6.8% N<sub>2</sub> costs 3.7 Pf. per m.<sup>3</sup>

H. INGLESON.

**Application of the [cracking] process to the manufacture of hydrogen-nitrogen mixtures from gases (e.g., natural gas etc.) rich in methane.** W. KLEMP and F. BRODKORB (Ber. Ges. Kohlentechn., 1930, 3, 362—368). **Cost of the process.** R. BESTEHORN (*Ibid.*, 368—370).—The process is particularly suitable for use with gases rich in methane, e.g., natural gas, distillation gases from lignite and coal, and the methane fractions from liquefaction of coke-oven gas. The chemical and economic advantages of the use of a cheap gas rich in methane over that of coke-oven gas are dealt with. Of the cost of hydrogen-nitrogen mixtures over 60% alone is accounted for in the cost of heating. The firing with a cheap gas of high calorific value would materially reduce working charges. Experiments clearly showed that definite advantages result from the use of gases rich in methane.

When carried out on the same scale as that envisaged with coke-oven gas the cost of 1 m.<sup>3</sup> of hydrogen-nitrogen mixture (3:1) made from practically pure methane would be 1.93 Pf., assuming methane to cost 1 Pf. per m.<sup>3</sup>. With methane at the price  $n$  Pf. the cost is  $(0.6n + 1.34)$  Pf. Hydrogen made from the same source (purest gas) would cost  $(0.74n + 1.85)$  Pf., or (technically pure gas)  $(0.78n + 1.52)$  Pf. The use of pure methane would necessitate the enlargement of the dolomite converters by about one quarter.

H. INGLESON.

**Continuous dehydration and de-oiling of gas tar.** M. A. CABRIER (Gas World, 1930, 93, 413).—Rapid dehydration and de-oiling of tar, to render it suitable for road-surfacing purposes, is carried out at Limoges gasworks in a special apparatus, designed to treat 2 tons of tar per 24 hrs. The crude tar is distilled to the required composition by circulating it, on the thermosiphon principle, through two communicating columns, one cold and the other heated. The vapour passes through an expansion chamber at the top of the heating column and thence to a condenser. It is suggested that the same system can be utilised for the treatment of other gasworks' liquors.

C. B. MARSON.

**Removal of sulphur from brown-coal tar oils by treatment with tar coke.** C. STAEMMLER (Brennstoff-Chem., 1931, 12, 43—45).—Tar oils, containing 0.42, 0.46, and 0.72% S, respectively, were distilled over the coke at 300—600°, both with and without the addition of air or hydrogen. The diminution in sulphur content of the oils, amounting only to 30—35%, was too low for the process to have any technical importance.

A. B. MANNING.

**Extraction of bitumen for purposes of analysis from bituminous road-making materials and**

determination of sulphur in bitumen. F. SEELIG (Chem.-Ztg., 1931, **55**, 145—146).—The constants of a bitumen fluctuate according to the method of dissolving, evaporating the solvent therefrom, and of its complete removal by stirring on a water-bath. A standard method is suggested for the last-named process, in which a vacuum or an atmosphere of nitrogen is employed. Details of an accurate method for determining sulphur in bitumen by combustion in oxygen are given. E. DOCTOR.

Application of a bromine method in determination of phenol and cresols. R. D. SCOTT (Ind. Eng. Chem. [Anal.], 1931, **3**, 67—70).—A method for determining the phenol or cresol content of liquids such as the wastes from dephenolising plants, gas liquors, etc. is described. An aliquot of the suitably purified material, containing about 0.02 g. of phenol or cresol in terms of phenol, is made up to 200 c.c. with distilled water; 25 c.c. of 25% potassium bromide solution are added and then 10 c.c. of dilute (1 : 1) hydrochloric acid. The temperature of the mixture is brought to 25° on a water-bath, 25 c.c. of 0.3% potassium bromate solution are added, and the mixture is kept at 25° for exactly 1 hr. with occasional shaking. Then 25 c.c. of 20% potassium iodide solution are added, and, after being kept at 25° for a further 30 min. with frequent shaking, the mixture is titrated with 0.1*N*-sodium thiosulphate. The results for phenol and *m*-cresol are not greatly affected by changes in the conditions, but with *o*-cresol there is a tendency towards high results and with *p*-cresol towards low results. The above method is an adaptation of the main factors involved in these changes, and it also provides a balance of the errors occasioned by the uneven bromination of *o*- and *p*-cresol. The difference between the figures for the sample and blank determination multiplied by 0.0015675 gives the weight of phenol and cresols in terms of phenol. E. H. SHARLES.

Catalytic reactions of sulphur compounds present in petroleum. I. High-sulphur naphthas in contact with nickel and iron catalysts. J. C. ELGIN, G. H. WILDER, and H. S. TAYLOR. II. Pure sulphur compounds in hydrocarbon materials in contact with nickel catalysts. J. C. ELGIN (Ind. Eng. Chem., 1930, **22**, 1284—1290, 1290—1293).—I. Nickel is more efficient than iron in removing sulphur from naphthas in the vapour phase. Sulphur may be completely removed in contact with the initially sulphur-free nickel catalyst, but catalytic activity decreases as the process proceeds until a state of constant, definite though reduced, activity is reached. Sulphur removed is converted into hydrogen sulphide. Hydrogen added to the vapour stream increases the extent of desulphurisation by effecting the removal of sulphur not affected by the catalyst in its absence. The percentage of sulphur removed under fixed conditions varies with the naphtha studied, and is due to variation in the nature of the sulphur compounds present. Nickel and iron catalysts do not absorb all the sulphur present under the conditions studied. The results suggest that removal of sulphur in the presence of hydrogen may be due to alternate sulphide formation with the catalytic surface and its reduction by hydrogen, and that variation in the

ratio of hydrogen pressure to sulphur compound pressure may increase the effectiveness of the catalytic action.

II. Alkyl mercaptans and sulphides undergo a reaction evolving hydrogen sulphide when in contact with a nickel catalyst at the steady state in the presence of naphtha vapour, the mercaptans being removed more readily than the sulphides. Thiophen was not affected unless hydrogen was added, the catalyst rapidly losing its activity. Addition of hydrogen effects the removal of a larger proportion of propyl sulphide sulphur than when no hydrogen is added. Increasing the ratio of hydrogen to naphtha increases the amount of sulphur removed. The results account for the difference in sulphur removal observed with different naphthas, and indicate that those containing relatively large proportions of thiophen sulphur are the most difficult to reduce catalytically. H. S. GARLICK.

Effect of time and temperature on the cracking of oils. J. C. GENIESSE and R. REUTER (Ind. Eng. Chem., 1930, **22**, 1274—1279).—Atmospheric-pressure vapour-phase cracking experiments were conducted on a representative Midcontinent gas oil. Three sizes of apparatus used were such that the time of contact varied from 0.75 to 4800 sec., and the temperature from 430° to 700°, the quantity of oil passing through the apparatus varying from 3000 to 300 c.c. Detailed results are given for a large number of runs, and a study of these shows that similar results can be obtained by increases in temperature or time. A temperature increase of 17° approximately halves the time of conversion. It is possible to consider time and temperature together as an index, and as this is increased the yield of gasoline increases rapidly to a maximum, which is slightly higher at the lower temperature, and then decreases slowly. The properties of the gasoline are the same for a given time-temperature index, and increase of this results in (a) an increase in the anti-knock value of the fuel, (b) a rapid increase in unsaturation, followed by a slow decrease, and (c) a rapid decrease in the critical aniline solution temperature. Gas yields increase rapidly to a maximum of 60—70% by wt. The gas:gasoline ratio increases considerably with time-temperature and only slightly with temperature for a given index. The unsaturated content of the gas increases with temperature at a given index only under severe cracking conditions. Packed cracking tubes result in higher coke yields, and recycling experiments show a considerable increase in refractoriness of the charging stock. H. S. GARLICK.

Thermal considerations of [mineral oil] cracking processes in baths of molten metals. N. MAYER (Petroleum, 1931, **27**, 141—142).—The coefficient of heat transfer depends not only on conduction, but also on the sp. heat and sp. gr. of a substance. Comparative figures for various metals are given. The best thermal effect will be obtained when the oil is finely dispersed in molten metal and the time of contact adequately extended. E. DOCTOR.

Occurrence of higher fatty acids in mineral oil distillates. E. HOLZMANN and S. VON PILAT (Brennstoff-Chem., 1931, **12**, 41—42).—The acids and phenols extracted from a spindle oil were distilled under reduced pressure, and the acids in the distillate separated from

the phenols by the method previously described (B., 1931, 7). The acid mixture, representing 0.0125% of the original oil, on cooling deposited crystals which were identified as arachidic acid. A further quantity of fatty acids not yet identified was separated from the liquid portion of the acid mixture. A. B. MANNING.

**Methane as a motor fuel.** J. BRONN (Brenstoff-Chem., 1931, 12, 27—29, 45—47).—The methane obtained as a by-product in the production of hydrogen by the fractionation of coke-oven gas at low temperatures forms a suitable fuel for heavy motor vehicles, especially in districts like the Ruhr. The engine requires no modification except replacing the carburettor by a suitable mixing valve. The exhaust gas contains little or no carbon monoxide, and only 1—2% of unburnt methane. No deposits are formed in the cylinder and the consumption of lubricating oil is lower than with liquid motor fuels. In comparative road tests it was found that about 0.9 cub. m. of methane was equivalent to 1 litre of benzol. It is estimated that under suitable conditions the running costs with methane should be about 30% lower than with petrol. A. B. MANNING.

**Knock in internal-combustion engines and the action of anti-knock compounds.** J. LORENTZEN (Z. angew. Chem., 1931, 44, 130—136).—Experiments on the combustion of mixtures of hexane and pentane with air in various proportions, and study of the time-pressure curves obtained, indicate that when knock occurs reactions take place which differ from the normal. Variation of the position of the ignition spark shows that knock is produced only when a wave of incomplete combustion travels through the cylinder, and is the result of the explosion of the intermediate products of combustion by a reflected compression wave when it meets the combustion wave. The normal velocity of propagation of the flame is approximately doubled when knock occurs. The production of turbulence in the gas mixture prior to ignition increases the speed of combustion, and under these conditions knock occurs only at initial pressures greater than 8 atm. If lead tetraethyl be mixed with the fuel before it enters the cylinder knock is not inhibited, but by introducing the products of combustion of the lead compound into the cylinder before ignition of the mixture knock is inhibited at pressures below 5 atm., whilst if turbulence is set up in the mixture knock does not occur at pressures below 9 atm. The theory of the action of anti-knock compounds is discussed.

H. F. GILLBE.

**Progress toward a uniform method of measuring detonation [of fuels].** T. A. BOYD (Ind. Eng. Chem., 1930, 22, 1301—1302).—The Sub-Committee on Methods of Measuring Detonation of the A.P.I. Co-operative Fuel Research Steering Committee has drawn up specifications covering the essential features of a tentative engine. Mixtures of *n*-heptane and isooctane have been adopted to form a standard scale of anti-knock quality, anti-knock values being expressed in terms of an "octane number," i.e., the percentage (by vol.) of isooctane necessary in a heptane-isooctane mixture to match the anti-knock value of any given fuel. H. S. GARLICK.

**Detonation characteristics of some paraffin hydrocarbons.** W. G. LOVELL, J. M. CAMPBELL, and

T. A. BOYD (Ind. Eng. Chem., 1931, 23, 26—29).—The relative knock ratings of 27 paraffin hydrocarbons have been determined in admixture with a commercial gasoline. Apparently in homologous series the tendency to knock increases with increasing length of the carbon chain, whilst in an isomeric series this tendency usually decreases as the number of side-chains is increased. The successive introduction of methyl groups into a carbon chain of given length also decreases the tendency to knock and by substantially a constant increment per methyl group added. H. S. GARLICK.

**Catalytic addition of hydrogen chloride to unsaturated hydrocarbons.** W. J. PIOTROWSKI and J. WINKLER (Przemysl Chem., 1931, 15, 25—36).—The optimal temperature for the reaction of addition of hydrogen chloride to unsaturated hydrocarbons present (32%) in the products of cracking petroleum lies between 70° and 150°; it is advantageous to commence the process at 70°, when chiefly secondary chloro-derivatives are obtained, and to complete it at 150°, when tertiary chloro-derivatives are the main products. The reacting substances should be thoroughly dried, as traces of moisture prevent the formation of secondary chlorides. The reaction is best catalysed by active charcoal saturated with zinc or stannic chloride; these catalysts are inactivated by sulphur-containing substances, which should previously be removed by partly poisoned catalyst before actual contact with the fresh catalyst. The following products were identified:  $\beta$ - and  $\delta$ -chloro- $\beta$ -methylbutane,  $\beta$ -chloropentane, and  $\delta$ -chloro- $\beta\beta$ -dimethylbutane.

R. TRUSZKOWSKI.

**Synthetic lubricating oils.** A. W. NASH, H. M. STANLEY, and A. R. BOWEN (J. Inst. Petroleum Tech., 1930, 16, 830—857).—A detailed account of work on the polymerisation of ethylene, already noted (Stanley B., 1930, 935).

**Acid treatment of lubricating distillates.** H. M. WEIR, W. F. HOUGHTON, and F. M. MAJEWSKI (Ind. Eng. Chem., 1930, 22, 1293—1300).—For the treatment of solar reduced oil sulphuric acid of 93% concentration is superior to 98% as a reagent for the removal of colour. The first action of the acid produces a deeper colour than that of the original oil, but thereafter the colour decreases as more acid is added. Oil loss increases with increase of acid concentration. The effect of temperature on the treatment is not very pronounced. Using small quantities of acid, the colour change is slight between 15.6° and 48.9°. For larger volumes of acid, low temperatures produce better results if the viscosity of the oil is reduced with a light oil. Oil loss does not change with variation of temperature. As the volume and concentration of the acid increase, the A.P.I. gravity increases and the viscosity and refractive index of the finished oil decrease. The heat of acid-treating reaction and change in gravity are directly proportional to the oil-treating loss, and are practically equal for all concentrations of acid. The acid consumed during the reaction is not proportional to the colour removed. The oil is not appreciably changed as a consequence of the action of the acid.

H. S. GARLICK.

**Paraffin concentration plant.** E. A. VON GROELING (*Petroleum*, 1931, 27, 75—83).—The processes in use for the separation and concentration of crude paraffin oils are described and discussed. Maximum concentration is best effected by cooling slowly in stages, pressing to remove crystallised products, and sweating the resulting cakes. The production of smaller cakes results in better separation of the products and greater throughput. Concentration by partial melting and re-pressing of the filter cakes is costly in comparison with other processes. A process combining cooling, pressing, and fractionation is described, and is considered an efficient one. Good separation can also be effected by sweating the filter cakes under pressure, and the method may be worked economically. The comparative working costs of the methods are discussed.

H. E. BLAYDEN.

**Crystallisation of paraffin wax.** E. KATZ (*J. Inst. Petroleum Tech.*, 1930, 16, 870—888).—Polish and Asiatic paraffin waxes were separated into narrow m.p. fractions by sweating and their behaviour when crystallised from solvents was followed. Crystals composed of irregular plates are deposited from saturated solutions in solvents such as benzine, kerosene, lubricating oil, benzol, carbon disulphide, etc. Good hexagonal plates are obtained by using dilute solutions of paraffin wax (m.p. 60—65°) in 96% alcohol. Low-melting paraffin wax crystallises from kerosene in round plates, developing long even needles without a visible layer surface. Needle formation arises in the abnormal course of crystallisation. Mottling in commercial paraffin wax is due to dissolved air or gases, and is the greater the more oil is present. Numerous photomicrographs illustrate the text.

H. S. GARLICK.

**Examination of paraffin, stearines, and similar materials.** F. DANGL (*Petroleum*, 1931, 27, 142—145).—A microscope and polarised light are used for determining the m.p. and solidif. point of paraffin, beeswax, etc.; the method enables an approximate estimate of the percentage of components in the substance to be made.

E. DOCTOR.

**Abstract of recent literature bearing on the hydrogenation of coal.** D. G. SKINNER (*Fuel*, 1931, 10, 109—137).

**Coke-oven walls.**—See VIII. Carbon monoxide filters.—See XXIII.

See also A., Mar., 307, Water-gas. 325, Activated carbons. 330, Gas analysis. Lubricants. 332, Petroleum. 341, Fluoranthene. 362, Berginised pyridine. 375, Cresols. 379, Blood anti-coagulant.

#### PATENTS.

**Furnaces employing pulverulent fuel.** CARBONRUNDUM CO., LTD., Assees. of R. C. BENNER, G. J. EASTER, and B. W. STROMBERG (B.P. 316,266, 25.7.29. U.S., 26.7.28).—The removal of the ash from the furnace is facilitated by adding a reagent, such as rouge, to the fuel, in such quantity as to bring the m.p. of the ash to 50—250° below the flame temperature of the burning fuel. An initial solid deposit of ash is then formed on the inner walls of the chamber and subsequent deposits

remain liquid and flow down to the ash pit. A furnace suitable for operation under these conditions is described.

A. B. MANNING.

**Ovens for producing semi-coke and similar products.** Soc. GÉN. DE FOURN. À COKE SYSTÈMES LECOCQ (B.P. 340,903, 12.4.30. Belg., 13.4.29. Addn. to B.P. 336,809; B., 1931, 143).—The metallic walls of an oven of the type described in the prior patent are constructed of superimposed triangular plates assembled so that they are not deformed by high temperatures and are easily renewed.

C. B. MARSON.

**Mechanical dehydration of raw peat.** W. E. EVANS. From MADRUCK, GES. F. MASCHINELLE DRUCK-ENTWÄSSERUNG M.B.H. (B.P. 338,950, 23.8.29).—The raw material is mixed with dry powdered peat, compressed, and finally heat-treated. The product is cooled again before the dust is recovered therefrom for admixture with a further batch of raw material. The quantity of dust in the mixture is greater than that of dry substance in the raw peat.

A. B. MANNING.

**Carbonisation of coal.** NAT. FUELS CORP., Assees. of G. A. BERRY and A. P. BEARDSLEY (B.P. 340,717, 16.11.29. U.S., 16.11.28).—Bituminous coal, in the form of briquettes or lumps, is converted into artificial anthracite by heat treatment in a suitable apparatus, whereby a smokeless product of the desired size, strength, density, and reactivity is obtained, and the accompanying distillation products are subjected to a minimum cracking effect. An upward or downward current of hot gas is passed through the coal contained in vertical retorts so that its temperature (using Pocohontas coal) is raised at the rate of 5° per min. At the temperature at which thermal decomposition and plasticity begin (275—400°), the current of hot gas entering the retorts is altered so that the rate of heating is less than 0.25° per min.; when the coal reaches a temperature of about 500° the rate of heating is increased again to 5° per min. The final temperature reached may be 600—900°, depending on the reactivity and density required in the final product. The carbonised product is cooled with a current of gas, its resulting sensible heat being used for further carbonisations. The carbonising unit is equipped with suitable regenerators, waste-heat boilers, and condensing equipment. [Stat. ref.]

C. B. MARSON.

**Distillation of lignite.** H. DEBAUCHE (B.P. 339,129, 17.12.29).—The lignite is carbonised in a retort of the type described in B.P. 270,921 (B., 1927, 547), under such conditions that the material is heated gradually and uniformly up to 800° out of contact with the heating gases effecting the distillation and as far as possible out of contact with the gases produced during the distillation. The briquettes made from the product, e.g., with 7½% of pitch and under a pressure of 225 kg./cm.<sup>2</sup>, possess great cohesion.

A. B. MANNING.

**Heat treatment of carbonaceous and other materials.** E. M. SALERNI, and E.M.S. INDUSTRIAL PROCESSES, LTD. (B.P. 338,939, 30.7.29. Cf. B.P. 273,528; B., 1927, 694).—The material is passed continuously through a horizontal retort the floor of which is formed of a number of transversely arranged troughs or depressions. Each trough is provided with

a stirrer comprising a number of paddles mounted on a horizontal shaft, and rotating in a direction opposed to that of the flow of material through the retort. The stirrers are wholly submerged below the level of the material in the retort. Means are provided for regulating the rate of feed to and discharge from the retort.

A. B. MANNING.

**Production of carbon black from oil-refinery residues.** W. W. TRIGGS. From THOMAS CARBON BLACK Co. (B.P. 340,482, 19.9.29).—The heavy distillates from the refinery are cracked under low pressure, and the resulting gas is added to the gas formed during the usual high-pressure cracking operations, the mixed gases then undergoing incomplete combustion.

C. B. MARSON.

**Destructive hydrogenation [of carbonaceous materials].** H. P. DEAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 339,040, 4.10.29).—The hydrogen for the process is brought into indirect effective heat-exchange relationship with the material in the reaction vessel by passing it through a heat exchanger formed of a number of tubular passages or of a coil immersed in the material. The reaction temperature is then readily controlled by varying the degree to which the hydrogen is preheated.

A. B. MANNING.

**Destructive hydrogenation of carbonaceous materials.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 340,470, 28.6.29).—The acid residues obtained during the industrial treatment of crude hydrocarbons with acids or condensing reagents are treated with neutralising or desulphurising agents (*e.g.*, Florida earth, lime, or iron oxide) and destructively hydrogenated at 370–500° with hydrogen under 5–1000 atm. pressure in presence or absence of catalysts immune to sulphur-poisoning (*e.g.*, cobalt sulphide or oxides of molybdenum, tungsten, or chromium).

H. E. BLAYDEN.

**Obtaining valuable distillates by destructive hydrogenation of carbonaceous materials.** STANDARD OIL DEVELOPMENT Co., Assees. of R. P. RUSSELL (B.P. 340,811, 2.1.30. U.S., 22.1.29).—A suspension of the finely-ground carbonaceous material in heavy oil is maintained at 400–450°, and hydrogen at pressures exceeding 25 atm. is passed through the liquid. The vapours produced are continuously withdrawn and condensed. A portion of the heated liquid is continuously withdrawn from the heating vessel, and the pressure upon it reduced so as to vaporise substantially all the liquid constituents, the condensed vapours being mixed with the suspension under treatment; the residue, consisting of ash, non-volatile asphaltic materials, etc., is removed.

H. E. BLAYDEN.

**Gasification of solid fuel.** S. G. S. DICKER. From SOC. ITAL. RICERCH. INDUSTRIALI (S.I.R.I.) (B.P. 338,911, 27.8.29).—The fuel is continuously and completely gasified in a current of oxygen and steam or oxygen and carbon dioxide. The nozzles supplying the gasifying agents extend towards the centre of the lower part of the gas producer in such a manner as to form a sharply defined zone of very high temperature which does not come into contact with the walls of the producer. A vertical wall divides the producer into two

parts, one of which contains the fuel to be gasified, whilst the other forms a duct for the removal of the gas produced. The wall is so placed that a gas mixture substantially free from carbon dioxide passes from a zone close to the high-temperature zone directly into the duct. The ash is carried away with the gas, and no grate is necessary in the producer. A. B. MANNING.

**Manufacture of gases from hydrocarbon materials.** A. S. RAMAGE (B.P. 338,905, 24.8.29).—Olefine hydrocarbons in the gaseous or vapour form, and substantially free from paraffin hydrocarbons, are passed over metallic iron at such a temperature and in the presence of such a concentration of steam that the iron is maintained in the metallic state. A gas is produced consisting principally of carbon monoxide, hydrogen, and methane. It may be scrubbed with wash-oil for the recovery of aromatic hydrocarbons.

A. B. MANNING.

**Production of gas from heavy hydrocarbon oils.** A. S. RAMAGE (B.P. 338,904, 24.8.29. Cf. B.P. 338,905, preceding abstract).—The oil vapours are passed first over ferric oxide and then over finely-divided iron, at progressively higher temperatures (480–700° or higher) and in the presence of progressively higher concentrations of steam (1–30% or more). The products comprise a combustible gas, the calorific value of which may be varied within wide limits (*e.g.*, 400–1500 B.Th.U./cub. ft.) by regulating the working conditions, and an oil, consisting essentially of aromatic hydrocarbons, suitable for use as a motor spirit.

A. B. MANNING.

**Apparatus for manufacture of water-gas.** MOTOR FUEL PROPRIETARY, LTD., and W. JOHNSON (B.P. 338,877, 26.8.29).—The apparatus comprises an outer annular generator containing a charge of carbonaceous material which can be alternately blasted with air and steam, and an inner generator extending vertically through the outer generator. The inner generator is adapted for gasifying pulverised fuel, which is injected thereinto, together with air or steam, through a central jet at the bottom. The inner generator can be heated by hot gases by-passed from the outlet of the outer generator.

A. B. MANNING.

**Manufacture of combustible gas.** HUMPHREYS & GLASGOW, LTD., Assees. of J. A. PERRY and W. H. FULWEILER (B.P. 380,822, 27.1.30. U.S., 6.5.29).—A non-poisonous gas for domestic or industrial use is produced by converting the carbon monoxide of blue-water-gas into methane. The carbon monoxide content of the blue-gas is reduced by appropriate means (*e.g.*, by washing it with ammoniacal solutions of copper salts) so that the CO:H ratio is about 1:3. The carbon monoxide and hydrogen in the product are converted into methane and water by passing the mixture over a heated catalyst (*e.g.*, nickel), the water being removed by condensation. The carbon monoxide removed from the original gas is recovered (*e.g.*, by heating the washings), mixed with steam, and treated with a catalyst (*e.g.*, heated activated iron oxide) so as to convert the mixture into hydrogen and carbon dioxide, which is removed by scrubbing the gas with water. The hydrogen and converted blue-gas are combined to give a gas having  $d_{0.41}$  and heating value approx. 540 B.Th.U./



cub. ft. Coal gas may be treated in a similar manner, and the hydrogen produced may be mixed with the treated coal gas or the converted or partly converted blue-gas. H. E. BLAYDEN.

**Utilisation of gases produced through distillation or similar treatment of bituminous materials, especially of the by-products of gas- and coke-manufacturing plants.** R. TERN (B.P. 338,869, 22.8.29).—The ammonia expelled from the gas liquor in known manner, and the combustion products of the gas-purification masses, are mixed and subjected to the influence of high-tension electrical currents. The solid product is used as a fertiliser. A. B. MANNING.

**Manufacture of hydrogen or of gases containing hydrogen from methane and similar hydrocarbons or gases containing these hydrocarbons.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 341,462, 3.10.29).—Suitable apparatus is that described in B.P. 328,643 (B., 1930, 751). A generator is surrounded for part of its height by a jacket and the carbon monoxide produced during the "blow" period is burned in the jacket with the aid of secondary air, the heat produced being transmitted to the coke in the generator. Hydrocarbon gas is then admitted through the jacket, where it is preheated, and then passes upwards through the coke bed, where it is decomposed into hydrogen and carbon, the latter being deposited on the coke. This carbon is burned during the "blow" period, and supplies sufficient heat for the process if pure hydrocarbon gases are used. If the hydrocarbon gases are diluted with inert gas or steam it is necessary to add coke. T. A. SMITH.

**Decomposition under the action of heat of a mixture of methane or other hydrocarbons and water vapours.** UNION CHIM. BELGE SOC. ANON. (B.P. 341,393, 3.9.29. Belg., 22.6.29).—The decomposition is effected in a heat exchanger in which the heating gases and gas to be treated pass countercurrent to each other. Packing material to retain the heat is supplied and the regenerative principle is also adopted. T. A. SMITH.

**Removal of hydrogen sulphide from gases.** C. J. HANSEN, Assee. of H. KOPPERS A.-G. (B.P. 340,663, 21.10.29. Ger., 22.11.28).—The gases are scrubbed with polythionate liquors containing iron, the formation of iron sulphide being inhibited by the addition of sulphurous acid to the scrubbing liquid. The used scrubbing liquors are treated with sulphur-containing gases to liberate iron sulphide, ammonium sulphate, and sulphur; the iron sulphide thus obtained is treated with sulphurous acid and the solution used cyclically in the process. The process may be carried out in a tower which is divided into an upper chamber, in which the liquor absorbs sulphurous acid, and a lower chamber, in which the gas is scrubbed. The scrubbing liquid is circulated continuously through the two chambers. C. B. MARSON.

**Purifying from naphthalene the distillation gases from coal.** GEWERKSCHAFT M. STINNES (B.P. 317,780, 25.7.29. Ger., 22.8.28. Cf. B.P. 305,545 and 311,326; B., 1930, 1055; 1931, 192).—Gases which are purified from naphthalene by washing with benzene, charging

them at a raised temperature with a solvent for naphthalene, and then cooling, are subjected to a further washing with a high-boiling oil, free from naphthalene, in order to remove any residual naphthalene, naphthalene solvent, or benzene. The oil after absorption of these residues is used in the initial stage of the naphthalene washing. The high-boiling oils are freed from naphthalene by passing a stream of gas, preferably free from oxygen, through the heated oil. A. B. MANNING.

**Purification of the gases containing acetylene obtained by treatment of carbonaceous substances in the electric arc.** I. G. FARBERIND. A.-G. (B.P. 340,787, 6.1.30. Ger., 16.1.29).—The gases are rendered suitable for the preparation of acetylene conversion products (e.g., acetaldehyde) by passing them over active porous material (e.g., active carbon or silica) to remove diacetylene and similar substances which produce colour in the products, and washing with ferrous sulphate or alkalis before or after treatment with the active material in order to remove cyanogen compounds. The substances extracted from the mixture are recovered. H. E. BLAYDEN.

**Utilisation of tar oils from low-temperature distillation of carbonaceous materials.** A. S. RAMAGE (B.P. 340,512, 17.9.29).—Vaporised tar oils from low-temperature distillation processes are mixed with an excess of water-gas and passed over metallic iron at 590—650°. The aromatic hydrocarbons produced are separated by condensation and scrubbing with a wash-oil. H. E. BLAYDEN.

**Recovery of benzene from aqueous solutions thereof.** KLÄR- U. ENTPHENOLUNGS-GES.M.B.H., Assees. of C. OTTO & Co., G.M.B.H. (B.P. 341,302, 10.2.30. Ger., 9.2.29).—Benzene is removed from waste waters from which phenol has been benzene-extracted by blowing in warm air and/or steam under pressure, e.g., in a sprinkler tower. C. HOLLINS.

**Treatment of [mineral] oils.** W. G. LEAMON (U.S.P. 1,769,788 and 1,769,790—5, 1.7.30. Appl., [A] 4.11.21, [B] 1.5.26, [C] 24.2.23, [D] 10.3.23, [E] 2.11.23, [F] 26.6.25, [G] 26.8.25. Renewed [C] 28.12.25, [F] 2.4.29).—(A) A relatively heavy mineral oil is distilled to pitch or coke, the distillate fractionated, and that portion condensing at or above 360° redistilled. The fraction condensing at 175—360° is cracked in the vapour phase in contact with a non-metallic refractory material of the pumice type kept at 540—575°. Tarry products formed in the cracking chamber are returned to the coking still. Cracked vapours are led away and fractionally condensed, the fraction containing kerosene being returned to the cracking chamber and the lightest fraction recovered as commercial condensate. (B) Vapours leaving the cracking chamber are separated from the fixed gases, which are metered, and by means of a differential pressure gauge in conjunction with electric temperature-controlling means, the temperature of the cracking chamber is automatically governed by the rate of flow of the fixed gases. (C) Cracked petroleum distillates, d 0.730—0.830, are stabilised by subjecting them to the action of <1% of a catalytic metal halide, e.g., anhydrous aluminium chloride, dissolved in ethyl ether, at a temperature favourable to polymerisation, but insufficiently



high to effect cracking; gummy and polymerisation products are separated from the distillate by redistillation. (D) A cracked motor fuel distillate is stabilised by treatment at ordinary temperatures with about 0.05% of anhydrous zinc chloride dissolved in methylated ether; precipitated polymerisation products are separated from the treated distillate by filtration through an absorbent. (E) A cracked motor fuel distillate is redistilled in the presence of a liquid mixture of zinc chloride and methylated ether, in which the quantity of zinc chloride is greater than can be held in true solution and does not exceed a minor fraction of 1% of the distillate being treated, the vapours being condensed to a motor fuel free from resinous or gummy matter. (F) Crude motor spirit is subjected to the combined action of a granular solid absorptive material, *e.g.*, fuller's earth, and a solution of a catalytic metal compound in a non-aqueous solvent miscible with the mineral oil, *e.g.*, zinc chloride dissolved in methylated ether, and the solid material and resultant polymerised products are separated from the motor fuel by distillation. The addition of a small quantity of dehydrating agent, *e.g.*, plaster of Paris, is recommended. (G) A mineral oil containing gum-forming constituents is subjected to the polymerising action of a solution of one or more catalytic metallic halides, *e.g.*, zinc chloride, alone or with a small proportion of aluminium chloride, in ethyl acetate alone or mixed with *n*-propyl alcohol. The addition of a very small quantity of an acid, *e.g.*, sulphuric or acetic, to the treating solution accelerates its action.

H. S. GARLICK.

**Continuous distillation of mineral oils and the like.** SOC. POUR L'EXPLOIT. DES PROC. AB-DER-HALDEN (B.P. 340,370 11.11.29. Fr., 17.1.29).—Coils for the superheating of steam, a still in which oil is passed into a supply of hot oil while superheated steam is admitted, and a preheating coil for the oil are all mounted in one setting so that one furnace may be used for heating the whole series. The hot gases from the furnace pass first over the steam-heating coils, then through the chamber containing the still, and finally over the preheating coils. The arrangement gives considerable fuel economy.

T. A. SMITH.

**Polymerisation of unsaturated hydrocarbon gases.** C. R. WAGNER (B.P. 341,391, 8.7.29).—Polymerisation reactions are exothermic and may give rise to high temperatures with formation of carbon and reduction of yield of desired products. This is avoided in practice by passing the furnace gases along banks of heating tubes in the same direction as the gas which is being heated. By this means the gas is rapidly heated to the desired temperature and during polymerisation loses heat to the furnace gases which are now cooler than the gas in the heating tubes. The gases are heated to 440–540° at 300–1000 lb./in.<sup>2</sup> pressure. T. A. SMITH.

**Preparation of high-molecular hydrocarbons.** J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 340,293, 27.7.29).—Boric acid and an organic compound containing two or more hydroxyl, carbonyl, or carboxyl groups on the same or on two adjacent carbon atoms, such as glycerol or lactic acid, may be used as a condensing agent for unsaturated hydrocarbons. Thus brown-coal tar, boiling range 225–325°, is agitated by means of

air at 130° with 10% of a 3:1 mixture of oxalic acid and boric acid for 4 hrs. After separating the deposits the oil yields 43% of lubricating oils on distillation.

T. A. SMITH.

**Refining of hydrocarbons.** T. T. GRAY, ASST. to GRAY PROCESSES CORP. (U.S.P. 1,768,683, 1.7.30. Appl., 12.11.26).—Cracked hydrocarbons containing sulphur are distilled in the presence of copper (or mercury) acetate. The vapours are passed through solid absorbents such as bauxite or fuller's earth, whereby the unsaturated compounds become polymerised and are returned to the still. Stable, sweet gasoline is obtained.

T. A. SMITH.

**Production of sulphonic acids from hydrocarbon oils.** G. PETROFF and P. SHESTAKOFF, ASSRS. to FIRST NAT. BANK (U.S.P. 1,761,744, 3.6.30. Appl., 30.12.25. Ger., 26.1.25).—White oil or liquid paraffin is oxidised by a current of air at 140–180° until it is dehydrogenated, whereby a small amount of fatty acid is produced, the product being then readily sulphonatable. The sulphonic acids may be used in the hydrolysis of fats, as emulsifying agents, or in the textile industries.

T. A. SMITH.

**Separation of sludge from petroleum residues.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 340,314, 26.9.29. Holl., 7.11.28).—The sludge is removed by diluting the residue with cracked distillate, pressure-distillate bottoms, etc. at 100°. The mixture is maintained at this temperature for 24 hrs., filtered, and the greater portion of the diluent distilled off.

T. A. SMITH.

**Working up the waste sulphuric acid produced in the washing of mineral and tar oils with sulphuric acid.** CHEM. FABR. BORK G.M.B.H., and J. GRUBENBECHER (B.P. 340,816, 23.1.30).—The waste acid is taken to the top of a tower, from which it is allowed to trickle downwards through a countercurrent of acid tar-oil vapours, which dissolve the resinous impurities in the sulphuric acid and collect as a light layer of liquid tar oil-resin mixture above the concentrated acid in the lower part of the tower; final traces of resins, acid tar condensates, and hydrocarbons still present in the sulphuric acid are removed by the addition of small amounts of water, the sulphuric acid then separating pure and quantitatively from the tar oil-bitumen mixture. The tar oil-bitumen mixtures may be worked up and used as a waterproofing material and in tarred road construction.

C. B. MARSON.

**Motor spirit.** A. RICHMOND (B.P. 341,415, 10.10.29).—The tar obtained by the carbonisation of bituminous coal at 550–600° is distilled up to 125–150° in a slow current of inert gas to remove aqueous liquors and light oils. The pressure is then reduced to 2–4 mm. and the second distillate (5–10%, b.p. 120–220°) is washed free from acids and bases and dried. The addition of 1 pt. of this oil to 40 pts. of petrol and 40 pts. of kerosene gives a satisfactory motor spirit.

T. A. SMITH.

**[Lubricating or insulating] oil composition.** H. K. IRRIG (U.S.P. 1,768,910, 1.7.30. Appl., 22.6.27).—Bases are separated from Californian gas oil by treatment with dilute acid. The acid extract is neutralised and steam-distilled. The bases may require purification by

redistillation or treatment with oxidising agents. Lubricating oils are improved by the addition of 1% of the bases.

T. A. SMITH.

**Manufacture and use of paraffin emulsion.** G. M. CLARK. From AGASOTE MILLBOARD Co. (B.P. 341,416, 11.10.29).—A mixture of montan wax (20–25 wt.-%) with paraffin wax and water is heated. The montan wax is then saponified by means of alkali and agitation is continued until the paraffin wax solidifies. Waterproof millboard or paper is produced by adding this emulsion to pulp (1–4 pts. of paraffin per 100 pts. of fibre).

T. A. SMITH.

**Burners for furnaces fired with pulverised, liquid, or gaseous fuel.** CLARKE, CHAPMAN & Co., LTD., and W. A. WOODSON (B.P. 342,797, 12.11.29).

**Burners for pulverised coal.** ALLGEM. ELEKTRICITÄTS GES. (B.P. 343,177, 19.11.29. Ger., 23.9.29).

**Apparatus for burning pulverised fuel.** J. G. MCKEAN and R. F. JONES (B.P. 342,905, 12.2.30).

**Apparatus for separating coke from quenching water.** SOUTH METROPOLITAN GAS Co., and C. C. CARPENTER (B.P. 342,714, 30.10.29).

**Dryers etc.**—See I. Polymerisation and oxidation of olefines. Alcohols. Esters. Carbazole. Storage of liquids.—See III. Carbon monoxide.—See VII. Tarred macadam. Structural materials.—See IX. Lubricants.—See XII.

### III.—ORGANIC INTERMEDIATES.

**Determination of water in glycerols.** H. F. HOYT and P. C. CLARK (Oil & Fat Ind., 1931, 8, 59–61).—Water in concentrated glycerols may be determined satisfactorily by application of the Bidwell–Sterling distillation method (cf. B., 1925, 268), using toluene as the boiling liquid and distilling for 1½–2 hrs. A very small amount of glycerol is carried over (0.1 g. or less; correction —0.15% or less on samples containing up to 10% of water), which may be determined in the distillate by the acetin method if the highest accuracy is required. Solvents of higher b.p., e.g., xylene, entrain more glycerol; isopropyl ether and benzene are not suitable as solvents.

E. LEWKOWITSCH.

**Phenols etc.**—See II.

See also A., Mar., 330, Vacuum fractionation. 333, Absolute alcohol. 334, Carboxylic anhydrides. 339, Nitriles. 341, Anthracene derivatives. Nitrocompounds. 343, Azo intermediates. 347, Detection of  $\beta$ -naphthol. 348, *iso*Eugenols. 354, Acetophenone. 355, Reduction of hydroxyanthraquinones. 375, Colour reactions.

#### PATENTS.

**Polymerisation of olefines.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 340,513, 20.9.29).—The polymerisation of ethylene, propylene, etc. is catalysed by phosphoric acids or salts or esters or phosphorus pentoxide below 400°, preferably in presence of a carrier, such as active charcoal, Florida earth, or silica gel, and at increased pressures. Liquid products are obtained.

C. HOLLINS.

**Polymerisation of diolefines.** J. Y. JOHNSON.

From I. G. FARBERIND. A.-G. (B.P. 340,008 and 340,474, [A, B] 19.8.29, [B] 7.5.30).—(A) The polymerisation of diolefines by means of alkali or alkaline-earth metals proceeds more uniformly in the presence of 0.05–5% of acetals, ketones, aldehydes, esters, or other compounds in which two valency linkings of a carbon atom are linked to one or two oxygen atoms, including organic acids when used in traces. Examples are: dibutyl acetal, ethylidene glycol, acetal from acetaldehyde (or crotonaldehyde) and  $\alpha$ -butylene glycol, benzophenone, stearic acid and vinyl ethyl ether, ethyl phthalate, oleic acid. (B) Butadiene (etc.) is polymerised smoothly by the usual catalysts (alkali metals etc.) in presence of up to 20% of an organic compound unattacked by sodium, but capable of replacing sodium by hydrogen in sodium triphenylmethyl, e.g., dioxan, furan, thiophen, acrylonitrile, phenylacetoneitrile, methylacetylene. Preferably there is also present an unsaturated (especially aliphatic) ether, e.g., vinyl,  $\alpha$ -styryl, propenyl, allyl, and propargyl ethyl ethers, vinyl isopropyl ether, *m*-tolyl vinyl ether,  $\alpha$ -naphthyl vinyl ether.

C. HOLLINS.

**Manufacture of oxidation products from hydrocarbon materials.** C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 341,130, 19.10.29).—A hydrocarbon, heated to below cracking temperature (e.g., 200–600°), is added to a mixture of oxygen and hydrocarbon below oxidation temperature (e.g., at 150°) in a reaction zone, whereby the temperature of the mixture is raised and oxidation occurs; the pressure should be 5–230 atm. Preferably the mixture of hydrocarbon and oxygen is made by dissolving oxygen under pressure in the hydrocarbon, e.g., by a countercurrent system. Pentane is thus oxidised at 278°/145 atm. to acetaldehyde, acetic acid, acetone, and ethyl, propyl, and butyl alcohols.

C. HOLLINS.

**Polymerisation of unsaturated organic compounds.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 340,004, 12.8. and 4.9.29).—Carbonyls of heavy metals catalyse the polymerisation of unsaturated fatty acids, their esters, diolefines, styrene, etc. Linseed oil with 1% of iron in a current of carbon dioxide gives after 4–5 hrs. at 300° a viscous oil, and after a further 2 hrs. an elastic solid; or with 0.35% of cobalt carbonyl in carbon dioxide after 5 hrs. at 300° a jelly; isoprene is converted into a rubber-like mass when gently warmed with 2% of iron carbonyl in a stream of air. Nickel carbonyl or iron carbonyl added with chromium propylnaphthalenesulphonate to a paint of polymerised butadiene in toluene accelerates hardening of the film.

C. HOLLINS.

**Catalytic hydrogenation and dehydrogenation of organic compounds. [Manufacture of alcohols from carbon oxides.]** E. I. DU PONT DE NEMOURS & Co. (B.P. 313,093, 6.6.29. U.S., 6.6.28).—As catalyst there is used a mixture of difficultly reducible basic and acidic oxides, together with 0.25–5.0% of an alkali metal compound, the acidic oxide being in excess; e.g., zinc oxide and chromium oxide, with 5% of anhydrous sodium chromate. Water-gas passed over this catalyst at 475°/275 atm. at a space-velocity of 40,000 gives a product (equal per hr. to volume of catalyst) containing 40% of methyl alcohol.

C. HOLLINS.

**Synthesis of alcohols [ethyl and higher alcohols from carbon monoxide and hydrogen].** G. NATTA (B.P. 340,656, 16.10.29).—The potassium, rubidium, or cesium salts of aliphatic acids, especially potassium formate, with or without the acetate, are used as catalysts on a porous carrier, *e.g.*, smithsonite.

C. HOLLINS.

**Manufacture of alcohols [from alkyl hydrogen sulphates etc.].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 341,167, 6.11.29).—A sulphuric, phosphoric, or other acid of suitable concentration for absorption of olefines is obtained by adding ammonium salt to the acid; *e.g.*, isobutylene is absorbed in sulphuric acid diluted with ammonium sulphate solution to 65% concentration.

C. HOLLINS.

**Manufacture of ketones [acetone from alcohol].** H. DREYFUS (B.P. 340,233, 19.8.29).—Primary aliphatic alcohols (except methyl), or corresponding aldehydes or esters, are led with oxygen, and preferably with an aliphatic acid vapour, at 350–550° over a metal oxide, hydroxide, or carbonate other than compounds of alkali or alkaline-earth metals (though these may be used in addition). Ethyl alcohol and acetic acid, passed with oxygen through a copper tube filled with ferric oxide and calcium carbonate at 450–480°, yield acetone.

C. HOLLINS.

**Manufacture of aliphatic [acetic] anhydrides.** BRIT. CELANESE, LTD., H. F. OXLEY, and L. FALLOWS (B.P. 340,484, 23.9.29).—Silica gel, with or without other catalysts (*e.g.*, calcium tungstate), converts acetic acid vapour into acetic anhydride at 550–650°. The exit vapours may be washed with benzene to remove the anhydride.

C. HOLLINS.

**Manufacture of aliphatic acids and esters [from ethers and carbon monoxide].** H. DREYFUS (B.P. 340,939, 1.10.29. Addn. to B.P. 319,030; B., 1930, 95).—Suitable catalysts for the process of the prior patent are non-acidic substances known to catalyse the production of acids from alcohols and carbon monoxide, *e.g.*, zinc oxide and/or tin oxide with or without sodium methoxide at 300–400°/100–200 atm.

C. HOLLINS.

**Manufacture of derivatives of  $\alpha$ -oxyallyl alcohol (propene-2 : 3-diol) [ $\beta$ -dihydroxypropylene].** SCHERING-KAHLBAUM A.-G. (B.P. 339,882, 17.2.30. Ger., 23.2.29).—The acetone compound of glycerol  $\alpha$ -chlorohydrin or similar compound,  $\text{CH}_2(\text{OH})\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\text{X}$  (X being halogen, arylsulphonyl, etc.), is distilled over caustic alkali to give the acetone compound of  $\beta$ -dihydroxypropylene, b.p. 107–108°/782 mm., from which the acetone may be removed by oxidation, *e.g.*, with perbenzoic acid. Organic tertiary bases (quinoline, diethylaniline) may replace the caustic alkali.

C. HOLLINS.

**Catalytic production of esters [by heating alcohols].** E. I. DU PONT DE NEMOURS & CO. (B.P. 313,575, 14.6.29. U.S., 14.6.28).—A primary alcohol vapour is passed at 350–400° over a difficultly reducible metal oxide, preferably of a group II metal, and preferably in combination with an acidic metal oxide (*e.g.*, as a chromite). A pressure of 70–280 atm. is advantageous. Examples are: *n*-butyl *n*-butyrate from butyl alcohol over manganese oxide; methyl formate from methyl

alcohol over magnesium, cadmium, and chromium oxides; ethyl acetate from ethyl alcohol over zinc chromite or the calcined precipitate from stannous chloride, ammonia, and ammonium chromate, or zinc copper chromite, or zinc cadmium chromite. [Stat. ref.]

C. HOLLINS.

**Preparation of organic compounds [sulphuric esters, alcohols, etc.] from hydrocarbons having one double linking in the molecule.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 339,592, 6.8.29).—Olefines are absorbed in sulphuric acid in presence of the catalysts of B.P. 323,748 (B., 1930, 359), 336,603–4 and 336,633 (B., 1931, 148). *E.g.*, cyclohexane is dissolved in 80% sulphuric acid containing 3% of ferricyanic acid; hydrolysis gives cyclohexanol.

C. HOLLINS.

**Production of vinyl esters of the halogen hydracids [vinyl halides].** I. G. FARBERIND. A.-G. (B.P. 339,727, 17.10.29. Ger., 7.12.28).—Acetylene and hydrogen halide are caused to react at 180° in presence of active carbon free from activating agents or containing residual phosphoric acid.

C. HOLLINS.

**Assistant and auxiliary agent for use in the textile and allied industry.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 340,272, 19.7.29).—Sulphonic acid derivatives of the esters or amides of tall oil are produced under severe conditions of working (cf. B.P. 272,967 and 303,917; B., 1928, 703; 1929, 236); these are stable to hot acids, soluble in concentrated caustic lyes and hard water, suitable for use as wetting agents, detergents, emulsifiers, etc., and form soluble salts with organic or inorganic bases. The products can be improved by the introduction of alkyl, hydroxyalkyl, or aralkyl groups by esterification, *e.g.*, with dimethyl sulphate.

E. LEWKOWITSCH.

**Manufacture of basic products derived from higher fatty acids.** I. G. FARBERIND. A.-G. (B.P. 339,359, 7.8.29. Addn. to B.P. 317,325; B., 1931, 237).—A primary or secondary arylamine, having a tertiary amino-group in a side-chain, is converted into its acyl derivative by heating with a higher fatty acid, ester, amide, acid chloride, or imino-ether, the condensation of oleyl chloride with *p*- $\beta$ -diethylaminoethoxyaniline being excluded. The palmityl derivatives of *p*- $\beta$ -diethylaminoethoxyaniline, m.p. 82° (hydrochloride, m.p. 108–111°),  $\beta$ -diethylaminoethyl *p*-aminobenzoate (hydrochloride, m.p. 116°), and *as*-ethyl- $\beta$ -diethylaminoethyl-*p*-phenylenediamine (hydrochloride, m.p. 81°), and the oleyl derivative of *as*-methyl- $\beta$ -diethylaminoethyl-*p*-phenylenediamine are described.

C. HOLLINS.

**Manufacture of condensation products from cyclohexylamines [and halogenated nitro-compounds].** W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 340,495, 30.9.29).—cycloHexylamines are condensed with nitro-compounds containing reactive halogen. The products may be reduced, desulphonated (with hot dilute sulphuric or hydrochloric acid), or acylated and reduced with or without subsequent hydrolysis. The following cyclohexylamines are described: 2:4-dinitro-, m.p. 154°; 4-chloro-2-nitro-, m.p. 104; 4-nitro-

2-sulpho- (yellow acid dye); 2:6-dinitro-4-sulpho- (yellow); 4-amino-2-sulpho-; 4-nitro-2-carboxy-, m.p. 253° (decomp.); 2-nitro-4-sulpho- (yellow); 2:4-dinitro-6-sulpho-, m.p. 240° (sodium salt, m.p. 260°, reddish-yellow); 4-nitro-, m.p. 100° (acetyl compounds, m.p. 114°, reduced, m.p. 143°; benzoyl compounds, m.p. 149°, reduced, m.p. 185°; 2:5-dichlorobenzoyl compound, m.p. 206°, reduced, m.p. 196°; *m*-nitrobenzoyl compound, m.p. 147°; reduced, m.p. 180°); 2-nitro-, m.p. 104°; 2:4-dinitro-, m.p. 154°; 4-amino-2:4-Dinitrocyclohexyl- $\alpha$ -naphthylamine, and products from *o*- and *p*-methylcyclohexylamines with 1-chloro-4-nitrobenzene (acetyl compounds, m.p. 112° and 135°, respectively, reduced, m.p. 84° and 140°, respectively) are also described. C. HOLLINS.

**Manufacture of aromatic amino-aldehyde compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,699, 24.9.29).—Aldehydes of the benzene or anthraquinone series containing reactive halogen substituents are condensed with carboxylamides or sulphonamides (which may carry *N*-alkyl-, -aryl, or -aralkyl groups) in presence of acid-binding agents, and the products are hydrolysed. 2-Chloro-5-nitrobenzaldehyde with *p*-toluenesulphonamide, potassium carbonate, copper, and cuprous chloride at 170–190° gives 5-nitro-2-*p*-toluenesulphonamidobenzaldehyde, m.p. 181–182° [phenylhydrazone, m.p. 214° (decomp.)], which is hydrolysed to 5-nitro-2-aminobenzaldehyde by 90–95% sulphuric acid at 60–80°. The following compounds are also described: 2-*p*-toluenesulphonamidobenzaldehyde, m.p. 203–205°; *N*-*p*-toluenesulphonyl-2-*p*-toluidinobenzaldehyde (converted into 2-methylacridine by sulphuric acid); 6-chloro-2-*p*-toluenesulphonamidobenzaldehyde, m.p. 150° (phenylhydrazone, m.p. 166°); 3-nitro-2:6-di-*p*-toluenesulphonamidobenzaldehyde, m.p. 162° (phenylhydrazone, m.p. 228°), and 3-nitro-2:6-diaminobenzaldehyde, m.p. 250–251°; 2:5-di-*p*-toluenesulphonamidoterephthalaldehyde and the 2:5-diamino-aldehyde, m.p. above 300°; 1-*p*-toluenesulphonamido-2-aldehydoanthraquinone. C. HOLLINS.

**Manufacture of carboxylic acid arylamides of the benzene series.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 340,661, 18.10.29).—Aroyl-aminoaroylaminophenols having no free carboxylic or sulphonic groups are prepared; they have affinity for cotton, and are suitable coupling components for ice colours. Salicylic acid is condensed with *p*-aminobenzoic acid in toluene in presence of phosphorus trichloride at 110°, and the product is condensed with *m*-aminophenol in dimethylaniline with addition of phosphorus trichloride at 110°. The product, m.p. 280° (decomp.), from *m*-cresotic acid and *m*-aminobenzoic acid is similarly condensed with *m*-aminophenol (product, m.p. 215°) and 5-amino-*o*-cresol (product, m.p. 180–182°); the product, m.p. 258–261°, from *m*-cresotic and 2-amino-*p*-toluic acids gives with *p*-aminophenol a compound, m.p. 240–243°. *p*-(3-Hydroxy-*p*-toluyl)aminobenzoic acid, m.p. above 350°, with *m*-aminophenol (product, m.p. 245–247°), and *p*-(2-hydroxy-*o*-toluyl)aminobenzoic acid, m.p. 274°, with *m*-aminophenol (product, m.p. 293–294°) give similar products. C. HOLLINS.

**Manufacture of [nuclear] alkylated phenols.** SCHERING-KAHLBAUM A.-G. (B.P. 340,682, 31.10.29. Ger., 29.11.28).—A dihydroxydiphenylmethane is heated with cyclohexanol or other hexahydrophenol at 220° in presence of a hydrogenation catalyst to give a phenol, a nuclear alkylated phenol, and a cyclohexanone. 4:4'-Dihydroxy- $\alpha$ -diphenylethane gives phenol and *p*-hydroxyethylbenzene. *p*-Hydroxyisopropylbenzene is obtained from 4:4'-dihydroxy- $\beta$  $\beta$ -diphenylpropane, and thymol (with *m*-cresol) from 2:2'-dihydroxy-4:4'-dimethyl- $\beta$  $\beta$ -diphenylpropane (prepared from acetone and *m*-cresol). C. HOLLINS.

**Manufacture of non-dyeing thio-derivatives of phenols.** I. G. FARBENIND. A.-G., and A. THAUSS (B.P. 340,007, 19.8.29. Addn. to B.P. 173,313 and 298,280; B., 1922, 139 A; 1928, 886).—In the process of B.P. 298,280 an excess of 50–150% of phenol facilitates the sulphurisation and the melt remains liquid. C. HOLLINS.

**Manufacture of ketone hydrazones of the phenylhydrazinesulphonic acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,619, 5.10.29 and 3.7.30).—Phenylhydrazine-*m*- and -*p*-sulphonates react with dialkyl, aryl alkyl, and hydroaromatic ketones if the solutions are maintained neutral to litmus. The alkali phenylhydrazine-*o*-sulphonates react with dialkyl and aryl alkyl ketones in aqueous solution. The resulting hydrazones give indolesulphonic acids when fused with zinc chloride. Examples are *o*-sulphophenylhydrazones of acetophenone and acetone; *m*-sulphophenylhydrazones of acetophenone, *p*-methylacetophenone, *p*-chloroacetophenone, and acetone; *p*-sulphophenylhydrazones of *p*-chloroacetophenone and acetone. C. HOLLINS.

**Manufacture of styrenes and their homologues.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,587, 30.9.29 and 26.4.30).—Ethylbenzene, or a homologue, is passed at 500–800° over a dehydrogenating catalyst, preferably comprising an irreducible metal compound, with 1–3% of a reducible metal compound. The catalyst may be pretreated with steam, nitrogen, hydrogen, ammonia, etc. at 300–600° to modify its surface and prevent cracking of the ethylbenzene. Suitable catalysts are: calcium oxide with 10% of magnesia (pretreated with ethylene at 700°) at 650°; aluminium phosphate and magnesia (pretreated with petrol at 700°) at 630°; silica gel (pretreated with acetylene and ethylene at 650°) at 650°; alumina, zinc oxide, and chromium oxide (pretreated with nitrogen at 580°) at 580° (this gives 65% of styrene); molybdenum oxide on pumice; zinc oxide; tungsten oxide, zinc oxide, alumina, and Florida earth; lithia on active carbon; molybdenum sulphide, alumina, and Florida earth, etc. C. HOLLINS.

**Recovery of carbazole.** I. G. FARBENIND. A.-G. (B.P. 340,585, 28.9.29. Ger., 24.10.28).—Tar fractions containing carbazole are treated with hydrogen, e.g., at 440°/200 atm. in presence of a molybdenum–chromium–manganese catalyst, whereby other substances present are converted into low-boiling liquids, and carbazole can be isolated by filtration from the cooled condensate or by distilling off the impurities. C. HOLLINS.

**Manufacture of derivatives of carbazole.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,550 and 340,625, 30.8.29).—(A) Nitroamino- or aminohydroxycarbazoles are deaminated by usual methods. 3-Nitro-2-aminocarbazole, m.p. 233°, gives 3-nitrocarbazole, m.p. 175°, which is reduced to the amine, m.p. 238° (formyl derivative, m.p. 239–240°; acetyl, m.p. 243–244°; benzoyl, m.p. 276–277°; toluene-*p*-sulphonyl, m.p. 188–189°); 1-nitro-2-aminocarbazole, m.p. 177°, gives 1-nitrocarbazole, and 4-hydroxycarbazole is obtained from 4-amino-9-hydroxycarbazole. (B) Halogens are removed by known methods from substituted halogenated carbazoles. 4-Aminocarbazole, m.p. 196°, is obtained from its 2:8-dichloro-, -dibromo-, or -diiodo-derivative by boiling in alcohol with alcoholic potassium hydroxide, palladised calcium carbonate, and hydrazine hydrate. 3-Aminocarbazole and 4-aminocarbazole-6-sulphonic acid are similarly prepared from their 2:8-dichloro- and -dibromo-derivatives, respectively. C. HOLLINS.

**Manufacture of 1:2:4[1:3:4]-triazoles.** A. BOEHRINGER, G. SCHEUING, and B. WALACH (B.P. 340,237, 16.9.29).—Acid hydrazides react with imino-esters or imino-chlorides to give 1:3:4-triazoles; preferably the imino-compound is produced *in situ*, e.g., from oximes, *O*-acylated oximes, or *O*-acylated acid amides carrying an *N*-substituent. Acetmethylamide is treated with phosphoryl chloride in pyridine and chloroform at 0° and the resulting lactim ester is stirred into benzhydrazide in chloroform to give 5-phenyl-1:2-dimethyl-1:3:4-triazole, m.p. 137°, b.p. 251°/14 mm. 1-Phenyl-2-methyl-5-ethyl-1:3:4-triazole, m.p. 152–153°, b.p. 219°/15 mm., is obtained from propionhydrazide and acetophenoneoxime *O*-benzenesulphonate or the oxime treated with thionyl chloride, or acetanilide treated with phosphorus pentachloride (the intermediate hydrazidine, EtCO·NH·NH·CMe:NPh, has m.p. 152–153°). The following 1:3:4-triazoles are also described: 1-phenyl-2-methyl-, b.p. 187°/5 mm.; 1-cyclohexyl-2-methyl-5-isobutyl-, b.p. 163°/0.2 mm. (hydrate, m.p. 67°; intermediate hydrazidine, m.p. 67°); 1-(*m*-4-xylyl)-2-methyl-5-isopropyl-, m.p. 80°, b.p. 182°/? mm. (hydrazidine, m.p. 196°); 1:2-diphenyl-5-methyl-, m.p. 163°; 1-cyclohexyl-2-ethyl-, m.p. 89°, b.p. 227°/11 mm. (picrate, m.p. 159°); 1-cyclohexyl-2-methyl-, m.p. 102°, b.p. 200°/1 mm.; 1-(3-methylcyclohexyl)-2-methyl-, b.p. 212°/1 mm.; 1-cyclohexyl-2:5-dimethyl-, m.p. 132°; 1-(4-methylcyclohexyl)-2-methyl-5-ethyl-, m.p. 75°, b.p. 232°/16 mm. (hydrate, m.p. 46°); 1-(2-methylcyclohexyl)-2-methyl-5-ethyl-, b.p. 185°/0.9 mm. (picrate, m.p. 145°); 1:2-pentamethylene-5-methyl-, m.p. 111–112°, b.p. 210°/10 mm. (hydrate, m.p. 62°); 1:2-( $\alpha$ -methylpentamethylene)-5-diethylmethyl-, b.p. 170°/0.5 mm. (hydrazidine, m.p. 205°); 1:2-pentamethylene-5-isobutyl-, m.p. 50°, b.p. 183°/0.4 mm. C. HOLLINS.

**Manufacture of preparations for production of dyes [of the Rapid Fast series].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 340,534, 1.10.29).—A nitrosoamine alkali salt (particularly from a chloroaniline) is mixed with a 2:3-hydroxynaphthoic arylamide free from carboxyl and sulphonc groups, and with a normal salt capable of binding water (anhydrous

sodium acetate). Examples are the following (the bases being in the form of nitrosoamine alkali salts): 2:5-dichloroaniline and *o*-phenetidine, *o*-anisidine, or anilide; 4-chloro-*o*-anisidine and *o*-anisidine; dianisidine and anilide; 3-nitro-*p*-toluidine and anilide. The arylamides may be used in the form of sodium salts. The use of sodium acetate enables the ingredients to be ground together to a dry powder. C. HOLLINS.

**Manufacture of anthraquinone and homologues thereof.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,509, 19.8.29. Addn. to B.P. 320,375; B., 1930, 233).—The products of the prior patent, or tetrahydro-anthraquinones or -anthrahydroquinones obtainable therefrom, are oxidised to anthraquinones by ferric or cupric salts (etc.) or by air or oxygen in presence of smaller amounts of such salts. The preparation of anthraquinone and 2:7-dimethylantraquinone is described. C. HOLLINS.

**Anthraquinone derivatives [manufacture of halogenated and hydroxylated anthraquinones].** P. F. BANGHAM, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 339,589, 4.6.29).—Halogenated phthalic anhydrides are condensed with halogenated phenols in presence of sulphuric acid (oleum) and boric acid to give halogenated anthraquinones, in which one or more  $\alpha$ -halogen atoms may be hydrolysed to hydroxyl. 4-Chlorophthalic anhydride and *p*-chlorophenol give 6-chloroquinizarin. 5:6-Dichloro-, 6:7-dichloro-, and 2:6:7-trichloro-quinizarins are similarly prepared. C. HOLLINS.

**Manufacture of  $\alpha$ -hydroxyanthrones.** I. G. FARBENIND. A.-G. (B.P. 340,639, 10.10.29. Ger., 17.10.28).—Acylated  $\alpha$ -hydroxyanthraquinones are reduced in neutral or weakly acid suspension at 50–100° with sodium hyposulphite, or zinc dust, or hydrogen in presence of nickel. Dilute acetic acid, alcohol, decahydronaphthalene, and water are suitable solvents. 1-Acetoxyanthraquinone gives 80–90% of a hydroxyanthrone, m.p. 238–240°; dihydroxyanthrone monoacetates are obtained from diacetates of chrysazin (m.p. 247–248°), quinizarin (m.p. 208°), 5:8-dichloroquinizarin (m.p. 220°), anthrarufin (m.p. 207°), and alizarin (m.p. 188–189°). Hydrolysis of the quinizarin product gives 1:4-dihydroxyanthrone, m.p. 220°. C. HOLLINS.

**Preparation of 2-amino-3-substituted-10-anthrones and *N*-substitution [acylated] products thereof.** NEWPORT Co. (B.P. 340,519, 28.6.29. U.S., 2.7.28. Cf. B.P. 314,804; B., 1931, 290).—Suitable *o*-benzylbenzoic acids are cyclised. 4-Chloro-3-aminodiphenylmethane-2'-carboxylic acid with weak oleum below 30° gives 3-chloro-2-amino-10-anthrone or, with acetic acid and phosphorus pentoxide, the corresponding acetyl compound, m.p. 253–255° (decomp.). 2-Amino-3-ethoxy-10-anthrone, m.p. 200–201°, and 2-amino-3-methoxy-10-anthrone, m.p. 190–192°, are similarly prepared. C. HOLLINS.

**Treatment of organic liquids to facilitate storage and handling.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,360 and 340,575, 4.7.29).—(A) The organic liquid, e.g., petrol, light petroleum, etc., is absorbed in a voluminous framework-like solid mass

produced in the liquid by precipitation of inorganic oxides or hydroxides, *e.g.*, by interaction of silicon tetrachloride or tin tetrachloride and ammonia gas. (B) The reaction between abietic or pimaric acid and a basic substance (ammonia) is effected so as to produce such a mass within the liquid (*e.g.*, cyclohexane, benzene). The liquid may be recovered from the solid mass by pressing. C. HOLLINS.

**Refrigerant.**—See I. Naphthalene from gas. Gases containing acetylene. Benzene-water. Hydrocarbons. Sulphonic acids.—See II. Vat-dye intermediates.—See IV. Formic acid.—See V. Fatty acid derivatives.—See XII. Accelerators for rubber.—See XIV. Absolute alcohol. Sulphite-liquor spirit.—See XVIII. Sterilising organic substances.—See XX.

#### IV.—DYESTUFFS.

**Anthraquinone vat colours containing the  $\cdot\text{CO}\cdot\text{NH}\cdot$  linking.** R. N. LULEK (Ind. Eng. Chem., 1931, 23, 96—98; cf. B., 1929, 709).—8-*Keto*-1:9-*anthraiso*thiazole-2-, -4-, and -5-*carboxylic acids* are prepared by heating 1-chloroanthraquinone-2-, -4-, and -5-*carboxylic acids* with ammonia and alkali polysulphide. Their chlorides yield dyes when condensed with aminoanthraquinones, but only in the case of the first are these dyes of value. Apparently new dyes from the 2-carbonyl chloride and 4-chloro-1-amino-, 5-chloro-1-amino-, and monobenzoyl-1:4-diamino-anthraquinones are described (cf. B.P. 326,487; B., 1930, 501); the diaminoanthraquinones give inferior products. The corresponding 8-*keto*-1:9-*anthraiso*selenazole-2-*carboxylic acid* is similarly prepared; its chloride gives products of a deeper shade with aminoanthraquinones, *e.g.*, 6-chloro-1-aminoanthraquinone. Condensation of 1-thiolanthraquinone-2-*carboxylic acid* with chloroacetic acid and alkali gives 8-*keto*-1:9-*anthra*thiophen-2-*carboxylic acid*, the chloride of which gives products of poor tinctorial power with the aminoanthraquinones.

H. A. PIGGOTT.

See A., Mar., 343, Azo dyes. 344,  $\alpha$ -Naphthol-orange. 347, Brominated sulphonic acids. 350, Triphenylmethane dyes. 355, Reduction of anthraquinones.

#### PATENTS.

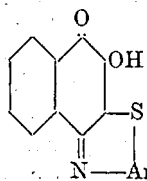
**Manufacture of [yellow] dyes of the tartrazine series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,009, 19.8. and 12.12.29).—Dihydroxytartaric acid or an oxalacetic ester is condensed with 1 or 2 mols., respectively, of phenylhydrazine-3:5- or -2:5-disulphonic acid or of *o*-tolylhydrazine-4:5-disulphonic acid; when oxalacetic esters are used the resulting pyrazolone is coupled with the corresponding diazobenzene-3:5- or -2:5-disulphonic or diazo-*o*-toluene-4:5-disulphonic acid. The tartrazines so formed give greenish-yellow dyeings and are suitable for wool or for lakes for paper.

C. HOLLINS.

**Manufacture of colour lakes and dye compositions suitable for use in [intaglio] printing.** I. G. FARBENIND. A.-G. (B.P. 319,370, 20.9.29. Ger., 22.9.28).—The alkaline-earth (calcium, barium) salts of dyes of the type: halogenated arylamine (non-sulphonated)

$\rightarrow$  2:6- or 2:7-naphtholsulphonic acid, are orange to bluish-red lakes stable to oil and water and therefore suitable for intaglio printing. Examples of first components are *o*-, *m*-, *p*-chloroanilines, 2:4-, 2:5-, 3:4-, 3:5-dichloroanilines, 4-, 5-, 6-chloro-*o*-toluidines, 4:6-dichloro-*o*-toluidine, 2-chloro-*p*-toluidine, 4-chloro-*o*-anisidine, 5-chloro-*p*-xylylidine, and 1-chloro- $\beta$ -naphthylamine. C. HOLLINS.

**Manufacture of mordant dyes.** I. G. FARBENIND. A.-G. (B.P. 339,410, 10.10.29. Ger., 10.10.28).—2-Hydroxy-1:4-naphthaquinone 4-sulphoarylimines are treated with sulphur in sulphuric acid to give acid dyes (annexed formula) which are green when after-chromed. Dyes from the *m*-sulpho-*p*-tolylimine, *m*-sulpho-anil, 6-sulpho-*m*-4-xylylimine, and *o*-sulpho-*p*-tolylimine, are described.



C. HOLLINS.

**Manufacture of [pigments and acid] dyes [from 3:6-dihalogeno-2:5-diarylamino-*p*-benzoquinones].** I. G. FARBENIND. A.-G. (B.P. 313,094, 6.6.29. Ger., 6.6.28).—3:6-Dihalogenobenzoquinones, carrying in 2- and 5-positions groupings  $\cdot\text{NH}\cdot\text{Ar}\cdot\text{NR}\cdot\text{Ar}'$  (where Ar and Ar' are aromatic residues, and R is hydrogen or alkyl, or where  $\text{Ar}\cdot\text{NR}\cdot\text{Ar}'$  represents carbazolyl or alkylcarbazolyl), are heated, preferably in a solvent (nitrobenzene) with a metal chloride or phosphorus pentachloride and/or with an oxidant (pyrolusite), to give blue to blue-grey or blue-green pigments, which on sulphonation yield acid dyes; the latter form pigments with the bases of B.P. 277,371. Products from 3:6-dichloro-2:5-di-(*N*-ethyl-3-carbazolylamino)- and -2:5-di-(*p*-anilinophenylamino)-1:4-benzoquinones are described. C. HOLLINS.

**Manufacture of [acid] dyes of the diphenyl-naphthylmethane series.** I. G. FARBENIND. A.-G. (B.P. 339,823, 3.1.30. Ger., 4.1.29).— $\alpha$ -Naphthaldehyde-2-sulphonic acids are condensed with an alkylated or aralkylated arylamine and the product is oxidised, with or without sulphonation.  $\alpha$ -Naphthaldehyde-2:4-disulphonic acid gives with diethylaniline a bluish-green, with benzylethylaniline a yellowish-green wool dye, without further sulphonation. C. HOLLINS.

**Manufacture of halogenated 4:5:8:9[1:2:7:8]-dibenzpyrene-3:10[3:6]-quinones.** I. G. FARBENIND. A.-G. (B.P. 339,489, 6.12.29. Ger., 6.12.28).—Chlorinated (or brominated) 1:2:7:8-dibenzpyrene-3:6-quinones are brominated (or chlorinated), preferably in chlorosulphonic acid solution. The 5':4''-dichloro-compound (from 1:4-di-*p*-chlorobenzoylnaphthalene) gives on bromination a scarlet vat dye, the 5'-chloro-compound a bluer shade. Chlorination of the 5':4''-dibromo-compound yields an orange vat dye. C. HOLLINS.

**Manufacture of [vat] dyes of the anthraquinone series.** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 339,516, 24.12.29).—The blue-green vat dyes obtained by sulphurisation of 4-methylbenzanthrone are purified by precipitation from sulphuric acid by dilution, or by allowing the leuco-compound to separate from the vat, filtering, and reoxidising.

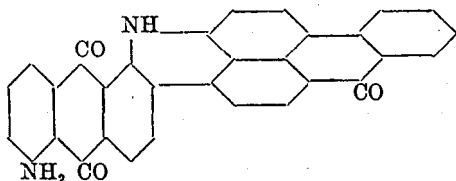
C. HOLLINS.



**Manufacture of vat dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,267, 3.9., 1.10., and 6.11.29).—An  $\alpha$ -aminoanthraquinone (2 mols.) is heated, preferably in a high-boiling solvent (nitrobenzene), with the acid chloride (etc.) of a naphthalenedicarboxylic acid, a diphenylmethanedicarboxylic acid, diphenyl-4:4'-dicarboxylic acid, or halogenated derivatives of these, excepting dicarboxylic acids which give inner anhydrides. The  $\alpha$ -anthraquinonylamides of naphthalene-2:6-, -2:7-, and -1:7-, diphenyl-4:4', 3:3'-dichlorodiphenyl-4:4', and diphenylmethane-4:4'-dicarboxylic acids are greenish-yellow vat dyes. Naphthalene-2:7-dicarboxylic 4-methoxy-1-anthraquinonylamide (orange) and 4-benzamido-1-anthraquinonylamide (red) are also described.

C. HOLLINS.

**Manufacture of vat dyes [of the anthraquinone series].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,396, 28.9.29).—A 1-amino-4 (-5 or -8)-3'-benzanthrnylaminoanthraquinone, which may be halogenated in the benzanthrone nucleus, is heated with an alkaline condensing agent (e.g., alcoholic potassium hydroxide; cf. B.P. 24,604 of 1908; B., 1909, 832) and the product (annexed formula) is acylated; or, prefer-



ably, the corresponding 1-acylamino-compound is heated with a non-hydrolysing alkaline condensing agent (e.g., sodium aniline). In the examples the acyl group is benzoyl (yellow), anthraquinone-2-carboxyl (yellow-olive), acetyl (olive) with 1:5- or 1:8-compounds, acetyl (olive) with the 1:4-compound. [Stat. ref.]

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone [oxazole] series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,633, 8.10.29. Addn. to B.P. 198,077; B., 1923, 647 A).—3:3'-Dihydroxybenzidine (or other di-*o*-dihydroxydiamine of the diaryl series) is condensed with 2-aldehydoanthraquinone,  $\omega$ -di- or -tri-chloro-2-methylantraquinone, or anthraquinone-2-carboxylic chloride to give a brown-olive vat dye of good tinctorial power.

C. HOLLINS.

**Manufacture of acid anthraquinone dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,266, 3.9.29).—4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with an aminoaryl ether of a polyhydric alcohol, e.g., with *o*- or *p*-aminophenyl  $\beta$ -hydroxyethyl or  $\beta$ -dihydroxy-*n*-propyl ether, to give blue acid dyes for wool.

C. HOLLINS.

**Manufacture of vat dyes of the 1:2-benzanthraquinone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,524, 29.7. and 25.10.29).—Complex carbazoles or acridines may be obtained by treating a 4- $\alpha$ -anthraquinonylamino-[1:2]-benzantraquinone, having a free 3- or 5-position, with acid or alkaline condensing agents; or by treating a 5- $\alpha$ -anthraquinonylamino-[1:2]-benzantraquinone, having a free 4-posi-

tion and containing at least one acylamino-group, with acid condensing agents, followed, if desired, by hydrolysis, and further substitution or elimination of the amino-group.

4- $\alpha$ -Anthraquinonylamino-[1:2]-benzantraquinone is converted by sulphuric acid at 130–135° or by sodium aluminium chloride at 150° or zinc chloride at 200° into a yellow vat dye, or by potassium pyrosulphate at 360° a brown olive. On the other hand, a blue-green vat dye is obtained with aluminium chloride in pyridine at 220–230° or with potassium hydroxide and *m*-cresol at 230–240°; a green vat dye of the same type is prepared by the action of chlorosulphonic acid at 15° on 4-(4'- or 5'-benzamido- $\alpha$ -anthraquinonylamino)-[1:2]-benzantraquinone. Other examples are: 4-(5'-amino- $\alpha$ -anthraquinonylamino)-[1:2]-benzantraquinone with sulphuric acid at 130–135° (intense red) or with potassium hydroxide and *m*-cresol at 210–220° (bluish-grey). 5-(4'-Benzamido- $\alpha$ -anthraquinonylamino)-[1:2]-benzantraquinone is converted by aluminium chloride in pyridine at 170° into a blue vat dye. The 5'-benzamido-compound gives, when warmed with chlorosulphonic acid at 50–55°, a similar product which may be hydrolysed with sulphuric acid to a reddish-blue vat dye becoming blue-green on elimination of the amino-group. Probable constitutions for the dyes are suggested.

C. HOLLINS.

**Dyes [from leuco-esters of the dibenzanthrone series].** R. S. BARNES, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 339,324, 31.5.29).—Oxidation in substance of leuco-esters of dibenzanthrone or its halogen derivatives under more drastic conditions than are employed for development on the fibre leads to vat dyes of deeper shade. Examples of oxidants are: sodium dichromate in 10% sulphuric or hydrochloric acid at 15–100°; potassium persulphate or permanganate, vanadium pentoxide or hydrogen peroxide in 20% sulphuric acid.

C. HOLLINS.

**Production and use of vat dyes [of the dibenzanthrone series].** C. SHAW, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 339,954, 11.6.29).—3:3'-Dibenzanthronyl or a derivative is oxidised with chromic and sulphuric acids and the product is fused with alkali. Blue-black vat dyes are thus obtained from 3:3'-dibenzanthronyl and its tetrachloro- and tetrabromo-compounds.

C. HOLLINS.

**Manufacture of dibenzanthrones and isodibenzanthrones containing chlorine and bromine.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,262, 21.6.29).—Chlorine and bromine are introduced into bromo- and chloro-derivatives, respectively, of dibenzanthrones and isodibenzanthrones, or both simultaneously into the non-halogenated compounds. 2:2'-Di-( $\beta$ -chloroethoxy)dibenzanthrone is brominated in chlorosulphonic acid at 60° to give a green-blue vat dye. Various nuclear chlorobromo-dibenzanthrones and isodibenzanthrones, giving blue-violet, navy-blue, and blue shades, are also described. (Cf. B.P. 325,525; B., 1930, 453.)

C. HOLLINS.

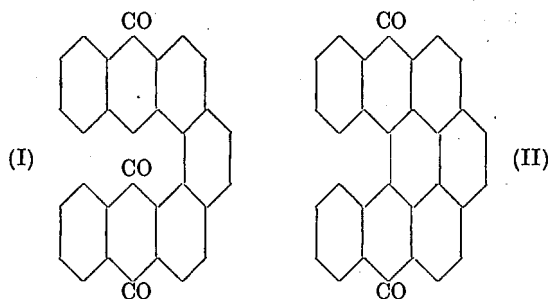
**Manufacture of fast vat dyes [pyrazolanthrone-carboxylic anthraquinonylamides].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,334, 1.10.29).—Pyrazolanthrone-3-carboxylic chlorides are condensed



with an aminoanthraquinone or other vatable aminodiketone; or 1-halogenoanthraquinone-2-carboxylic chloride is similarly condensed and then treated with hydrazine. The products may be alkylated or aralkylated. Vat dyes from pyrazolanthrone-3-carboxylic chloride and  $\alpha$ -aminoanthraquinone (yellow), 1:4- or 1:5-diaminoanthraquinone (0.5 mol., yellow), 1-amino-methoxyanthraquinone (orange), and 1-amino-4-benzamidoanthraquinone (orange-brown), and from *N*-methylpyrazolanthrone-3-carboxylic chloride and  $\alpha$ -aminoanthraquinone (yellow) and 1-amino-4-benzamidoanthraquinone (orange-brown), are described.

C. HOLLINS.

**Manufacture of vat dyes [dianthrones etc.].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,626, 8.8.29).— $\omega\omega'$ -Tetrabrominated 2:2'-dimethyldianthraquinonyls or the corresponding *ms*-benzo- or -naphtha-dianthrones are treated with alkaline or acid condensing agents having a reducing action, hydrolysis of the dibromomethyl groups before condensation being avoided in the case of the dianthraquinonyls. The products may subsequently be halogenated.  $\omega\omega'$ -Tetrabromo-2:2'-dimethyl-1:1'-dianthraquinonyl when warmed at 70–80° with pyridine, water, sodium hydroxide, and sodium hyposulphite gives 1:2:9:10-diphthaloylphenanthrene (I), m.p. 345° (decomp.), a yellow-orange vat dye; or with copper and sulphuric acid at 40–50°, *ms*-*allo*-naphthadanthrone (II), m.p. 350°, an orange vat dye, convertible into *ms*-anthradianthrone or its halogen derivatives. Tetrabrominated



2:2'-dimethyl-*ms*-benzdianthrone, containing part of the bromine in the nucleus, is converted by boiling pyridine into a bromo-*ms*-*allo*-naphthadanthrone, a red vat dye. 4:4'-Dichloro-*ms*-*allo*-naphthadanthrone, from dichlorotetrabromo-2:2'-dimethyl-1:1'-dianthraquinonyl with copper and sulphuric acid, is an orange vat dye. Alcoholic potassium hydroxide converts  $\omega\omega'$ -tetrachloro-2:2'-dimethyl-*ms*-naphthadanthrone into *ms*-anthradianthrone, a yellow vat dye.

C. HOLLINS.

**Manufacture of [vat] dyes and intermediate products [of the dianthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,659, 8.7.29).—*ms*- or *allo*-*ms*-Naphthadanthrones or *ms*-anthradianthrones are nitrated and the nitro-groups are wholly or in part reduced or replaced directly by halogen. *allo*-*ms*-Naphthadanthrone is mononitrated and gives a chloro-compound (orange-red vat dye) on treatment with benzoyl chloride; di- and tetra-nitro-compounds are described. *ms*-Naphtha- and *ms*-anthra-dianthrones are also mononitrated.

C. HOLLINS.

**Manufacture of azo dyes [pigments and ice colours].** I. G. FARBENIND. A.-G. (B.P. 319,247, 18.9.29. Ger., 18.9.28).—2:3-Hydroxynaphthoic  $\alpha$ - or  $\beta$ -naphthylamide is coupled in substance or on the fibre with a diazotised alkyl 5-halogenoanthranilate, e.g., methyl 5-chloroanthranilate  $\rightarrow$   $\beta$ -naphthylamide (scarlet-red). [Stat. ref.]

C. HOLLINS.

**Manufacture of azo dyes [pigments and ice colours].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 339,620, 10.7.29. Addn. to B.P. 336,938; B., 1931, 151).—A 2:3-hydroxynaphthoic 5-alkoxy-*o*-anisidide or 6-alkoxy-*m*-anisidide (i.e., 2- or 3-methoxy-4-alkoxyanilide) is coupled in substance or on the fibre with non-sulphonated, non-carboxylated diazo, tetrazo, or diazo-azo compounds; e.g., 2:5-dichloroaniline  $\rightarrow$  3:4-dimethoxyanilide (brown-red); 4-chloro-*o*-toluidine  $\rightarrow$  2:4-dimethoxyanilide (red).

C. HOLLINS.

**Manufacture of azo dyes containing chromium.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,243, 19.9.29).—A 3-methylpyrazolone obtained from an aminosalicic acid is coupled with a diazotised arylamine having no hydroxyl or carboxyl group *ortho* to the diazo group, and the product is prechromed. Examples are: *m*-aminobenzoic acid  $\rightarrow$  1-(2-hydroxy-3-carboxy-5-sulphophenyl)-3-methylpyrazolone (greenish-yellow on wool); 4-chloro-*o*-toluidine  $\rightarrow$  1-(2-hydroxy-3-carboxyphenyl)-3-methylpyrazolone (yellow).

C. HOLLINS.

**Products from cyclohexylamines.**—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Mercaptolysis of pine wood.** B. HOLMBERG (Ing. Vetensk. Akad. Handl., 1930, No. 103, 5–75; Chem. Zentr., 1930, ii, 2770–2772; cf. B., 1928, 705).—The formula  $C_{36}H_{90}O_8(OMe)_4(OEt)_2$  for lignin acetal is preferred to Hägglund and Urban's formula. Of the aldehydes examined, only cinnamaldehyde and furfuraldehyde gave homogeneous products when condensed with thioglycollic acid in alcoholic hydrogen chloride, the furan ring being ruptured. Aldoses condense, but the very soluble products are not readily isolated. Cellulose is unattacked. Lignin gives a mixture of ligno-dithioglycollic and tetrathioglycollic acids (obtained by hydrolysis of the ethyl esters), from which by heating with mercuric chloride solution about half of the thioglycollic acid is removed with simultaneous reduction and formation of vanillin. Prolonged treatment with hydrochloric acid affords lignopentathioglycollic acid in small yield. The reactivity of lignin is diminished by treatment with acids. Formaldehydemercaptalacetic acid [methylenedithiolacetic acid; Holmberg and Mattison, A., 1907, i, 475], m.p. 120–125°, may be prepared from trioxymethylene and thioglycollic acid; the ethyl ester has b.p. 188–190°/14 mm. The following dithiolacetic acids are described: ethylidene-, m.p. 109–110° (ethyl ester, b.p. 156–158°/2–2.5 mm.); benzylidene-, m.p. 126–127° (ethyl ester,  $d_4^{20}$  1.180); vanillylidene-, m.p. 136–137°; piperonylidene-, m.p. 138–139°; furfurylidene-, m.p. 107–109°. Fission of the thiol group by ionised mercury takes place according to the equation  $CHR(R'S)_2 + 2 HgCl_2 + H_2O = 2 R'S \cdot HgCl + R \cdot CHO + 2 HCl$ , the reaction being followed by titration of the acid formed. In experiments with pine wood, 10 g. of

sawdust, previously extracted with acetone, were heated under reflux for 4 hrs. with 100 c.c. of *N*-alcoholic hydrogen chloride; only 10% of the lignin remained undissolved. The mixture contained 75% of lignodi- and 25% of lignotetra-thioglycollic esters. Energetic treatment with alkali and acid gives more of the di-acid. In concentrated aqueous hydrochloric acid thioglycollic acid at the ordinary temperature converts the whole of the lignin into alkali-soluble products.

A. A. ELDRIDGE.

**Effect of beating on certain chemical and physical properties of pulps.** C. E. CURRAN, F. A. SIMMONDS, and H. M. CHANG (Ind. Eng. Chem., 1931, 23, 104—108).—Chemical examination of beaten and unbeaten sulphite and sulphate pulps confirms that there is no chemical change in the pulps during 4 hrs.' beating. A slight oxidation of sulphate pulp during beating was noted, and is attributed to alkali adsorbed during cooking. X-Ray spectrographs confirm the identity of beaten and unbeaten pulps. A method for determining oxycellulose is described in which the volume of carbon dioxide evolved on boiling the pulp with 12% hydrochloric acid is measured.

T. T. POTTS.

**Manufacture of insulating board from corn-stalks.** O. R. SWEENEY and W. E. EMLEY (U.S. Bur. Stand. Misc. Publ., 1930, No. 112, 27 pp.).—The shredded stalks, in some cases after digesting with water either with or without the addition of chemicals, *e.g.*, lime, soda, sodium sulphide, were pulped by means of a Holland beater, a swing-hammer mill, or a rod mill (the latter is indicated). The pulp was run through a Jordan refiner and washed to remove dirt. The effect of different sizing materials was studied: rosin size and aluminium sulphate added in proportions to give  $\eta_H$  4.5 gave the best results, and there was no advantage in using more than 2% of rosin size on the dry weight of the fibre. The pulp, which should have a consistency of 1.5—2.5%, was fed on to a modified Oliver filter and the mat pressed in three sets of heavy rolls in series operated at a pressure sufficient to reduce the moisture content to 70%; too great a pressure, although increasing the strength of the board, reduces its insulating properties. The dryer used was of the roller type. The boards have properties similar to those of commercial boards, but they will not withstand frequent or continuous immersion in water. The economics of the process are given.

D. K. MOORE.

**Swelling of cellophane and rayon cloth by caustic soda solution. Cellophane and cellulose acetate as dialysis membranes. Distribution of impurities in recovery of waste soda from the viscose factory by dialysis.** K. TANEMURA and S. MIYOSHI (J. Soc. Chem. Ind., Japan, 1930, 33, 431—434 B).—When immersed in 6.12% caustic soda solution a circular form of cellophane becomes elliptical. The maximum swelling in caustic soda at 20° takes place with a 10% solution, but in the case of rayon cloth the web is disintegrated by an 8—13% solution; swelling is diminished by ethyl alcohol, dextrose, and other solutes, and, in the case of sodium chloride, carbonate, and sulphate, rapidly reaches a minimum which is uninfluenced by further addition of the salt.

The relative depressing power of cations of alkali metals and anions on the swelling of these cellulosic materials by caustic soda solution is indicated. The rates of diffusion of solutions of crystalloids through cellophane and cellulose acetate films are proportional to the diffusion coefficients of the solute, except where the latter has a specific action on the membrane, making its passage easier; *e.g.*, caustic soda and acetic acid show increased rates of diffusion through cellophane and cellulose acetate, respectively, owing in the one case to the swelling action of soda on cellophane and in the other to the solvent power of acetic acid on cellulose acetate. Experiments on the diffusion of 0.1*N*-lithium chloride in mixed solvents of water and ethyl or methyl alcohol through cellophane and cellulose acetate membranes, and of *N*-sodium hydroxide in water and ethyl alcohol through cellophane, show that swelling of the dialysis membrane by the solvent helps the diffusion of the crystalloid, and also that the presence of a solvent which depresses the swelling retards diffusion. By proper manipulation waste soda from the viscose factory may be purified by analysis through a cellulosic membrane, the hemicellulose, a greater part of the sodium carbonate, and a part of the sodium sulphate and chloride remaining undiffused during the time that the sodium hydroxide passes through the membrane.

F. R. ENNOS.

**Permeability of packing materials to water and water vapour.** (MISS) R. COHEN (Chem. Weekblad, 1931, 28, 159—162).—The permeabilities of such packing materials as cellophane, duratex, and triacel, as compared with that of parchment, have been determined. The data obtained are so irregular that general conclusions only are drawn. The permeability to moisture is inversely related to the electrical insulating power, both being connected with the presence or absence of free hydroxyl groups.

S. I. LEVY.

**Acetylcellulose.** F. OHL (Farbe u. Lack, 1931, 51, 64).—An account is given of the preparation of commercial acetylcellulose, and of the various factors influencing its properties. Methods of chemical examination and tests for determining quality are described.

F. R. ENNOS.

**Cellulose nitroacetate.** D. KRÜGER (Cellulosechem., 1930, 11, 220—228).—The mixed ester may be prepared by treating at low temperatures cellulose with a mixture of nitric, sulphuric, and acetic acids together with acetic anhydride. When the mixture consists of sulphuric and nitric acids (1 : 1) and acetic acid and anhydride (1 : 1), no acetyl groups are introduced until the ratio mineral acid:acetylating agents falls below 2.7, nitrocellulose being the sole product. When the reaction mixture contains but small concentrations of acetylating agent, the nitrogen content of the product is high (12.75%), but on increasing this concentration to the critical ratio, 2.7, the nitrogen content falls to 10.8%. The solubilities of nitrocelluloses prepared in this way, in ether-alcohol, are identical with those of nitrocelluloses of the same nitrogen content prepared in the usual way with aqueous nitrating mixtures. It is, therefore, suggested that the solubility of nitrocellulose is conditioned only by the nitrogen content, and that the degree of hydro-

lytic and oxidative degradation has little effect, except in modifying the speed of dissolution and the viscosity of the resulting solutions. Decreasing the ratio of the reactants from 2.7 gives products of decreasing nitrogen, and increasing acetyl content. Mixtures poor in mineral acids lead to a product approximating to a triacetate. At an intermediate range of compositions (ratio about 0.4), the hydrolytic and oxidising action of the bath is considerable. With the introduction of acetyl groups the product tends to become soluble in the reaction medium. The solubility in ether-alcohol and the inflammability of the product decrease with increasing acetyl content. It was, however, found impossible to produce, by this process, a nitroacetate which was soluble in ether-alcohol, capable of being plasticised by camphor, comparatively non-inflammable, and which retained the original fibre form in the reaction bath. The reaction mixture used by Nishida (B., 1914, 476), and reported as affording a nitroacetate, can only yield a nitrocellulose. The discrepancy is probably due to an incorrect method of analysis. T. H. MORTON.

**Determination of pentosans.** C. KULLGREN and H. TYDÉN (Ingeniörs Vetenskaps Akad. Handl., 1929, No. 94, 3—62; Chem. Zentr., 1930, ii, 3177).—When furfuraldehyde is distilled with 13.5% hydrochloric acid and sodium chloride the distillation factor is constant the loss of furfuraldehyde on distillation, *e.g.*, from pentosans, is about 3%, depending on the duration of heating. Determination of the furfuraldehyde by means of bromine is criticised. Interaction with bromate occurs even in slightly acid solution, oxidation occurring first, and then, more slowly, bromine substitution unless the acid content is between 0.5 and 1%. The former reaction can be catalysed by ammonium molybdate. The method (details given) is less sensitive than are the older methods towards changes of temperature and bromate content. The hydroxymethylfurfuraldehyde produced on distillation of polyoses with hydrochloric acid affects the result; redistillation and correction for the furfuraldehyde decomposed are recommended. Wood is preferably distilled for 100 min. with hydrochloric acid; the formaldehyde formed from the lignin does not interfere. Formic acid, acetic acid, and acetaldehyde have only a slight effect. In the distillation of sulphite cellulose a correction for sulphur dioxide, determined by titration with iodine, must be applied. A. A. ELDRIDGE.

**Modern developments in the manufacture of viscose and viscose silk.** H. SCHMIDT (Chem. Fabr., 1931, 73—77, 85—90, 97—100).—A review.

**Nitrocellulose.**—See XXII.

See also A., Mar., 311, Cuprammonium hydroxide-cellulose. 338, Lignosulphonic acids.

#### PATENTS.

**Manufacture of textile products.** BRIT. CELANESE, LTD. (B.P. 339,896, 5.3.30. U.S., 5.3.29).—Natural silk, which is to be woven with cellulose ester or ether silk, is degummed in the yarn and then sized with sizing agents which are readily removed by hot water or other reagent which does not deleteriously affect cellulose ester or ether silk. Suitable sizes may contain gelatin, starch, waxes, soaps, drying oils, etc. D. J. NORMAN.

**Purification of chemical wood pulp.** J. D. RUE ASSR. to CHAMPION FIBRE CO. (U.S.P. 1,771,064, 22.7.30. Appl., 20.8.28).—The raw pulp is partly bleached, *i.e.*, treated with 75—85% of its total bleach requirements, at a consistency of 8—12%, washed, and agitated for 10 min.—6 hrs. (preferably 0.5—1 hr.) at 21—100° (preferably 52°) with sufficient caustic soda to give a solution of 5—12% concentration and a pulp consistency of 5—10%. The washed pulp is finally bleached with about 4% of bleaching powder at a pulp consistency of 3—4%. Washing in each case is preferably carried out in the presence of sufficient acid or acid-reducing agent to bring the  $pH$  of the liquor to 2—4. The final product contains 88—94% of  $\alpha$ -cellulose. D. J. NORMAN.

**Drying of pulp.** R. D. KEHOE (U.S.P. 1,770,409, 15.7.30. Appl., 2.6.28).—The pulp suspension is centrifuged and the resulting moist fluffy lumps are fed on to a conveyor and dried in a countercurrent of hot air. At intervals along the drying chamber the air is withdrawn from below the pulp, passed over heating units, and reintroduced above the pulp. The heating units are so adjusted that the temperature along the dryer gradually rises to a maximum and then slowly decreases toward the exit end. D. J. NORMAN.

**Manufacture of sulphite wood pulp.** W. E. B. BAKER (U.S.P. 1,773,419, 19.8.30. Appl., 3.6.26).—Sulphite wood pulp of abnormally high lignin content is obtained by pretreating the chips with very dilute caustic alkali solution at, *e.g.*, 100° for 1—2 hrs. in the presence of ammonia gas to increase the pressure. This treatment renders the  $\alpha$ -lignin insoluble in the sulphite cooking liquor and, at the same time, shortens the cooking time by opening up the chips and removing resins. High yields are obtained of a strong pulp suitable for bag and wrapping paper. D. J. NORMAN.

**Digestion of fibrous material.** S. D. WELLS (U.S.P. 1,771,598, 29.7.30. Appl., 11.7.25).—A closed digester containing the fibrous material is completely filled with a non-acid digesting liquor, and the contents are allowed to remain under pressure until the material is thoroughly impregnated. A portion of the liquid is then removed from below while steam is introduced to take its place, air being thereby excluded; the digestion is afterwards carried out in the usual manner. F. R. ENNOS.

**By-products recovery in the digestion of cellulose materials.** G. HAGLUND (U.S.P. 1,772,251, 5.8.30. Appl., 3.5.28. Swed., 13.5.27).—Alkali sulphate is electrolysed in a cell in which the electrodes are enclosed by porous walls, the cathodic solution of alkali hydroxide being drawn off and treated with sulphur compounds to form a suitable lye in which the cellulose raw material is boiled. The spent lye is separated from the cellulose, and treated with the anodic solution of sulphuric acid, together with more sulphuric acid in excess, to precipitate the organic matter which, after collection of the volatile products formed, is separated from the solution; the latter is then crystallised to yield alkali sulphate, which is dissolved and again electrolysed, whilst the mother-liquor is returned for the concentration of other boiling lye. F. R. ENNOS.

**Manufacture of artificial silk.** W. II. FURNESS, Assr. to CELLOCILK Co. (U.S.P. 1,770,750, 15.7.30. Appl., 6.4.27).—A concentrated solution of copper hydroxide, ammonia, and cellulose in approximately minimum dissolving proportions is diluted with water so as to contain about 4% of cellulose and spun directly into a precipitating bath with production of filaments which are very small in relation to the spinneret orifices and are completely solidified almost immediately. The formed thread is then stretched to a point where, in the finished state, it has the same elongation wet as dry, a marked resistance to elongation over a wide range of load, approximately the same elongation for all degrees of relative humidity normally obtainable, and no appreciable yield points in its elongation curve. F. R. ENNOS.

**Precipitating baths for artificial silk.** NOVASETA A.-G. ARBON (B.P. 341,025, 8.10.29. Switz., 8.10.28).—Dissolved oxygen and carbon dioxide are removed from water that is to be used for precipitating baths in the stretch-spinning of cuprammonium silk by addition of sodium hyposulphite and an alkali hydroxide.

D. J. NORMAN.

**Manufacture of threads, bands, etc. of cellulose.** GLANZSTOFF-COURTAULDS GES.M.B.H., Assees. of VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 340,564, 1.10.29. Ger., 16.10.28).—Before spinning, viscose is mixed with a small proportion, *e.g.*, 0.7%, of a sulphonated fatty oil or a derivative thereof (Turkey-red oil), insufficient in amount to modify appreciably the properties of the finished product.

F. R. ENNOS.

**Manufacture of artificial threads.** I. G. FARBENIND. A.-G. (B.P. 341,021, 3.10.29. Ger., 5.10.28).—In the dry-spinning of solutions of organic derivatives of cellulose a greater degree of stretching is made possible, and therefore a considerably stronger filament is obtained if, immediately after extrusion of the filament, the solvent is, as far as possible, replaced by a swelling agent. To effect this, the upper part of the spinning cell is partitioned off as a swelling chamber and is fitted internally with a spiral gutter in which a continuous flow of the swelling agent is maintained.

D. J. NORMAN.

**Manufacture of artificial threads by the dry-spinning method.** I. G. FARBENIND. A.-G. (B.P. 340,570, 2.10.29. Ger., 5.10.28).—After leaving the spinning nozzle, the liquid threads enter a chamber which is freed from air and filled with the vapour of a swelling agent, which brings about an exchange of solvent for swelling agent; the jelly-like threads are then stretched and dried by a current of air in an adjoining chamber.

F. R. ENNOS.

**Manufacture of artificial filaments or threads.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 340,471 and 340,504—5, 20.7.29).—(A) Filaments, continuously with their production by the dry-spinning process, are lubricated, moistened, or sized and afterwards wound and twisted in a centrifugal box, 6—10 in. long and of relatively small diameter (3—4 in.), which is rotated at 12,000—20,000 r.p.m. The filaments are led into the box (B) through a guide by means of an ejector device supplied for the requisite short space of time with a blast of compressed air, or (C) by means of a spiral of sheet metal, the end of the filament being

passed to an outer turn of the spiral, whence it is carried by rotation of the centrifugal box to the centre of the spiral which forms a tube-like guide. F. R. ENNOS.

**Dry-spinning of solutions of cellulose derivatives.** BRIT. CELANESE, LTD., E. KINSELLA, J. BOWER, J. F. BRIGGS, and R. P. ROBERTS (B.P. 341,075, 7.10.29. Addn. to B.P. 325,233; B., 1930, 413).—The solution is extruded into a chamber through which an evaporative medium flows in countercurrent to the filaments while an additional evaporative medium is introduced inside or outside the bundle of filaments in the vicinity of the jet.

F. R. ENNOS.

**Manufacture of artificial threads and apparatus therefor.** COURTAULDS, LTD., W. H. GLOVER, and G. D. BOND (B. P. 339,745, 4.11.29).—The deleterious effects of currents and eddies set up in the spinning funnel during the stretch-spinning of, *e.g.*, cuprammonium silk are minimised by surrounding the thread by a conical or cylindrical foraminous screen. Monel metal gauze is a suitable material.

D. J. NORMAN.

**Manufacture of artificial filaments, yarns, ribbons, etc.** BRIT. CELANESE, LTD. (B.P. 341,148, 26.10.29. U.S., 1.11.28).—In the wet-spinning of solutions of organic derivatives of cellulose the spinning solution is maintained at a temperature above that of the spinning bath. This permits the use of a more concentrated spinning solution and facilitates the production of strong filaments of fine denier.

D. J. NORMAN.

**Manufacture of hollow artificial filaments, straws, etc.** BRIT. CELANESE, LTD. (B.P. 341,388, 26.10.29. U.S., 1.11.28).—A solution of an organic derivative of cellulose in a mixed solvent preferably of low b.p. (*e.g.*, methylene chloride and methyl alcohol) is spun into a coagulating liquid (such as mineral or vegetable oils) in which one constituent at least of the solvent mixture is insoluble or incompletely soluble; this liquid is heated above the b.p. of this insoluble constituent. (Cf. B.P. 341,148; preceding abstract.)

F. R. ENNOS.

**Manufacture of artificial materials from cellulose.** I. G. FARBENIND. A.-G. (B.P. 340,138, 30.12.29. Ger., 29.12.28).—The freshly precipitated fibres of cellulose hydrate from a wet-spinning process are stretched over rollers; one or more of which are heated, and irrigated with a suitable liquid to minimise crystallisation of spinning-bath salts.

F. R. ENNOS.

**Manufacture of artificial filaments or threads.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 340,929—30, 2.7.29).—Solutions of cellulose derivatives are extruded into a vigorous evaporative atmosphere and the filaments, after formation of a skin-like outer layer, are passed into an atmosphere of solvent or other vapour to soften this outer layer; the softened filaments are then stretched by applying tension (A) throughout their whole length, (B) wholly or partly to that portion which has been softened, and are subsequently freed from solvent in another evaporative atmosphere and again stretched after reaching the elastic limit.

F. R. ENNOS.

**Manufacture of hollow artificial threads.** H. KARPLUS (U.S.P. 1,770,310, 8.7.30. Appl., 9.12.27. Czechoslov., 21.12.26).—High-boiling petroleum fractions

or other hydrocarbons are incorporated with the spinning solutions (viscose, ammoniacal copper oxide, cellulose acetate or nitrate) and are afterwards removed by subjecting the spun filaments to heat and vacuum, thereby leaving void the spaces previously occupied by these substances. F. R. ENNOS.

#### Production of endless bundles of artificial fibres.

I. G. FARBERIND. A.-G. (B.P. 342,027, 13.11.29. Ger., 16.11.28).—Fibre bundles of 40,000–100,000 deniers, the individual filaments of which have dry and wet strengths, respectively, of 4 and 2 g. per denier for a filament denier of 1.4, are obtained from viscose by collecting the filaments from a number of spinning nozzles and passing the freshly precipitated and still acid-impregnated fibre bundle thus obtained through a bath 2–12 m. in length containing water at 70–100° at a speed of 40–60 m./min. and subjecting the thread to tension, *e.g.*, 0.5–0.8 kg./mm.<sup>2</sup> Dilute acid spinning baths must be used. D. J. NORMAN.

**Manufacture of [elastic] artificial silk.** G. COUDENE (B.P. 340,602, 3.10.29. Fr., 4.10.28).—Artificial silk is immersed in water or water vapour, in which it is allowed its natural elongation, and is afterwards dried under conditions which prevent contraction.

F. R. ENNOS.

**Manufacture of artificial ribbons, straw, etc. [of flattened cross-section].** H. DREYFUS (B.P. 341,034, 3.10.29).—A number of filaments of cellulose acetate or other thermoplastic derivatives of cellulose are subjected to heat and pressure, *e.g.*, by heated rollers, so that they coalesce; other yarns, metallic wires, dyes, powders, or wax-like materials may be incorporated in the flattened product.

F. R. ENNOS.

**Treatment of threads, filaments, etc.** COURT-AULDS, LTD., and J. W. CHAYTER (B.P. 341,317, 22.2.30).—Spun cakes of artificial silk from the centrifugal box are treated with a washing or other liquor while they are supported on three or more rods (suitable arrangement described and figured), carrying a number of such cakes.

F. R. ENNOS.

#### Manufacture of waterproof fibrous products.

A. L. CLAPP (B.P. 319,745, 26.9.29. U.S., 27.9.28).—Milk bottles etc. are made by mixing a cellulose pulp with a thermoplastic waterproofing material such as paraffin wax and then moulding this at a temperature above the m.p. of the wax; the resulting articles are sterilised by heating the mixture before moulding.

A. J. HALL.

**Manufacture of capsules made of cellulose esters which are capable of shrinking and are stored in air.** KALLE & Co. A.-G. (B.P. 319,736, 12.9.29. Ger., 27.9.28).—The water-swollen capsules are treated with a water-soluble, non-volatile liquid (glycerin, polyglycol, monoglyceryl lactate, etc.) which has no greater swelling action on the cellulose ester than water, with or without a preliminary immersion in a dilute aqueous solution of the impregnating liquid or of a salt. F. R. ENNOS.

**[Preventing shrinkage of water-swollen] bodies, such as capsules, made of cellulose esters.** KALLE & Co. A.-G. (B.P. 317,728, 17.8.29. Ger., 20.8.28).—Shrinkage of the capsules etc. on storage at a low

temperature is prevented by immersing them for a short time in a salt solution, which is washed out therefrom by immersion in water before use. [Stat. ref.]

F. R. ENNOS.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 341,237, 16.12.29. Switz., 15.12.28).—Alkali-cellulose is treated in the cold with a mixture of carbon disulphide and an alkyl or aralkyl halide, and the product, after washing with water, is warmed with an organic base (piperazine, diethylethylenediamine, etc.); the resulting material, which retains its original fibrous structure and has a satisfactory grip and tensile strength, has a strong affinity for acid dyes. F. R. ENNOS.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 342,167, 21.2.30. Switz., 11.4.29).—Alkali-cellulose is treated with a solution of a heterocyclic compound containing one or more N:C-Hal groups, *e.g.*, cyanuric chloride, dichloroquinazoline, tribromopyrimidine. The treated material is rinsed with hot water, then with very dilute hydrochloric acid, and finally with hot water. The product is thus immunised to direct cotton dyes and shows a good affinity for basic dyes and insoluble cellulose acetate dyes, whilst the fibres show no loss of strength on prolonged storage.

D. J. NORMAN.

**Manufacture of esters of cellulose.** A. NATHANSON (B.P. 340,651, 15.10.29. Ger., 16.10.28).—An aliphatic ester (C to C<sub>6</sub>) of cellulose is treated in a solvent (acetone, chloroform, ethers) with phosphoryl chloride or other phosphorus halogen compounds (*e.g.*, an organic chlorophosphine) in small amount (3%) and an organic base (pyridine, quinoline, aniline), whereby degraded cellulose molecules are polymerised and a product of high viscosity is obtained, which gives threads of good tensile strength.

C. HOLLINS.

**Manufacture of cellulose esters or ethers.** BRIT. ACETATE SILK CORP., LTD., F. W. STOYLE, G. A. EDWARDS, and A. G. LIPSCOMB (B.P. 340,555, 23.9.29).—Cotton linters or high-grade wood-pulp is activated before esterification or etherification by treatment at 10–50° for 12–48 hrs. with 2–10% of chloromethyl ether, chloromethyl alcohol, carbonyl chloride, acetyl chloride, propionyl chloride, or other halogen compound of the type Hal·CH<sub>2</sub>·OR or Hal·CO·X, where R is hydrogen, alkyl, or aryl and X is halogen, alkyl, or aryl, preferably in a solvent (acetic acid, trichloroethylene, benzene, tetrahydronaphthalene). The activating agent may be removed by hydro-extraction, or neutralised with sodium acetate.

C. HOLLINS.

**Manufacture of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 341,147, 26.10.29. U.S., 26.10.28).—Nitro-organic esters of cellulose (cellulose nitroacetate) having more than 10% N are prepared by treating cellulose at 0–15° with fuming nitric acid and an esterifying agent (acetic anhydride) in the presence of a diluent (acetic acid) if desired; a catalyst is unnecessary. Alternatively, esterification may be effected by using a catalyst (phosphoric acid) in the presence of acetic and nitric acids without acetic anhydride. F. R. ENNOS.

**Manufacture of cellulose nitro-acetates.** I. G. FARBERIND. A.-G. (B.P. 319,285, 18.9.29. Ger., 19.9.28).

—Cellulose is treated at 50–100° with acetic acid, acetic anhydride, and ammonium or urea nitrate, in the presence of 0.3–1% of sulphuric acid. F. R. ENNOS.

**Manufacture of paper and the like and preparation of the pulp.** A. E. WHITE. From MEAD PULP & PAPER Co. (B.P. 339,333–4 and 339,599, 28.8.29).—(B, c) High yields of pulp of good colour and strength suitable for high-grade printings are obtained from grasses, woods, etc., particularly hard woods such as oak, walnut, mahogany (saw-mill waste), and chestnut (tanning extract waste), by a mild digestion with a substantially neutral liquor followed by a two-stage chlorination treatment. Thus chestnut chips after a preliminary steaming are impregnated at 125° for about 2 hrs. with a liquor containing 13% of sodium sulphite and 5% of sodium bicarbonate (both calc. as sodium carbonate on the wt. of wood) in sufficient water to give an 8–12% solution of sodium sulphite. Excess of liquor is then blown off and the chips are heated to at least 160–180° for 1–4 hrs., depending on the degree of cooking required. The  $p_H$  of the liquor should be maintained at 5–9, any deviation from a predetermined optimum pressure-temperature ratio for this  $p_H$  range being corrected by the relief of excess pressure or by injecting carbon dioxide into the digester. The cooked and washed chips are then opened up in a rod mill, screened, diluted to a consistency of 4–6%, and treated with sufficient of a 0.2–0.4% solution of chlorine to satisfy 80–90% of the total chlorine requirements of the pulp. After about  $\frac{1}{2}$  hr. the pulp is washed with dilute soda solution and brought to a consistency of 20–30%, at which concentration it is passed downward through a special chlorinating tower (A) in which a predetermined depth of chlorine is automatically maintained by a sensitive pressure-recording device. The rate of travel of the pulp should be such that this second chlorination lasts about 15–30 min. The treated pulp is then washed through a water-seal at the bottom of the chlorinator by jets of water (also automatically controlled) and steeped in 0.1% caustic soda solution for  $\frac{1}{2}$  hr. The pulp is finally bleached at a consistency of about 5% with hypochlorite solution containing 1% of active chlorine on the dry weight of pulp. D. J. NORMAN.

**Stencil sheet.** S. HORN (U.S.P. 1,770,697, 15.7.30. Appl., 8.10.27).—A fibrous base, *e.g.*, yoshino paper, is coated with a solution of esters of polysaccharides and liquid waxes, *e.g.*, sperm oil, in a suitable volatile solvent or solvent mixture. Tempering agents, *e.g.*, fatty acids, fatty oils, naphthenic acid glycerides, etc., may also be added. The resulting coating is permanent and unaffected by changes of temperature. D. J. NORMAN.

**Stencil manufacture.** A. B. DAVIS, Assr. to A. B. DICK Co. (U.S.P. 1,771,165, 22.7.30. Appl., 5.8.26).—Aralkyl, *e.g.*, benzyl, *p*-chlorobenzyl, and phenylethyl, ethers of cellulose are dissolved in a volatile solvent and applied to yoshino paper. Softeners, lubricants, and colouring agents may be added. Details of a suitable composition are given. D. J. NORMAN.

**Stencil sheet material.** W. W. TRIGGS. From SELECTOGRAPH Co. (B.P. 339,952, 16.9.29).—Glue or gelatin (32 pts.) is soaked in cold water, and, after heating

the mixture to 70°, linseed oil (12 pts.) containing a drying agent (litharge) is stirred in until the whole is emulsified. Then 40 pts. of glycerin are added, the emulsion is thinned with hot water, and when cool applied to a fibrous support such as yoshino paper.

F. R. ENNOS.

**Production of fibrous material.** R. B. RESPESS, Assr. to RESPATS, INC. (U.S.P. 1,770,430, 15.7.30. Appl., 7.8.28).—Wood waste rich in resin is cooked in a caustic solution to soften and reduce it to bundles of fibres which retain a large proportion of the original gums, and is afterwards kneaded in a reducing mill with an abrasive (sand). After separation of the abrasive, the fibrous pulp is sheeted, pressed, and heated.

F. R. ENNOS.

**Manufacture of fibrous compositions.** E. P. STEVENSON and H. A. BURON, Assrs. to RICHARDSON Co. (U.S.P. 1,771,150, 22.7.30. Appl., 1.11.28).—Asphaltic material (80 pts.), after reduction to a viscous, semi-liquid state by heat, is mixed with 20 pts. of wet paper or fibrous pulp saturated with water until substantially homogeneous. During the mixing process, the charge is treated with 50 wt.-% of water and later reduced to pulp in a beater engine.

F. R. ENNOS.

**Waterproof paper products and the like. Water-resistant paper or paper-board product.** A. L. CLAPP, Assr. to FLINTKOTE Co. (U.S.P. 1,771,744–5, 29.7.30. Appl., 9.6.26. Renewed [A] 24.3.27).—A dispersion of asphalt (m.p. 38–116°) in (A) sodium silicate, (B) a soap solution, *e.g.*, resin size, is incorporated with a pulp suspension in sufficient quantity to give up to 40% of asphalt on the weight of dry pulp. Excess of a precipitant, *e.g.*, alum, is then added and the resulting stock formed into sheets. These are dried and heated to fuse the asphalt. Softening agents, *e.g.*, mineral oil, and/or other thermoplastic waterproofing agents may be mixed with the asphalt. D. J. NORMAN.

**Drying [of fibrous or cellulosic] goods.** R. BENNET (B.P. 339,817, 30.12.29).—The material, *e.g.*, wood boards, preheated if desired, is placed in contact with air, carbon dioxide, or other suitable water-soluble gas under pressure to promote dissolution of the gas in the water contained in the capillaries of the material. On subsequently reducing the pressure below atmospheric the dissolved gas is released and drives the capillary water to the surface. Suitable apparatus is described.

D. J. NORMAN.

**Means for identifying materials [detecting alterations in cheques etc.].** D. J. BLOCK (U.S.P. 1,771,612, 29.7.30. Appl., 26.1.28).—Compounds which fluoresce in ultra-violet light, *e.g.*, quinine and its salts, uranium salts, and particularly the glucoside, aesculin, are used in the preparation of safety paper. Erasures on paper thus treated show up as dark patches when examined in ultra-violet light. D. J. NORMAN.

**Extraction of formic acid from residual lye from the purification of cellulose.** PAPETERIES NAVARRE (B.P. 342,119, 22.1.30. Fr., 30.11.29).—The spent soda liquor from the process of B.P. 318,868 (B., 1929, 1010) is distilled with an acid (sulphuric acid, *d* 1.83) or an acid salt which is stronger than formic acid. D. J. NORMAN.



[Machine for] separating bark or bast from its supporting structure. D. W. DRON, and DRON ENGINEERING CO., LTD. (B.P. 342,672, 25.10.29).

Handling cakes of spun artificial silk. VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 343,253, 28.12.29. Ger., 3.1.29).

Soda recovery.—See I. Paraffin emulsion.—See II. Bleaching.—See VI. Structural materials.—See IX. Sulphite liquor spirit.—See XVIII. Cellulose compounds.—See XXI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Treatment and dyeing of furs. M. DOROGUE (Halle aux Cuirs, Tech. Suppl., 1930, 110—117; Chem. Zentr., 1930, ii, 3108).—The tanning and dyeing processes in the preparation of imitation furs from rabbit skins are discussed. A. A. ELDRIDGE.

Printing with sulphur dyes. C. SCHWARTZ, H. COLLIN, and M. GRANDERYE (Scaled Note No. 1679, 30.11.06. Bull. Soc. Ind. Mulhouse, 1930, 96, 627—630). Report by A. LAU (*Ibid.*, 630—632).—Bright shades having excellent fastness are obtained in printing sulphur dyes on cotton fabrics by using a printing paste containing a high proportion of glycerin and a mixture of glucose with a small amount of caustic alkali instead of sodium hyposulphite; printing is followed by steaming in the absence of air at 105—106° for 3—6 min., in which process the temperature of the fabric may rise to 115°. A suitable printing paste contains sulphur dye 20—120 g., anhydrous dextrose 60—100 g., caustic soda (32%) 120—150 g., glycerin 180—250 g., and gum solution 620—380 g. Thioindigo Red B requires the use of sodium formaldehyde-sulphoxylate instead of dextrose and caustic soda. LAU reports favourably on the process, and observes that, although several similar processes have been published, the importance of the glycerin is diminished when steaming is effected in a moisture-saturated atmosphere. A. J. HALL.

Continuous damping and steaming of printed wool fabrics. F. BINDER (Sealed Notes Nos. 1527—8, 28, and 30.3.05. Bull. Soc. Ind. Mulhouse, 1930, 96, 625—626). Report by A. WOLF (*Ibid.*, 626—627).—A higher efficiency in the fixation of colours printed on wool fabric is obtained by moistening the fabric, immediately before the usual steaming process, by passage in contact with an endless, wet cotton, moleskin fabric over a series of wooden rollers; at one point in its travel the moleskin fabric is washed free from absorbed colour and mangled so that it retains a suitable amount of moisture. WOLF reports favourably, but states that the method, although formerly used to a large scale, has now been abandoned. A. J. HALL.

Soaking of silk. H. A. NEVILLE and T. H. MARSHALL (Ind. Eng. Chem., 1931, 23, 58—62).—The hydration of silk has been investigated dilatometrically. When pure water is used the volume of the system contracts, indicating the hydration of the silk, then increases owing to bacterial action. In soap solutions and dye baths no increase in volume is noted, owing, it is claimed, to the antiseptic action of these wetting media. Hydration is greatest in acid solutions, it also being necessary to avoid

alkaline baths owing to the tendency for sericin to be dissolved, thus weakening the filaments. Where soap is used as a dispersing agent in oil emulsions, increase in concentration of soap causes decreased hydration. Triethanolamine is shown to give highly satisfactory hydration when used in oil emulsions (as the oleate) or with sulphonated oils. T. T. PORTS.

Chromium acetate.—See VII. Turkey-red oil.—See XII.

## PATENTS.

Bleaching (A) [of fibrous materials], (B) of pulp. (c) Treatment of wood pulp. L. BRADLEY and E. P. MCKEEFFE, ASSTS. to BRADLEY-MCKEEFFE CORP. (U.S.P. 1,768,819—20 and 1,768,823, 1.7.30. Appl., [A] 2.3.21, [B] 16.3.22, [C] 13.6.29. Renewed [A] 18.10.29. Can., [C] 1.12.21).—Chemical wood pulp etc. is (A) bleached by regulated additions of sodium permanganate and hypochlorite, prepared by the action of chlorine on sodium manganate and alkali formed electrically or by fusion, the soluble compounds produced being removed before each fresh addition of bleaching agent; the insoluble oxide of manganese is afterwards dissolved by means of sulphur dioxide, bisulphite, etc.; (B) partly bleached by treatment with acid hypochlorite solution, and, after separation of the liquor, completely bleached by means of alkaline hypochlorite solution; or (c) incompletely bleached with alkali hypochlorite, and, after removal of the solution, is further treated with alkali and alkali peroxide at 60°. F. R. ENNOS.

Bleaching [of cellulose]. L. BRADLEY and E. P. MCKEEFFE, ASSTS. to BRADLEY-MCKEEFFE CORP. (U.S.P. 1,768,821—2, 1.7.30. Appl., 14.3.28).—Wood pulp is bleached by a first treatment with a liquor containing (A) reactive chlorine, or (B) a manganate or permanganate, insufficient in amount to effect complete bleaching, followed by washing out of the solubilised impurities and then completing the bleaching with a second treatment with an oxidising liquor; such a multi-stage bleaching process leads to economy in bleaching reagents and a superior grade of bleached pulp. A. J. HALL.

Bleaching of [cellulose] textile fibres. H. P. BENSON (U.S.P. 1,773,721, 26.8.30. Appl., 7.11.28).—The usual natural and added impurities present in grey cotton fabrics are eliminated more rapidly and with much less than the usual consumption of water by first treating the fabric with a starch-liquefying enzyme, washing, impregnating with milk of lime, and then exposing the fabric to ultra-violet light for about 6 hrs., followed by thorough washing. The beneficial effect of the ultra-violet light is diminished by washing out the milk of lime before exposure. A. J. HALL.

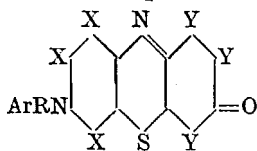
Dyeing with vat dyes. J. E. G. HARRIS, J. MORTON, and MORTON SUNDOWN FABRICS, LTD. (B.P. 340,267, 19.6.29).—The injurious action of an alkaline vat on wool etc. is prevented by adding to the vat an amount of boric acid (1.0—1.5 times the weight of caustic alkali present) sufficient to reduce the alkalinity without complete neutralisation. Usually the dyeing takes the shade of the free (acid) leuco-compound; oxidation is slow and may require a souring bath of hot dilute acetic acid. Caledon Red BN and Red Violet 2RN show good



affinity when dyed by this process. Conditions for numerous other vat dyes are exemplified.

C. HOLLINS.

**Manufacture of fast printings [of thiazine-quinone dyes] on vegetable fibre.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 341,119, 15.10.29).—The fibre is printed, by the methods used for vat dyes, with thiazinequinones of the annexed formula, in which



Ar is aryl, R is hydrogen, alkyl, or aralkyl, X is hydrogen or a substituent, and Y is hydrogen or halogen. Examples are the quinone dyes from chloranil and 2-amino-5-anilinothio-*m*-cresol (reddish-blue), 2-amino-5-diphenylaminothio-*m*-cresol (greenish-blue), 2-amino-5-*m*-chloroanilino-3-methoxythiophenol (blue), and 2-amino-5-*p*-toluidinothio-*m*-cresol (blue). The direct printing method is preferred.

C. HOLLINS.

**Coloration of textile or other materials [especially cellulose ethers or esters].** BRIT. CELANESE, LTD., G. H. ELLIS, T. OCKMAN, and H. C. OLPIN (B.P. 340,541 and 340,572, 25.6.29).—The fastness of amino-anthraquinone dyeings to the combined action of light and acids is improved by treating the material, before, during, or after dyeing, with (A) uncoloured alkylated aminodiaryls (tetramethyl-4 : 4'-diaminodiphenylmethane, tetramethylbenzidine), or (B) alkylated, aralkylated, or arylated carbamides, thiocarbamides, or guanidines (*s*-diisooamylthiocarbamide, tetramethylthiocarbamide), which are retained in the material during any subsequent treatment.

C. HOLLINS.

**Dyeing of regenerated cellulose materials.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 340,479, 26.8.29).—Level violet to blue shades are obtained by application of disazo dyes of the type: *p*-aminoazo compounds  $\rightarrow$  1 : 5 : 7-aminonaphtholsulphonic acid or an *N*-derivative thereof; the aminoazo compound may contain carboxyl, alkyl, halogen, or nitro-substituents and either 1 or 2 sulphonic groups. Examples of aminoazo components are: aniline  $\rightarrow$  Cleve acid; technical aminosulphonic acid  $\rightarrow$   $\alpha$ -naphthylamine; *p*-nitroaniline-2-sulphonic acid  $\rightarrow$  cresidine; 4-aminoazobenzene-4'-sulphonic acid; *p*-nitroaniline  $\rightarrow$  Cleve acid;  $\beta$ -naphthylamine-4 : 8-disulphonic acid  $\rightarrow$  cresidine.

C. HOLLINS.

**Production of fast tints on artificial silk of regenerated cellulose.** SOC. CHEM. IND. IN BASLE (B.P. 318,832, 9.9.29. Switz., 7.9.28).—Viscose silk is dyed with prechromed azo dyes (excluding those containing a cyanuric residue) obtained by coupling *J*-acid or an *N*-substituted *J*-acid with a diazotised *o*-aminophenol or anthranilic acid (excluding aminosulphonic acids) containing not more than 1 sulphonic or carboxyl group. The dyes of B.P. 296,680 and 295,594 (B., 1929, 891; 1930, 277) are excluded. Examples are the chromium compounds of 5-nitro-*o*-aminophenol  $\rightarrow$  phenyl-*J*-acid (blue); anthranilic acid  $\rightarrow$  phenyl-*J*-acid (violet); 4-nitro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  di-*J*-acid (greenish-blue); *o*-aminophenol-4-sulphonamide  $\rightarrow$  carbonyl-*J*-acid (lilac).

C. HOLLINS.

**Production of a design on textile material.** G. H. HUDSON (B.P. 341,931, 26.7.29).—A coloured embossed design is produced by placing the textile material against a moulded surface covered with a dry film of colour, superimposing a moistened lamina (*e.g.*, a sheet of felt), and then pressing with a heated member so that steam passes through the textile material, dissolves dye from the surface of the mould, and transfers it to the textile material. The mould is perforated to allow escape of steam.

A. J. HALL.

**Device for rapidly ascertaining quantities of ingredients necessary for preparing dye-baths.** I. G. FARBENIND. A.-G. (B.P. 339,862, 3.2.30. Ger., 2.2.29).—A cardboard sheet carrying numbers on one or both sides slides between cover sheets provided with names and cut-out openings so that a series of concentrations with the appropriate quantities of auxiliary ingredients, *e.g.*, for normal, warm, or cold indanthrene vats, can be read off at different positions of the moving card.

C. HOLLINS.

**Manufacture of fabrics containing effect threads fast to boiling.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,501, 30.9.29).—Effect threads more stable to boiling water than cellulose acetate threads or partly acetylated cotton threads or cotton threads coated with cellulose nitrate are manufactured from cellulose nitroacetates. Stability is ensured by the presence of about 1% of combined nitrogen in the nitroacetate.

A. J. HALL.

**Manufacture and treatment of artificial filaments, yarns, threads, etc.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 341,057, 5.9.29).—Effect materials in the form of metallic, coloured, luminous, phosphorescent, or delustring powders, or of short fibres of natural or artificial materials, are applied to filaments of organic derivatives of cellulose or of regenerated cellulose, while they are still sticky through retention of solvent or treatment with a solvent or softening agent, by means of a gaseous stream carrying the materials which is directed across the path of the filaments in the course of their production.

F. R. ENNOS.

**Delustring of [cellulose] artificial silk.** G. TAGLIANI, ASS. to MUNITEX CORP. (U.S.P. 1,770,114, 8.7.30. Appl., 20.4.28).—Artificial silk materials consisting of regenerated cellulose are treated with an alkalisng solution such as a 14% alcoholic solution of caustic soda or potash and then, before or after drying, with an esterifying agent such as a carboxylic acid chloride, a sulphonyl chloride, or the anhydride of the corresponding acid, dissolved in an organic solvent (a 20% solution of *p*-toluenesulphonyl chloride in carbon tetrachloride is suitable).

A. J. HALL.

**Delustring of artificial silk fibres or rayon.** O. F. MULLER, ASS. to NYANZA COLOR & CHEM. CO., INC. (U.S.P. 1,769,850, 1.7.30. Appl., 11.5.29).—Artificial silk materials are delustred by treatment in an emulsion containing paraffin wax, soap, glue, and an abrasive material such as chalk, kaolin, zinc oxide, and fuller's earth.

A. J. HALL.

**Treatment [delustring] of materials made of or containing organic derivatives of cellulose.**

**BRIT. CELANESE, LTD.** (B.P. 318,467, 2.9.29. U.S., 1.9.28).—The lustre of cellulose acetate silk is reduced by precipitation therein of a white insoluble substance after pretreatment with a swelling agent. Good results are obtained by precipitating barium, calcium, and strontium salts; formic and acetic acids and acetone are suitable swelling agents. In some instances (*e.g.*, with barium thiocyanate) one of the substances involved in the process of precipitation may also be used as the swelling agent. A. J. HALL.

**[Softening] treatments for textiles, leather, and the like.** H. T. BÖHME A.-G. (B.P. 317,468, 2.7.29. Ger., 18.8.28).—Exceptional smoothness and pliability are conferred on the materials by treating them with alcohols corresponding to the higher fatty acids, such as stearic and oleic acids, dissolved or dispersed in a liquid vehicle such as benzene, turpentine or pyridine, and then removing the vehicle. Particularly good results are obtained with artificial silks. A. J. HALL.

**Manufacture of stiff paper, linen, silk, or like sheets metallised on one or both sides.** W. RUPPERT (B.P. 340,991, 30.9.29. Ger., 5.11.28).—Webs of paper (*etc.*) are coated with an artificial resin varnish, dried, and superimposed to give a product of the required thickness. Metal foil is then applied to one or both surfaces and the whole subjected to heat and pressure. D. J. NORMAN.

**Protection of wool, fur, hair, *etc.* from attack by moths and other textile pests.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,318—9, 27.9.29).—(A) Selenious acid or a selenite or selenate (*e.g.*, ammonium or ethylenediamine salt), or (B) a thioacylamide or a thioacylarylamide, *e.g.*, thiobenzamide, thioacetanilide, thioisovaleric, thioisohexanoic, or thiophenylacetic anilide, is used as mothproofing agent. C. HOLLINS.

**Preparation of products for impregnating, protecting, and preserving cellulose materials.** S. TCHAYEFF (B.P. 341,092, 11.10.29. Fr., 17.10.28).—A mixture of the fluoride or sulphate of copper or zinc with a phenol (other than nitrophenols and phenol-sulphonic acids) or a naphthol is claimed. D. J. NORMAN.

**Purification of solvents for use in dry cleaning.** L. E. GAUME, Assr. to MIRACLEAN Co. (U.S.P. 1,770,266, 8.7.30. Appl., 18.10.24).—Purification of organic solvents is effected in a cylindrical vessel, by successive treatments with acid and alkaline solutions, each treatment comprising spraying the purifying liquor into the solvent, followed by a period of circulation of the mixture through a pump and subsequent separation of the purifying liquor by settling and decantation. A. J. HALL.

**Steaming and drying machines [for garments].** H. A. SHIELDS (B.P. 342,252, 16.10.29).

**Wool-cleaning and -scouring apparatus.** H. G. and T. R. HOWELL (B.P. 343,264, 10.1.30).

**Drying.**—See I. Sulphonic acids.—See II. Auxiliary agents.—See III. Dibenzanthrone dyes.—See IV. Cellulose derivatives.—See V. Cleansing agents.—See XII. Lacquers.—See XIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Theory of contact processes [for sulphuric acid manufacture].** J. FISCHLER (Przemysl Chem., 1931, 15, 73—76).—The expression  $P = 1.53a\sqrt{(d-u)/u}$  is derived, in which  $P$  represents the daily production of sulphuric acid by a given contact plant,  $a$  is a constant for the given plant, and  $u$  is the coefficient of activity of the catalyst. The values of  $P$  given by this expression are in good agreement with experimental results. R. TRUSZKOWSKI.

**Intensive sulphuric acid manufacture [by the chamber process].** E. OWSIANY (Chem.-Ztg., 1931, 55, 76).—The author's process consists in the intimate mixture of the gases with atomised liquid in such a way that they are broken up, compressed, and allowed to expand. Two of his units, which are not described, were inserted in a lead-chamber plant before and after the leading chamber and resulted in an increased output of 28% together with easier working. The intensity of reaction in the units was 270—400 times that in the chambers. It is intended that the apparatus shall replace not only chambers, but Glover and Gay-Lussac towers, and shall be applied to other processes of gas absorption. C. IRWIN.

**Sodium sulphite from waste gases from the manufacture of contact sulphuric acid.** I. N. KUZMINYCH (J. Chem. Ind., Moscow, 1930, 7, 1734—1737).—The waste gases are scrubbed by a sodium carbonate solution, which then flows to a reservoir, where sodium sulphite crystallises out; the mother-liquor is again passed through the scrubbing tower, after addition of sufficient soda to maintain an alkaline reaction after passage. R. TRUSZKOWSKI.

**Solving "mixed-acid" problems by use of determinants.** E. P. DEATRICK (Ind. Eng. Chem., 1931, 23, 34—35).—The use of determinants simplifies the solving of many chemical calculations which are otherwise tedious. A worked example is given. H. INGLESON.

**Loss of material and corrosion of apparatus during the evaporation of solutions of ammonium nitrate.** L. WASILEWSKI and W. BADZYŃSKI (Przemysl Chem., 1931, 15, 61—71, 83—95).—Solutions of ammonium nitrate evolve ammonia on concentration in glass vessels; this process is most intense about 140°, and ceases in slightly acid solution, so that no ammonia is lost if 0.5 g. of nitric acid per kg. of ammonium nitrate be added to the solution. In the presence of sheet iron the distillate contains nine times as much ammonia as in its absence; at the same time fairly large quantities of nitric acid distil over, and part of the iron dissolves. As the quantity of added nitric acid is increased from 0 to 1.0 g. per kg. of 40% solution, loss of ammonia falls from 0.4049 to 0.0085 g.; loss of nitric acid at first falls from 0.3181 to 0.1376 g. when 0.4364 g. of nitric acid was added, and then rises to 0.5644 g. when 1.067 g. of nitric acid were added; the corrosion of iron at first rises from 0.1882 to 0.3073 g. in presence of 0.4364 g. of extra nitric acid, and falls practically to zero in higher acid concentrations, pointing to passivation of iron, the product being in these cases colourless

and free from iron. Acidification of the solution by phosphoric acid (0.568 g.) gives better results than nitric acid; the loss of ammonia and nitric acid is half as great, and no corrosion takes place. Sulphuric acid, potassium hydrogen sulphate, trisodium phosphate, and calcium hydrogen phosphate are unsuitable, corrosion of iron and loss of ammonia occurring in all cases. As good results as with phosphoric acid are obtained by the addition of 50 c.c. of an aqueous extract of superphosphate (350 c.c. of water to 150 g. of superphosphate) to 1 kg. of 40% ammonium nitrate. Evaporation in the presence of acid-resistant metals ("Avesta 832" and Krupp's "V2A" iron sheet) give better results in the presence of additional nitric or phosphoric acid. Evaporation in a cast-iron vessel gives unsatisfactory results where neutral solutions or solutions acidified with nitric acid are taken (considerable corrosion and loss of ammonia and nitric acid); in the presence of phosphoric acid or superphosphate extract containing 3% of sulphuric acid corrosion and loss of material are reduced to a minimum.

R. TRUSZKOWSKI.

**Dehydration of Glauber's salt.** M. A. RAKUSIN (Metallbörse, 1930, 20, 1909—1910; Chem. Zentr., 1930, ii, 3324).—Various methods, with or without the application of heat, are described. A. A. ELDRIDGE.

**Manganese in bleaching powder.** O. H. KÜRSCHNER (Pharm. Ztg., 1931, 76, 184—186).—The total manganese content (0.003—0.012%) and the proportion of this which appears as permanganate when the sample is boiled with water (cf. Dixon and White, A., 1927, 843) have been determined for ten commercial samples of bleaching powder. There is no connexion between the proportion of manganese oxidised and the oxygen value of the powder. To obtain permanganate-free bleaching liquors, the powder should be treated with cold water or cold sodium carbonate solution.

H. E. F. NOTTON.

**Preparation of chromates by direct dissolution of chromite in acid.** K. I. LOSEVA and T. M. ZOLOTYCH (J. Chem. Ind., Moscow, 1930, 7, 1795—1799).—Half of the chromium content of Ural chromite is dissolved by the action, during 8—10 min., of sulphuric acid ( $d$  1.83) at 150—160°; the remainder cannot be extracted by the action of fresh sulphuric acid or of hydrochloric acid.

R. TRUSZKOWSKI.

**Preparation of chromium acetate [for mordanting].** O. METZGER, M. GRANDERYE, and M. ZURCHER (Sealed Note No. 2235, 31.3.13. Bull. Soc. Ind. Mulhouse, 1930, 96, 632—634). Report by M. BATTEGAY (*Ibid.*, 634—635).—A solution of chromium acetate is prepared by gradually adding sodium bisulphite ( $d$  1.36; 450 g.) to a solution of sodium dichromate (170 g.), 30% acetic acid (335 g.), and sodium acetate (45 g.), allowing the temperature of the mixture to rise to b.p., and then allowing the sodium sulphate to separate by crystallisation from the chromium acetate formed. If excess of acetic acid is present (5 mols. instead of 3 mols.) a violet chromium acetate  $\text{Cr}(\text{OAc})_2\text{OH}$  is formed in addition to the green acetate  $\text{Cr}(\text{OAc})_3$ , but this precipitates and can afterwards be added to the green acetate. BATTEGAY cites a similar process of Scheurer (B., 1892, 33).

A. J. HALL.

**Determination of small quantities of nitrogen and carbon in technical hydrogen.** G. HEYNE (Tech.-Wiss. Abh. Osram-Konzern, 1930, 1, 343—348; Chem. Zentr., 1930, ii, 3059—3060).—*Nitrogen*: (a) The hydrogen is burned with excess of oxygen in a silica capillary tube containing a fairly closely-fitting platinum wire and heated by means of a small flame; excess of oxygen is absorbed in pyrogallol solution and the residual nitrogen measured. (b) The hydrogen is oxidised in a perpendicular hard glass tube containing cupric oxide and heated at 400°. Cylinder hydrogen contained 0.04—0.7 vol.-% of nitrogen. *Carbon*: The hydrogen is oxidised in a perpendicular hard glass tube, 650 mm. long, the upper (500 mm.) portion containing cupric oxide at 400° and the lower portion platinised alumina at 600°; the gas is passed downwards, addition of oxygen being unnecessary. The carbon dioxide is collected in barium hydroxide solution and determined titrimetrically. Cylinder hydrogen contained only a few thousandths to a hundredth of 1 vol.-% of methane or carbon monoxide.

A. A. ELDRIDGE.

**Determination of hydrogen in gaseous mixtures.** G. N. SCHUTOV (J. Chem. Ind., Moscow, 1930, 7, 1801—1802).—The gas is mixed with an equal volume of air and the mixture is passed over dry palladised pumice until no further decrease in volume takes place; this process is at ordinary temperatures completed in 3 min. The hydrogen content is calculated from the difference in volume. This method cannot be applied in the presence of carbon monoxide, unsaturated hydrocarbons, and acidic gases. R. TRUSZKOWSKI.

**Hydrogen and hydrogen-nitrogen mixtures.** **Carbon monoxide-hydrogen mixtures.**—See II. **White lead.**—See XIII. **Iodine in shrimps.**—See XIX. **Carbon monoxide.**—See XXIII.

See also A., Mar., 303, Sols. 311, Glauber's salt. 320, Sylvine. 324, Iodine trichloride. 339, Cyanic acid and carbamide.

## PATENTS.

**Manufacture of sulphuric acid of a purity known commercially as chemical purity.** W. S. ALLEN, Assr. to GEN. CHEM. CO. (U.S.P. 1,771,520, 29.7.30. Appl., 29.8.24).—Gases containing sulphuric anhydride from the contact process, after being freed from suspended impurities, are passed into a reaction chamber into which steam is admitted, the gases being at such a temperature that the sulphuric acid formed is maintained in the state of vapour without formation of mist. The vapour is passed through an air-cooled condenser into an absorption tower, whence liquid sulphuric acid is drawn off.

W. J. WRIGHT.

**Manufacture of ammonia.** INST. F. PHYSIKAL. GRUNDLAGEN DER MEDIZIN (B.P. 340,862, 28.2.30. Ger., 1.3.29).—The reaction chamber, in which the hydrogen-nitrogen mixture is subjected to dark electric discharges, consists of a device similar to the Siemens ozone tube and can be heated above 132°, the walls of the chamber being provided with catalysts, e.g., cobalt nitrate. The rate of flow of the gases is adjusted so as to prevent undue decomposition of the ammonia.

W. J. WRIGHT.

[Catalyst for the] ammonia synthesis process. A. T. LARSON, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,771,130, 22.7.30. Appl., 29.3.27).—The catalyst consists of iron promoted with potassium and magnesium oxides, together with an oxide of chromium, titanium, silicon, vanadium, lanthanum, tantalum, molybdenum, aluminium, tungsten, uranium, zirconium, manganese, or boron. W. J. WRIGHT.

Production of ammonium sulphates. R. C. BENNER and A. H. HENNINGER, Assrs. to GEN. CHEM. CO. (U.S.P. 1,769,938, 8.7.30. Appl., 30.6.26).—Sulphur dioxide is introduced into an ammoniacal solution so as to give a solution containing at least 2 mols. of bisulphite per mol. of sulphite, this solution being then heated to 200° under a pressure of 700 lb./in.<sup>2</sup> After separation of sulphur, the solution is evaporated and the crystallised salt calcined. W. J. WRIGHT.

Manufacture of potassium chloride and sodium and potassium carbonates. (A) R. D. PIKE and (B) R. CUMMINGS and L. V. STECK, (B) Assrs. to (A) (U.S.P. 1,770,995, 22.7.30. Appl., 6.6.28).—Wyomingite is leached under pressure with a mixed solution of sodium carbonate and Green River soda brine, the solution being evaporated to give crystalline sodium carbonate monohydrate. The mother-liquor is cooled to salt out potassium chloride crystals, evaporated in a secondary evaporator to yield additional sodium carbonate, and again cooled to yield additional potassium chloride. Part of the final mother-liquor is returned to the secondary evaporator, the remainder constituting the net yield of potassium carbonate. W. J. WRIGHT.

Preparation of high-percentage calcium cyanamide. N. CARO and A. R. FRANK (B.P. 341,299, 6.2.30. Ger., 8.2.29).—Calcium carbamate is converted directly into high-percentage cyanamide by treating it at 600–800° with ammonia and carbon monoxide. The waste gases may be condensed to ammonium carbamate, which may be used for producing the calcium carbamate. W. J. WRIGHT.

Production of calcium carbamate. N. CARO and A. R. FRANK (B.P. 341,386, 6.2.30. Ger., 8.2.29. Cf. B.P. 341,299; preceding abstract).—An aqueous ammoniacal solution of ammonium carbamate, saturated with ammonia and carbon dioxide under pressure, is caused to react with a calcium compound in solution or suspension, the mixture being cooled to –8° to –10° to precipitate the calcium carbamate. W. J. WRIGHT.

Treatment of aluminium oxide. T. A. MITCHELL, Assr. to L. M. HUGHES (U.S.P. 1,772,360, 5.8.30. Appl., 29.7.29).—The crude oxide is calcined with an alkaline-earth compound, preferably a carbonate and capable of forming an aluminate, in presence of a non-alkali sulphate capable of reacting with the alkaline-earth compound to form an alkaline-earth sulphate. W. J. WRIGHT.

Manufacture of products containing aluminium oxide and one or several sulphides. INTERNAT. PATENT CORP. (B.P. 341,160, 2.11.29. Swed., 2.11.28).—In the process described in B.P. 232,549 (B., 1925, 957) the formation of heavy-metal carbide and its conversion into aluminium carbide is prevented by

introducing a large part of the heavy metal, *e.g.*, iron, in the form of lumps; these sink to the bottom before they can combine with the carbon, and react with the aluminium. If heavy-metal sulphides, which lose their sulphur at a temperature below that of the furnace, are employed, iron granules may be added and the mixture formed into briquettes, a material such as bauxite, that normally contains or absorbs water, being also added, so as to produce rust and thus cause the briquettes to adhere. W. J. WRIGHT.

Manufacture of aluminium sulphate. M. B. ROBINSON, and ALUMINA CO., LTD. (B.P. 340,475, 23.8.29).—In the treatment of uncalcined fireclay with sulphuric acid, the former is ground to pass 40–200-mesh, and the acid is maintained at  $d$  1.55–1.42, so that the ore remains in suspension, weak aluminium sulphate solution from previous operations being added for this purpose, as required. W. J. WRIGHT.

Detecting the presence of carbon monoxide. G. LJUNGGREN (B.P. 341,269, 20.1.30).—Palladous chloride test-paper is impregnated with a solution of a buffer substance, *e.g.*, sodium acetate, the concentration of this being higher than that of the palladous chloride. W. J. WRIGHT.

Dryers etc. Gaseous mixtures.—See I. By-products from gas. Hydrogen from methane etc. Hydrogen sulphide from gases. Sulphuric acid from oils.—See II. Cellulose by-products.—See V. Zinc sulphide. Titania.—See XIII. Insecticides. Fungicide.—See XVI.

## VIII.—GLASS; CERAMICS.

Influence of chemical compositions on the physical properties of soda-lime glasses. J. W. GILL and W. C. RUECKEL (J. Amer. Ceram. Soc., 1931, 14, 21–29).—Nineteen glasses, the range of compositions of which was 5–15% (Ca,Mg)O, 10–20% Na<sub>2</sub>O, and 70–80% SiO<sub>2</sub>, were melted and fired for 2 hrs. Results indicate that softening temperatures are controlled largely by the soda content, whilst density and refractive index are dependent on the content of lime; the two last-named factors increase approximately in direct proportion with the increase in total bases.

R. J. CARTLIDGE.

Felspar and its effect in pottery bodies. R. F. GELLER and A. S. CREAMER (J. Amer. Ceram. Soc., 1931, 14, 30–71).—Eighteen potash felspars and one soda felspar were examined chemically and petrographically, and the comparative behaviour of the felspars in vitreous and semi-vitreous bodies was studied. Results indicate that refractoriness increases and the softening range decreases with increase in K<sub>2</sub>O, but these may be influenced by the content of iron oxide (Fe<sub>2</sub>O<sub>3</sub>) and of uncombined silica; the true sp. gr. ranged from 2.565 for potash felspars to 2.628 for soda felspars; the density of the felspar decreases as the firing temperature increases. The translucency of vitreous bodies increased with increase of K<sub>2</sub>O content in the potash felspars. The thermal expansion of both vitreous and semi-vitreous bodies was highest for those containing high-soda felspars, and the semi-vitreous bodies which

were cooled slowly showed the highest average expansions, disregarding the rate of heating.

R. J. CARTLIDGE.

**Effect of hydrogen-ion concentration on the sedimentation of clay.** N. BEEMAN (J. Amer. Ceram. Soc., 1931, 14, 72—87).—Small quantities of hydrochloric acid proved to be effective flocculating agents, the maximum effect being produced by 0.01*N*-, 0.02*N*-, and 0.001*N*-acid, whilst corresponding concentrations of caustic soda were without effect. Flocculation was produced by 0.1*N*-caustic soda, but the effect was less than that produced by the 0.002*N*-hydrochloric acid. Data given indicate that a separation by elutriation is uncertain without  $p_H$  control.

R. J. CARTLIDGE.

**Determination of crystallite by means of X-rays.** L. GREBE (Z. tech. Physik, 1930, 11, 428—429; Chem. Zentr., 1930, ii, 3175).—Debye lines are obtained with the specimen and with a pure comparison substance, and the intensities are compared. The intensity is proportional to the crystallite content, and not to the particle size. The method is applied to quartz in clay etc.

A. A. ELDRIDGE.

**Semi-silica versus lime-bonded silica for coke-oven walls.** H. D. BENNIE (Gas World, 1930, 93, Coking Sect., 113—116).—Semi-silica material of reputable make has a refractoriness under load of 50 lb./in.<sup>2</sup> of 1470°, whereas good silica bricks will withstand the same load up to 1670°. Under 26 lb./in.<sup>2</sup>, semi-silica has a refractoriness of 1620°, whilst that of lime-bonded silica is unchanged, *i.e.*, 1670°; the enormous advantage which silica appears to have under the conditions of the standard load of 50 lb./in.<sup>2</sup> is therefore appreciably reduced when the bricks are tested under a working load. Lime-bonded silica, even when burned to an extent which would be prohibitive in cost for ordinary works' production, has a much greater thermal (reversible) expansion than that of semi-silica, and also a greater permanent expansion. It is usual to employ silica bricks when the oven walls are to be operated at temperatures above 1500°, but, considering the refractoriness of semi-silica under a working load together with its small expansion, it should be possible to employ such a material at flue temperatures of 1500°. Lime-bonded bricks spall more readily than those of semi-silica. Semi-silica bricks possess the peculiar property of improving in use as regards conductivity, diffusivity, and refractoriness, without any accompanying disadvantages.

C. B. MARSON.

**Physical properties of glasses in relation to their composition. II. Mechanical properties. III. Viscosity.** G. GEHLHOFF and M. THOMAS (Z. tech. Physik, 1926, 7, 105—126, 260—278; Chem. Zentr., 1930, ii, 2686).

**Portable furnace. Tunnel dryers.**—See I.

See also A., Mar., 284, Thermoluminescent glasses. 328, Iron in silicates. 332, Clay.

#### PATENTS.

**Absorption glass.** F. GELSTHARP, ASST. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,771,435, 29.7.30. Appl., 11.12.26).—An absorptive glass not subject to discoloration with time is obtained by incorporating

$\frac{1}{4}$ — $\frac{1}{2}$  oz. of cobalt oxide per 1000 lb. of sand in a batch composed of sand 1000, soda ash 300, limestone 310, didymium-free cerium hydroxide 50, common salt 25, salt cake 60, coal 3, "arsenic" 5. M. PARKIN.

**Manufacture of compound glass.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 340,445, 21.2.30. Fr., 4.5.29).—The glass plates and interposed sheet of plastic material, after assemblage in a bath of a plastifying agent as described in B.P. 316,955 (B., 1930, 1029), are subjected to slight pressure and then autoclaved under a gaseous pressure of, *e.g.*, 3—5 kg./cm.<sup>2</sup> for about 3 hrs., preferably at 80—85°.

L. A. COLES.

**Laminated sheet glass [permeable to ultra-violet rays].** BRIT. CELANESE, LTD. (B.P. 340,927, 15.9.30. Ger., 28.9.29. Addn. to B.P. 306,397; B., 1930, 714).—The external sheets consist of quartz glass, and the intermediate one of a composition consisting of a cellulose derivative, *e.g.*, cellulose acetate, plasticised with an organic tartrate, *e.g.*, dibutyl tartrate.

W. J. WRIGHT.

**Tempering of glass.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY, ET CREY (B.P. 340,108, 27.11.29. Fr., 13.3.29).—Sheets of glass are "tempered" or "toughened" by being first heated to 550—620° (according to composition) while suspended vertically, and then cooled rapidly by jets of air directed normally to their faces.

M. PARKIN.

**Pyrometer tubes.** F. C. SIMMS (U.S.P. 1,773,825—6, 26.8.30. Appl., [A] 4.11.27, [B] 14.7.28).—A composite tube having (A) two or (B) three layers is described, each layer having different properties and the whole being indissolubly fired together. The innermost layer may consist of doubly-fired porcelain. The next is a mixture of 30% of fused alumina, 70% of high-temperature clay, with dextrin as binder, the whole layer being subjected to only the one firing to form a layer of low coefficient of expansion and good conductivity which enables the tube to be presented to the heat quickly without cracking. The outermost layer in (B) may consist of magnesite, which is highly resistant to slags and corrosive gases.

B. M. VENABLES.

**Manufacture of refractory materials.** METALLGES. A.-G. (B.P. 340,487, 23.9.29. Ger., 23.10.28).—Granular magnesium orthosilicates that expand on being fired are mixed with powdered substances such as magnesium hydrosilicates that contract on being fired and form orthosilicates, and with powdered magnesium oxide or magnesite, the mixture being heated to a high temperature without melting.

W. J. WRIGHT.

**Clay.**—See I.

#### IX.—BUILDING MATERIALS.

**Road-making materials.**—See II. **Insulating board.**—See V.

See also A., Mar., 310, Hydrated silicates.

#### PATENTS.

**Production of a heat-conducting bond.** BABCOCK & WILCOX, LTD. FROM FULLER LEHIGH Co. (B.P. 340,868, 5.3.30).—Finely-divided carborundum is treated with an excess of sodium hydroxide solution over the

amount required to unite with the free silicon, and heated to the b.p. to expel the evolved hydrogen; the product is then mixed with sodium silicate and glycerin and, if desired, with a small amount of castor oil and iron oxide.

W. J. WRIGHT.

**Production of structural materials from organic fibrous materials, the pores of which are completely filled with high-melting pitch.** E. RUDIN and H. KOLLBRUNNER (B.P. 340,749, 13.12.29. Switz., 13.12.28).—Fibrous organic materials are impregnated with low-melting tar pitch at below 150° and then heated in an air or gas stream so that the pitch filling the pores is converted into high-melting pitch. Corrugated cardboard plates thus treated maintain their shape at 60—70°.

C. B. MARSON.

**Manufacture of tarred macadam and the like.** J. F. WAKE (B.P. 335,525, 24.4.29. Cf. B.P. 266,420; B., 1927, 334).—The sticking together of tarred aggregates during long storage or transit is prevented by coating them first with bitumen or a heavy bituminous compound, then with tar or a tar compound having about 0.5% of tarry acids and 2% of naphthalene, the latter coating comprising 20—75% of the total material used in the two coatings, and finally with a thin coating of oil or low-viscosity tar.

M. PARKIN.

**[Construction of] bituminous road surfaces.** C. G. FOX and S. STOCKELL (FOX, STOCKELL & Co.). From H. FRITSCH (B.P. 340,768, 23.12.29).

**Production of hard bituminous carpets for paving roads, streets, permanent way of railways, etc.** K. A. HALBACH (B.P. 341,016, 5.9.29).

**Increasing [by means of saw cuts] the absorption capacity of woods which are difficult to impregnate.** RÜTGERSWERKE-A.-G. (B.P. 342,786, 9.11.29. Ger., 15.12.28).

**Tar oil mixtures.**—See II. **Cellulose materials.** **Fibrous goods.**—See V. **Foundry moulds.** **Metal cement.**—See X. **Films on metal.**—See XXI.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Effect of nickel on the temperature of metastable and stable Al transformation of eutectic iron-carbon and iron-carbon-silicon alloys.** A. MERZ and F. FLEISCHER (Giesserei, 1930, 17, 817—825; Chem. Zentr., 1930, ii, 2300).—Nickel causes a depression of 12° per 1%; silicon causes a rise which diminishes with increasing quantities. Hysteresis is increased by increasing nickel and silicon contents. The existence of the graphite eutectic is affirmed.

A. A. ELDRIDGE.

**Action of oxygen on the corrosion of iron and steel in aqueous solution.** H. ENDO and S. KANAZAWA (Sci. Rep. Tôhoku, 1930, 19, 425—435).—Exposure of iron to the action of water or dilute salt solutions results in the formation of ferrous ions and a film of hydrogen on the surface of the metal. The presence of dissolved oxygen or aeration of the solution by bubbling air through it results in the removal of the hydrogen film and oxidation of the ferrous ions to ferric, followed by the deposition of rust and further corrosion of the metal owing to the formation of local elements between the

aerated and unaerated parts of the metal. Corrosion is hastened by the presence of many salts in the solution.

A. R. POWELL.

**Dependence of cold-rolling on the rate of dissolution of non-ferrous metals.** B. GARRE (Korrosion u. Metallschutz, 1930, 6, 200—201; Chem. Zentr., 1930, ii, 3190).—The dissolution velocity of pure tin, copper, and lead diminishes with the degree of rolling. The presence of small amounts of impurities in tin increases its resistance compared with pure tin, but such metal is attacked more readily than annealed metal.

A. A. ELDRIDGE.

**Chlorination roasting of copper and silver ores.** F. R. HIEMANN (Metall u. Erz, 1930, 27, 474; Chem. Zentr., 1930, ii, 2951).—A description and discussion.

A. A. ELDRIDGE.

**Age-hardening mechanism in copper-aluminium alloys.** S. KOKUBO and K. HONDA (Sci. Rep. Tôhoku, 1930, 19, 365—409).—The density, electrical resistance, and hardness of aluminium alloys containing 2—12% Cu have been determined after quenching from 545°, ageing at 15° for 10—20 days, and annealing at 60—500° for 6 hrs. The density of the quenched alloys is slightly greater than that of the forged alloys before heat treatment and is not appreciably affected by ageing below 150°, except that a slight minimum occurs at about 60°. Ageing at higher temperatures produces a marked and rapid decrease of density to a well-defined second minimum at 270°, followed by an almost equally sharp rise again to 500°, at which temperature the original density is restored. The difference between the minimum density at 270° and that immediately after quenching increases linearly with the copper content up to 6%, then decreases linearly but at a slower rate. The electrical resistance of quenched alloys is greater than that of annealed alloys (the difference increasing with the copper content), and increases during ageing at 15° to a maximum, then slowly decreases with more prolonged ageing. Rise of ageing temperature produces a slow decrease in the resistance up to 120°, then a rapid decrease to a minimum at about 350°, followed by a slight increase up to 500°. The hardness of the alloys increases on quenching and further rises during ageing at the ordinary temperature; the hardness-ageing temperature curves show a small but well-marked minimum at 60° in alloys with 2—10% Cu, a very pronounced maximum at 170° in alloys with 6—12% Cu, a less pronounced maximum at 200° in the alloy with 4% Cu, and a slight hump in the curve at 250° in the alloy with 2% Cu. Thermal analysis of quenched specimens of the 6% Cu alloy aged at 20° and aged at 150° showed four distinct phases in the resulting curves. In the first case there is a small, irregular heat evolution between 20° and 250°, a well-marked heat evolution at 250—330°, a much smaller heat evolution at 380—450°, and a marked heat absorption above 450°; in the second case heat is absorbed up to 250°, but the other phases are the same. From the above results it is suggested that there are three stages in the age-hardening of copper-aluminium alloys, viz., lattice distortion due to displacement of the copper atoms within the solid solution lattice, precipitation of the compound CuAl<sub>2</sub> from the lattice in a loosely packed state, and coagulation of

the  $\text{CuAl}_2$  with the building up of its characteristic lattice structure. These three stages overlap to some extent; the first starts at room temperature and reaches its maximum at  $150^\circ$ ; the second commences at  $60^\circ$ , reaches its maximum at  $250^\circ$ , and disappears above  $330^\circ$ ; and the third starts at  $250^\circ$ , reaches its maximum at  $350^\circ$ , and is complete at  $450^\circ$ . At temperatures above  $300^\circ$  redissolution of the compound commences and increases slowly up to above  $500^\circ$ , when it becomes rapid. This theory is shown to be in accord with the observed changes of the density, resistance, and hardness.

A. R. POWELL.

**Rock-blasting.** H. AOYAMA (J. Fac. Eng., Tokyo, 1930, 19, 25—69).—From determinations of the modulus of elasticity, density, the thermal conductivity, and Poisson's ratio for various Japanese rocks, the rate of propagation of the explosion wave with various explosives, the maximum temperature at the seat of explosion, and the range over which the effects of the explosion are felt have been calculated. The results are recorded in tables and graphs.

A. R. POWELL.

**Removal of zinc from white metal.** E. R. THEWS (Metallbörse, 1930, 20, 1433—1434; Chem. Zentr., 1930, ii, 2823).—The slag must contain at least 20%  $\text{CaO}$  in order almost completely to remove the zinc as oxide; if less lime is present the oxide remains dissolved in the slag. A suitable slag mixture for scorification in the reverberatory furnace is specified.

A. A. ELDRIDGE.

**"Chromprotekt" [covering liquid for chromium-plating baths].** B. RASSOW and L. WOLF (Chem.-Ztg., 1931, 55, 73—76).—"Chromprotekt" is a German proprietary preparation for adding to chromium-plating baths with the object of preventing the escape of poisonous chromic acid vapours or spray. It is a petroleum distillate with a trace of an ester added to disguise the odour. It was found that its use produced dull patches on the plated article, which required polishing as well as cleaning. Some reaction appeared to occur between the surface liquid and the bath, the chromic acid being gradually reduced. Though the preparation is not readily ignited itself, an electric cable moistened with it burns easily and fire risks are therefore not absent. Experiments have also shown that with its use chromic acid still passes into the air of the room to some extent, and its own vapours are not free from objection. It is, therefore, concluded that its use offers no appreciable advantage.

C. IRWIN.

**Effect of adding colloids to electrolytic solutions for preparing the anodic film on aluminium.** S. SETOH and A. MIYATA (Suppl. Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 15, No. 12, 6 pp.).—The electrolytic formation of protective films of oxide on an aluminium anode in 2% oxalic acid and also in 3% chromic acid has been studied. The addition of sodium silicate increases the anode potential and decreases the scratch-hardness of the film produced. The film obtained in the presence of sodium silicate is also less resistant to corrosion than that produced in its absence.

E. S. HEDGES.

**Dust in blast furnaces.**—See I. Corrosive solutions.—See VII. Metals for dairies.—See XIX.

See also A., Mar., 296—7, Alloys. 291 and 312, Passive iron. 324, Pure metals.

## PATENTS.

**Manufacture of irons and steels.** H. A. DE FRIES, Assr. to LUDLUM STEEL Co. (U.S.P. 1,775,339, 9.9.30. Appl., 27.4.28).—The use of the compound  $\text{FeAl}_3$  as a deoxidiser for iron and steel is claimed. The substance is added to the molten metal, in amount slightly in excess of that required to reduce the oxides present, just before the desired alloying elements are added.

A. R. POWELL.

**Manufacture of iron and steel articles which are subjected during manufacture to the action of a testing load.** VEREIN. STAHLWERKE A.-G. (B.P. 342,378, 6.11.29. Ger., 31.12.28. Addn. to B.P. 197,330; B., 1924, 627).—The testing load is applied at least partly by shocks or vibrations set up by the rotation of an unbalanced mass with suitable coupling or by an electrically operated vibration system.

A. R. POWELL.

**Production of [iron] welding wires.** VEREIN. STAHLWERKE A.-G. (B.P. 343,418, 5.8.30. Appl., 6.8.29).—The wires are made from the upper part of the ingot containing the shrinkage holes and slag inclusions.

A. R. POWELL.

**Metal-hardening processes. [Case-hardening of iron and steel.]** W. FAIRWEATHER. From SINGER MANUFG. Co. (B.P. 342,940, 18.3.30).—The parts of the article which are not to be case-hardened are electroplated with a thin layer of chromium and the article is then subjected to the usual case-hardening process.

A. R. POWELL.

**Galvanising sheet metal.** B. P. FINKBONE, Assr. to AMER. ROLLING MILL Co. (U.S.P. 1,776,852, 30.9.30. Appl., 30.11.25).—The use of pure zinc as a galvanising metal is claimed; the steel sheet to be galvanised passes into the molten zinc through a pair of rolls working in a box-like compartment filled with a zinc ammonium chloride flux. The rolls are kept clean by means of a layer of abrasive material, e.g., sand, floating on the metal surface below the layer of flux.

A. R. POWELL.

**Pickling composition [for steel].** H. J. JOHN, Assr. to N. A. EMERY (U.S.P. 1,776,858, 30.9.30. Appl., 2.4.27).—A pickling solution containing 2—20% of a 2:1 mixture of hydrochloric and nitric acids with 2—5% of sodium chloride is claimed. To prevent spray being thrown off the surface of the bath the latter is covered with a layer of sawdust containing palladium sponge to absorb the hydrogen generated.

A. R. POWELL.

**[Inhibitor for] the pickling of metals and like processes.** IMPERIAL CHEM. INDUSTRIES, LTD. From GRASSELLI CHEM. Co. (B.P. 342,601, 4.4.30).—The inhibitor claimed is a thiuram sulphide, especially tetramethylthiuram disulphide.

A. R. POWELL.

**Casting of steel and other metals.** A. G. E. HULTGREN (U.S.P. 1,725,859, 16.9.30. Appl., 28.7.28. Swed., 8.8.27).—The molten metal is cast into a mould, and after it has solidified round the sides of the mould and crystals have begun to form spontaneously from independent nuclei in the middle of the casting the mould itself is agitated or the still molten metal stirred to cause



the middle portion of the casting to crystallise with a fine-grained crystal structure. A. R. POWELL.

**[Heat treatment of] cast iron-alloy articles.** **[Cast iron] metal for casting purposes.** MIDLAND MOTOR CYLINDER CO., LTD., P. PRITCHARD, and T. W. PICKEN (B.P. 342,334—5, 28.10.29).—(A) Articles made of the cast iron claimed in (B) are annealed at 680°, cooled slowly to render them workable, machined to the desired shape, and then hardened by annealing at 780—820° and cooling in air. (B) The alloy comprises 3% C, 4.35—6 (4.5)% Ni, 1.6—1.9 (1.75)% Cr, 1% Mn, and 1.6—2.5 (1.8)% Si, and the remainder (88%) iron. A. R. POWELL.

**Preparation of magnetic [soft iron] materials.** ELECTRICAL RES. PRODUCTS, INC., Assees. of P. P. CIOFFI (B.P. 342,360 and 342,676, 29.10.29. U.S., 13.12.28).—(A) Armco iron is heated for at least 30 min. at 1500° in a 3:7 mixture of hydrogen and nitrogen, cooled rapidly to 20°, reheated at 880° for 1 hr., and cooled slowly to 20°. (B) Iron or an iron alloy with more than 95% Fe is melted in an atmosphere of hydrogen and the pressure of hydrogen is gradually increased during cooling until it reaches at least 10 atm. at 880°. The metal is then cooled rapidly, re-annealed in hydrogen at 880—1000°, and allowed to cool slowly. A. R. POWELL.

**[Iron-nickel-cobalt] magnetic alloys.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELECTRICITÄTS GES. (B.P. 342,982, 24.5.30. Ger., 25.5.29).—Alloys containing 20—50% of iron, nickel, and cobalt and 2.5% Si are claimed, especially that produced by adding 2.5% of silicon to an alloy of 45% Ni, 30% Fe, and 25% Co. A. R. POWELL.

**Production of ferrotitanium alloys.** W. and II. MATHESIUS (B.P. 343,007, 5.11.29).—The alloys are prepared by aluminothermic reduction of a mixture of ilmenite and pyrolusite so proportioned as to give an alloy containing about 30% Ti, 15—20% Mn, and 55—50% Fe. A. R. POWELL.

**[Chromium-]alloy steel.** J. A. SUCCOP, Assr. to HEPPENSTALL CO. (U.S.P. 1,775,615, 9.9.30. Appl., 12.6.30).—Non-rusting razor blades are prepared from a martensitic steel containing 0.5—2% C, 5—20% Cr, 0.1—1.75% Mn, 0.1—2% Mo, 0.25—3.5% Ni, 0.1—2% Si, and 0.1—1.5% V. A. R. POWELL.

**Manufacture of malleable iron-chromium alloys.** P. R. KUEHNRICH (B.P. 342,295, 25.10. and 23.11.29).—Malleable chromium steels highly resistant to chemical action contain 28—48% Cr and 0.2—1.5% C; the alloy with 33% Cr and 0.33% C is specifically claimed. Such alloys are unhardenable by heat-treatment and may be forged and rolled either hot or cold. A. R. POWELL.

**Manufacture of a tin-coated chromium-iron alloy.** L. SCHULTE, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,776,603, 23.9.30. Appl., 25.5.26).—A low-carbon steel with 9—16% Cr is rendered passive by immersion for a few minutes in concentrated nitric acid, plated with tin in a fluoride bath, and heated at 450—600° or immersed in a molten nitrate bath for a few minutes to render the tinplate bright and firmly adherent. A. R. POWELL.

**Manufacture of [silicon-iron-copper] alloys.** U. DE BERKER, W. MACHIN, and W. B. O'B. Goudielock. Assrs. to P.M.G. METAL TRUST, LTD. (U.S.P. 1,777,192, 30.9.30. Appl., 5.3.30. U.K., 12.12.29).—Hard copper alloys having the properties of a high-grade bronze are obtained by the addition to copper of 1.5—5% Si, 0.2—11% Fe, and 0—1% P. The additions are made in the form of a hardener alloy containing 30—70% Fe, 60—20% Cu, 10—50% Si, and 0.1—10% P. A. R. POWELL.

**Sulphur-resisting alloys [of iron, cobalt, or nickel].** HERAEUS VACUUMSCHMELZE A.-G., and W. ROHN (B.P. 342,868, 13.1.30).—Addition of at least 9% Al to nickel, cobalt, or iron alloys containing manganese and/or chromium is claimed to render them inert to the action of atmospheres containing sulphur at temperatures up to 1000°. [Stat. ref.] A. R. POWELL.

**[Chloridising roasting] treatment of [sulphide] ores.** T. P. HOLT (U.S.P. 1,775,767, 16.9.30. Appl., 11.7.28).—The ore is subjected in a relatively coarse form to a chloridising roast with salt in a blast roasting apparatus and the hot roasted product is quenched in a dilute salt solution obtained from a subsequent stage of the process. The leached residue, which is relatively porous, is mixed with more salt and fuel and again blast-roasted and leached as before; this procedure is repeated until the maximum economic extraction is obtained, leach liquors obtained in the second and subsequent roasting operations being used for quenching and leaching later charges at an earlier stage of the process. A. R. POWELL.

**Pyrometallurgical copper refining.** H. H. STOUT (B.P. 317,399, 8.8.29. U.S., 15.8.28).—Copper is oxidised at 1110° until it is converted into the copper-cuprous oxide eutectic mixture, and is then transferred to a refractory-lined chromium-steel reduction furnace preheated at 1220° in which the oxide is reduced by means of carbonaceous fuel or sulphur-free reducing gases without the aid of external heat. A. R. POWELL.

**Obtaining metals [copper, lead, and silver] from bituminous [shale] ores.** C. GOETZ (B.P. 342,293, 23.10.29).—The ore, e.g., Mansfield copper shale, is subjected to destructive distillation in a horizontal rotary retort at 600—700°, whereby the copper, silver, and lead compounds are reduced to metal which is subsequently recovered by fine-crushing of the residue and concentration on high-speed percussion tables. The concentrate is subsequently refined electrolytically. The addition of about 1% of gypsum to the shale is claimed to result in an improved extraction. A. R. POWELL.

**[Phosphor-copper brazing] solders.** ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of O. W. ELLIS (B.P. 342,592, 25.3.30. U.S., 26.3.29).—An alloy of copper with 2.5—6% P is repeatedly rolled at 350—650° until the thickness has been gradually reduced to 0.04—0.021 in., when the alloy can be cold-rolled to 0.015—0.01 in. without fracture. This solder melts at a lower temperature than the usual brazing solder containing zinc and/or silver, and is especially suitable for use in joining copper or monel metal parts. A. R. POWELL.

**Production of sheet-like metal grains [for bronze powders].** HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 341,561, 22.10.29. Ger., 22.10.28).—The particles of metal powders are flattened in a steel ball mill through which is passed a current of inert gas which blows out the finished and partly-finished flattened leaflets of metal into a gas elutriator in which the latter fall to the bottom, and thence to a second mill, where the flattening process is finished. The discharge from the second mill is blown by means of the gas current into the top of the elutriator, so that the heavier particles are again returned to the second mill.

A. R. POWELL.

**Aluminium alloy.** H. C. HALL and T. F. BRADBURY (B.P. 342,729, 6.11.29. Addn. to B.P. 323,353; B., 1930, 198).—The iron and the nickel contents of the alloy claimed in the prior patent are kept within the limits 0.2–0.6 and 1.5–2.5%, respectively, and magnesium is omitted or kept below 0.03%.

A. R. POWELL.

**Zinc-aluminium alloy.** H. C. HALL (B.P. 343,320, 24.2.30).—An alloy containing 1–3.5% Cu, 0.1–1.5% Fe, 0.1–1.5% Si, 35–65% Al, and the remainder zinc is claimed. Up to 1% of manganese, magnesium, titanium, molybdenum, vanadium, and/or sodium may be added as a scavenger and to refine the grain size.

A. R. POWELL.

**[Aluminium-copper] alloy.** A. G. KRAUS, Assr. to J. S. KIRSTEIN (U.S.P. 1,776,948, 30.9.30. Appl., 26.7.29).—An alloy having the tensile strength of steel, a colour resembling that of gold, and a high resistance to tarnishing in the atmosphere comprises copper with 0.5–1% Sn, 1% Ni, 5% Al, and 0.1–1% Be.

A. R. POWELL.

**Production and refining of magnesium-cerium alloys.** I. G. FARBENIND. A.-G. (B.P. 342,586, 21.3.30. Ger., 15.4.29).—The alloys are melted under a flux of magnesium chloride and/or carnallite, to which is added anhydrous cerium chloride in amount such that the flux contains twice the percentage of cerium present in the alloy.

A. R. POWELL.

**Protection of aluminium, magnesium, or their alloys against the action of corrosive agents.** C. BOULANGER (B.P. 342,256, 24.10.29. Fr., 10.11.28).—The articles are boiled or anodically oxidised in a bath containing about 1% of sodium carbonate and about 0.5% of a soluble salt of a metal which forms at least two oxides, the higher of which is an acid-forming oxide and the lower is insoluble in alkali solutions. Sodium manganate, vanadate, tungstate, molybdate, uranate, and niobate are specifically claimed.

A. R. POWELL.

**Combining aluminium or its alloys with other metals or alloys by welding.** A. R. and J. M. HETZEL (VEREIN. SILBERHAMMERWERKE HETZEL & Co.), Assces. of H. WACHWITZ (B.P. 343,156, 15.11.29. Ger., 17.11.28).—The metal to be attached to the aluminium is first coated with a thin layer of a readily fusible alloy of aluminium with cadmium, bismuth, tin, or zinc, by means of heat and pressure, and the aluminium or aluminium alloy is then attached to this layer in the usual way.

A. R. POWELL.

**Production of beryllium.** H. S. COOPER, Assr. to BERYLLIUM CORP. OF AMERICA (U.S.P. 1,775,589,

9.9.30. Appl., 8.8.28).—The crystals of beryllium obtained by electrolysis of a fused mixture of sodium and beryllium chlorides are pressed into coherent rods, discs, or the like, and these are melted under molten barium chloride with or without sodium chloride.

A. R. POWELL.

**Production of sodium and potassium.** J. YLLA-CONTE (B.P. 342,609, 25.4.30. Spain, 27.4.29).—Sodium or potassium hydroxide or carbonate is heated with carbon under reduced pressure and the evolved gases containing the metal vapour are passed through two cooling chambers; in the first of these the metal is condensed to the liquid form, and in the second the gases are cooled to reduce their volume and so facilitate the work of the pump.

A. R. POWELL.

**Moulding processes [for foundries].** J. B. DURAND (B.P. 319,777, 23.9.29. Fr., 29.9.28).—Moulds and cores for use in metal casting comprise sand and fine cement mixed in suitable proportions with water; the mixture is rammed lightly into the mould box and, after setting, may be painted with cement and water or with black water-paint.

L. A. COLES.

**Metal cement.** W. R. CHAPIN (U.S.P. 1,775,670, 16.9.30. Appl., 29.4.29).—The cement comprises a mixture of 5–10% of powdered borax glass, 10% of ferrosilicon (15% Si), and 75–80% of silicomanganese (70% Mn, 20% Si, and 10% Fe). The material is used for joining iron parts together by heating them in contact with the cement at 1275°.

A. R. POWELL.

**Removing oil from metal chips etc.** H. N. SKERRETT. From TIMKEN ROLLER BEARING CO. (B.P. 340,992, 2.10.29).—Used oil is recovered by passing metal turnings etc. on a conveyor belt under a spray of hot water and/or steam and collecting and separating the oil-water mixture which drips from the mass.

C. A. KING.

**Electroplating articles with chromium.** A. BUTZIGER (B.P. 342,477, 24.12.29).—Small articles (screws, nuts, etc.) are plated in a cradle which is rocked so that at each swing it is partly lifted out of the plating bath. The cradle is so coupled with the anodes that the relative proportions of each which are lifted out of the bath are maintained constant throughout the operation.

A. R. POWELL.

**Electrodeposition of metals and alloys.** L. L. LINICK (U.S.P. 1,777,121, 30.9.30. Appl., 3.11.27).—Hydrogen under pressure is passed into the electrolyte through a tubular anode. It is claimed that in this way bright, fine-grained deposits may be obtained from, e.g., chromium-plating baths of much lower concentration than the normal at relatively low current densities and voltages.

A. R. POWELL.

**Preparation of metals for painting.** J. H. GRAVELL (B.P. 342,661, 2.11.29. U.S., 23.5.29).—See U.S.P. 1,765,331; B., 1931, 165.

**Superficial ornamentation for wood, metal, etc.** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 343,168, 16.11.29. Ger., 25.1.29. Addn. to B.P. 319,006).

**Metallised sheets of paper etc.**—See VI. Light-sensitive films on metal etc.—See XXI.

## XI.—ELECTROTECHNICS.

"Chromprotekt" for chromium-plating baths. Films on aluminium.—See X.

See also A., Mar., 329, Spectrophotometry. 330, Glow discharge on gases. 389, Therapeutic electrodes.

## PATENTS.

[Heat-conducting] metal-vapour arc lamps. SIEMENS & HALSKE A.-G. (B.P. 343,110, 10.10.29. Ger., 11.10.28).

Luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 343,053, 7.11.29. Ger., 12.7.29).

Solids in liquids.—See I. Pure gases. By-products from gas. Insulating oil.—See II. Cellulose by-products.—See V. Bleaching.—See VI. Ammonia.—See VII. Case-hardening. Magnetic materials. Tin-coated iron. Metals from ores. Protection of metals. Beryllium. Electrodeposition.—See X. Insulating material.—See XIII. Vitamins.—See XIX. Antirachitic substances.—See XX.

## XH.—FATS; OILS; WAXES.

Detection of less than 0.5% of sesamé oil in mixtures of solid fats. R. LUCENTINI (Annali Chim. Appl., 1931, 21, 82—83).—50 G. of the fat are extracted for about 2 hrs. with 100 c.c. of 95% alcohol, the extract being allowed to cool and then filtered. The alcohol is evaporated on a water-bath and the cold residue dissolved in 5 c.c. of light petroleum and treated with 2 drops of alcoholic furfuraldehyde solution and 5 c.c. of concentrated hydrochloric acid solution. After the test-tube has been gently inverted several times, the liquid assumes a rose-red colour in presence of a very small proportion of sesamé oil. T. H. POPE.

Determination of the fat content of oilseeds. M. MONHAUPT (Chem.-Ztg., 1931, 55, 70).—The well-crushed seeds (4 g.) are placed in a thick, round-bottomed, glass flask (80 c.c.) with 5 or 6 steel balls (10—12 mm. diam.) and 10—12 g. of sand. The flask is tightly corked and shaken violently for 1—2 hrs. After settling, an aliquot portion (20 c.c.) of the clear solution is pipetted off and evaporated to determine its oil content ( $f$ ); correcting for the volume of the oil contained in the solution, the percentage of oil ( $x$ ) in the seed is given by  $x = 1000f / (20 - 1.0753f)$ .

E. LEWKOWITSCH.

Linseed oil. I. Apparent oxygen-absorption curve. E. YAMAGUCHI (Mem. Fac. Sci. Eng., Waseda, 1930, No. 7, 144—146).—Experiments made over periods ranging up to 1200 hrs. confirm that the absorption of oxygen by varying films of linseed oil contained in a thermostat at 30° tends to continue indefinitely. The reaction is assumed to be mainly unimolecular and is accompanied by the evolution of volatile matter containing water, oxides of carbon, aldehydes, and volatile acids, part of this volatile matter being liberated as gas under the conditions of experiment, while part accumulates on the surface of the linseed oil film.

Experimental results confirm a mathematical expression formulated to show the relation between time and the apparent oxygen absorption, the latter being expressed as an exponential form of the time of exposure with a correction term for the volatile matters formed.

E. A. RYDER.

Preparation of soluble oil [Turkey-red oil]. C. SUNDER (Sealed Note No. 2595, 30.10.24. Bull. Soc. Ind. Mulhouse, 1930, 96, 635—636). Report by T. VOLTZ (*Ibid.*, 636—642).—An increased yield of the true sulphuric ester of castor oil may be obtained in the sulphonation of this oil by means of chlorosulphonic acid (cf. G.P. 260,748; B., 1913, 798) if sufficient of a tertiary base (pyridine or dimethylaniline) is present to form an additive compound with the chlorosulphonic acid; an excess of the base does not retard the sulphonation. In elaborating Sunder's process, VOLTZ was able to confirm the initial formation of the trisulphonic ester of castor oil,  $C_3H_5(O \cdot CO \cdot C_{17}H_{32} \cdot O \cdot SO_3Na)_3$ , and from this he prepared the corresponding sulphonated ricinoleic acid by saponification with caustic soda and the ethyl ester of ricinoleic acid by further treatment with alcoholic hydrochloric acid. The trisulphonic ester of castor oil yielded duller shades than those given by ordinary sulphonated castor oil when used for oiling fabric which was afterwards dyed with alizarin. It is concluded that sulphonated castor oil products decrease in value as a mordant for alizarin as the proportion of fixed sulphuric acid increases. A. J. HALL.

New oils. E. STOCK (Farben-Ztg., 1931, 36, 830).—The analytical constants of samples of seed oil from *Moquilia tomentosa*, Benth., *Enocarpus* oil, Udilo seed oil, and of two fish oils are tabulated. S. S. WOOLF.

Fatty acids in mineral oil. Stearine etc.—See II. Water in glycerols.—See III.

See also A., Mar., 335, Linolenic acid and esters. 380, Acids of cod-liver oil and lard. 401, Soya-bean lecithins.

## PATENTS.

Separation of impurities from oils and fats. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,390, 5.7.29).—The fats are emulsified with a small amount (0.05—10%) of neutral or acidic immiscible liquids which have no chemical action on the impurities, e.g., water, solutions of electrolytes, formamide, acetic acid, emulsifying agents, such as soap, etc., and small quantities of finely-divided solids, e.g., charcoal, being added as desired; the emulsion produced is then separated mechanically by filtration or centrifuging, and the bright, slime-free oils obtained may be deacidified, if desired, by any physical method, e.g., treatment with a mist of steam. E. LEWKOWITSCH.

Manufacture of (A—C) derivatives of higher fatty acids (A) containing nitrogen, (C) containing sulphur. A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 339,675, 340,011—2, 11.9.29).—Polyhalogenated fatty acids above  $C_8$ , their esters or amides, prepared according to B.P. 336,623 (B., 1931, 149) are treated (A) with alcoholic ammonia under pressure at 120—150°; (B) with aqueous or alcoholic caustic alkali or aqueous weak alkalis (ammonia, sodium carbonate, phosphate, acetate,

ammonium formate, urea); (c) with solutions of sulphides or polysulphides. In each case part or all of the halogen is removed. (c) The products of (A) are similarly treated with sulphides. Examples are: (A) hexachlorinated ricinoleic or stearic acid, or chlorinated linoleic acid with alcoholic ammonia at 140–150° to give soap-like products; (B) hexachlorinated ricinoleic acid with aqueous potassium carbonate at 140–150° to give a rubber-like mass, or with aqueous urea at 110–120° to give a sticky rubber-like product; tetrachlorinated oleic acid with 20% sodium hydroxide at 120–125° to give an unsaturated hydroxy-acid containing chlorine; ethylanilide of tetrachloro-oleic acid with alcoholic potassium hydroxide under reflux to give an unsaturated viscous oil; (c) trichloropalmitic acid with aqueous sodium sulphide at 120–125°; pentachlorostearic acid treated with sodium hydroxide and then with sodium sulphide.

C. HOLLINS.

**Treatment of drying oils.** BRIT. THOMSON-HOUSTON Co., LTD., and W. J. SCHEIBER (B.P. 319,218, 17.9.29. U.S., 17.9.28).—The conversion of drying oils by continued heat-treatment successively into boiled, gelled, and de-gelled oils can be greatly accelerated by the addition to the oil of about 1% of certain amines, *e.g.*, benzidine, diphenylamine, *m*- or *p*-phenylenediamine, diisopropylamine.

E. LEWKOWITSCH.

**Recovery of oils, fats, and waxes from adsorptive materials. [Purification of bleaching earths.]** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,394, 6.9.29).—The oily absorptive material, *e.g.*, bleaching-earth residues from the filter-presses, is treated at above 70° with at least 50% of water (or other non-solvent, *e.g.*, glycerin) and with a suitable powdered solid material, *e.g.*, bran, zinc blende, flowers of sulphur (up to about 20% of the earth present), or active carbon (not more than 15%), which is more easily wetted by the oil than is the earth; emulsifying agents, *e.g.*, soaps, may also be used. The earth separates in a practically oil-free condition. The additional solid chosen is preferably one with a low adsorptive power for oil, and may be incorporated with the absorptive earth etc. before the latter is used in the oil refining. The water may be replaced by inert vapours or gases, *e.g.*, steam or nitrogen.

E. LEWKOWITSCH.

**Cleansing agents.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,232, 17.8.29).—A cleansing paste for removing copying ink etc. from the hands etc. consists of a cellulose ether swollen in water, *e.g.*, methyl cellulose containing 85% of water (80–100 pts.) mixed with organic solvents, *e.g.*, ethylene glycol ethyl ether (50–60 pts.). Soap and glycerin (*e.g.*, 40 and 10 pts., respectively) may be added.

E. LEWKOWITSCH.

**Non-freezing lubricants.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,294, 27.7.29).—Lubricants of various consistencies are prepared by emulsifying fatty and mineral oils etc. with glycol or a hydroxyl-bearing ether thereof, *e.g.*, ethylene glycol monoethyl ether, by the help of a dispersing agent, *e.g.*, Turkey-red oil. Diluents, *e.g.*, alcohols, acetone, and small quantities of metallic salts such as calcium chloride, or alkaline materials may be added.

E. LEWKOWITSCH.

**Sulphonic acids.**—See II. **Polymerised compounds.** **Auxiliary agents.** **Basic products.**—See III. **Stencil sheets.**—See V. **Hormone preparations.**—See XX.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Preparation of precipitated and fused driers [for paints etc.].** A. W. C. HARRISON (Farbe u. Lack, 1931, 16–17).—Precipitated driers prepared from sodium linoleate or resinate and solutions of neutral metal salts are pure and free from colour changes, and are entirely soluble in hot turpentine, benzene, etc. Fused driers do not always give a stable solution, and are frequently dark in colour due to prolonged heating. Complete details of five methods of preparation are given, three dealing with precipitated and two with fused driers.

F. C. HARWOOD.

**Preparation of precipitated white lead of high opacity.** A. I. KOGAN (Farben-Ztg., 1931, 36, 826–828).—The physical and chemical properties of the white pigments obtained by passing carbon dioxide under controlled conditions through basic lead acetate solutions of various concentrations were determined. It is shown that as the reaction proceeds to the formation of excess of carbonate (over the requisite amount for white lead) the opacity falls. A further series of experiments, in which the pigment was precipitated progressively by passing successive quantities of carbon dioxide through a solution containing a limited amount of lead showed that the opacity of the composite pigment prepared in this way rises to a maximum with the number of precipitations. Full experimental details and results are tabulated, and the degrees of dispersion are graphed and illustrated photomicrographically. The sp. gr. of these precipitated white leads is somewhat low, *viz.*, 5.95–6.25 (cf. the usual figure for white lead, 6.5–6.8).

S. S. WOOLF.

**Reaction of inorganic basic pigments with linseed oil.** K. CHARISIUS and E. KINDSCHER (Farben-Ztg., 1931, 36, 780–783).—Zinc oxide, basic carbonate and sulphate white leads, “disperse” and “normal” red leads, and litharge were mixed with raw linseed oil and stored with vigorous daily agitation in completely filled glass-stoppered bottles for some months, a “blank” being run on the oil itself. The supernatant oil and the oils extracted from the sediment by ether and by 9:1 benzol-alcohol mixture were examined. Whilst zinc oxide and white leads of both types have little reactive power in these circumstances, litharge reacts to a marked degree, the oil assuming a dark red-brown colour and a heavy sediment being formed, approx. 9% of the original lead content being obtained from the supernatant and extracted oils. Of the red leads, the effect of the litharge content of the “normal” variety outweighs that of the fine state of subdivision of the “disperse” kind, the former reacting to a much greater extent than the latter, which has the same order of reactivity as white lead, less than 1% of lead being taken up. About 4% of the “normal” red lead goes into solution. The acid values and the oxyacid contents of the oils receive consideration. It was found, in general, that the results obtained in the absence of air

were in agreement with those previously obtained (cf. Kindscher, B., 1930, 337) in the presence of air.

S. S. WOOLF.

**Errors in manufacture and use of nitro-lacquers.** HÜTTER (Farbe u. Lack, 1931, 5, 15).—Deterioration of nitro-lacquers on storage is traceable to errors in use or, more often, in manufacture. Choice of solvent is most important, as too rapid drying cools the lacquer film with possible condensation of moisture; nitro-lacquers are particularly sensitive to changes in humidity and cloudy films often result. There should therefore be a sufficient admixture of higher-boiling solvents. Evaporation from badly closed containers alters the viscosity, and as the viscosity of the nitrocellulose itself varies, it is important to determine this and to control the addition of solvent by sp. gr. measurements and not by volume. The percentage ratio of nitrated cotton to solvent should be ascertained. During manufacture, room temperature and humidity of the air must be regulated, whilst in use, quick drying at a comparatively high temperature followed by long cooling is often advantageous.

F. C. HARWOOD.

**Opaque colours and nitro-lacquers for the leather industry.** R. KRAUS (Farbe u. Lack, 1931, 27—28).—Methods of application of the coatings by spraying are reviewed and criticised, and means whereby the adherence and elasticity of the pigment film and its resistance to low temperature may be tested are described.

F. C. HARWOOD.

**Constitution of artificial resins.** G. WALTER (Z. angew. Chem., 1931, 44, 136—137).—A preliminary account, without details, of an examination of the urea-formaldehyde condensation.

**Water-gas tar.**—See II.

See also A., Mar., 380, "Hydrax."

#### PATENTS.

**Production of zinc sulphide pigments.** A. and L. WRESCHNER (B.P. 340,808, 20.1.30. Ger., 25.1.29).—Zinciferous material is heated with highly concentrated sulphuric acid before leaching with water. For precipitating the purified zinc sulphate solution, barium sulphide is wholly or partly replaced by ammonium or sodium sulphide, and a small amount of a compound of boron or other light metal is added during the precipitation to increase the fastness to light of the pigment. Access of oxygen during calcination must be prevented by the introduction of nitrogen or carbon dioxide into the furnace.

W. J. WRIGHT.

**Production of composite pigments containing titanium dioxide.** J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (U.S.P. 1,766,592, 24.6.30. Appl., 13.3.28).—Pigments suitable as substrata, e.g., silica, barium or calcium sulphate, lithopone, are agitated with an aqueous colloidal dispersion of titanium dioxide (e.g., one containing a peptising quantity of a suitable acid such as hydrochloric acid), a coagulant, e.g., sulphuric acid, is added, and the composite pigment thus formed is filtered, washed, and calcined at 900—1000°.

S. S. WOOLF.

**Manufacture of coloured lacquers.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,936, 8.8.29).—

Cellulose ester or ether lacquers are coloured with the alkaline-earth (calcium) salts of sulphonated azo dyes of the type: nitroarylamine  $\rightarrow$  acetoacetic arylamide. Examples are: acetoacetic sulpho-*o*-anisidide coupled with diazotised 5-nitroanthranilic acid, *o*-nitroaniline, or 5-nitro-*o*-toluidine- $\omega$ -sulphonic acid.

C. HOLLINS.

**[Coating] compositions containing organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 342,144, 1.2.30. U.S., 5.2.29).—Arylsulphonamide-aldehyde resins, e.g., benzene-, toluene-, xylene-sulphonamide-formaldehyde resins, are particularly suitable for use in coating compositions, lacquers, varnishes, or adhesives which contain an organic derivative of cellulose dissolved in a vehicle containing *inter alia* a glycol ether or a derivative thereof.

D. J. NORMAN.

**Manufacture of urea-formaldehyde condensation products.** A. V. KELLER (B.P. 340,372, 13.11.29).—Urea and a solid polymeride of formaldehyde, e.g., paraformaldehyde, are mixed in the dry state with a small percentage of an alkaline catalyst, e.g., sodium hydroxide, saturated steam is passed into the dry mixture, and an acid catalyst, e.g., acid sodium phosphate, is added, the whole being heated to complete the reaction.

S. S. WOOLF.

**Production of cloudy phenol-formaldehyde condensation products.** HEROLD A.-G., and A. JAEGER (B.P. 341,471, 12.10.29).—Esters of aromatic compounds containing a hydroxyl group, e.g., phenol, naphthol, anisyl alcohol, with sulphuric or phosphoric acids, or with such compounds as 1-isopropynaphthalene-4-sulphonic acid, benzenesulphonic acid, etc., are added to the resin, preferably at a time when the distillation for the removal of water is almost completed. Cloudiness develops at an advanced stage in the hardening of the resin.

E. LEWKOWITSCH.

**Manufacture of synthetic resins.** E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,771,508, 29.7.30. Appl., 14.8.26).—A phenol is treated with formaldehyde and furfuraldehyde in the absence of a catalyst at a temperature above the b.p. of the mixture, water is distilled off, and the resin formed is heated until the desired viscosity is attained.

S. S. WOOLF.

**Artificial resins.** I. G. FARBENIND. A.-G. (B.P. 315,835, 1.7.29. Ger., 19.7.28).—The use of not more than 10% of a cellulose ether (other than an alkyl ether) as softening agent for artificial resins derived from phenol and capable of being hardened, e.g., phenol-formaldehyde resins, is claimed.

S. S. WOOLF.

**Artificial resins.** BRIT. THOMSON-HOUSTON CO., LTD., Assces. of R. H. KIENLE (B.P. 317,797, 22.8.29. U.S., 22.8.28).—Glycol or other dihydric alcohol is used in addition to alcohols of higher hydroxyl content in the preparation of "alkyd" condensation products. [Stat. ref.]

S. S. WOOLF.

**Recovery of utilisable products from residues obtained from distillation of turpentine oil, gum resins, and like resinous substances.** E. D. FELDMAN (B.P. 340,338, 4.10.29).—Such residues, with which a catalyst, e.g., finely-divided nickel, has been admixed, are distilled at 100—200° in a current of hydrogen under

any desired pressure, or treated with hydrogen and subsequently distilled, high-grade turpentine oil being obtained.

S. S. WOOLF.

**Manufacture and use of [resinous] insulating material.** RÖHM & HAAS A.-G. (B.P. 340,677, 28.10.29. Ger., 26.10.28).—Mixtures of polymerides of acrylic acid or of its homologues, or derivatives of these, *e.g.*, esters, with mica, paper, polymerised oil, linolyn, etc. are claimed.

S. S. WOOLF.

**Polymerised compounds. Storage of liquids.**—See III. Tartrazine dyes. Colour lakes. Pigment dyes.—See IV. Cleansing agents.—See XII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Early stages of oxidation in rubber. Quantitative application of the pyrrole test.** J. W. TEMPLE, S. M. CADWELL, and M. W. MEAD, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 377—380).—A sample of aged rubber (*e.g.*, 1 g. in small pieces) is digested with fused ammonium acetate (2—3 g.) for several minutes; the mixture is then distilled with steam and the ether extract of the distillate is treated with an alcoholic pine-wood extract to which hydrochloric acid has been added. The ensuing red colour is compared with a set of permanent standards. This test detects the beginning of the ageing process at a much earlier stage than the increase in the acetone-soluble matter or the decrease in tensile strength. The results suggest a difference in character between ageing in compressed oxygen, and in air at higher temperatures, natural ageing falling between the two. It is believed that lævulaldehyde, or similar substance, which is responsible for the test, is formed at an early stage, and that in the subsequent course of physical deterioration, especially when aided by heat, it undergoes gradual conversion into products which do not give the pyrrole reaction.

D. F. TWISS.

**Preparing micro-sections of rubber.** T. F. STEELE (Ind. Eng. Chem. [Anal.], 1930, 2, 421—422).—The specimen, approx.  $8 \times 2 \times 0.5$  mm., is placed between two smooth blocks of bass-wood,  $30 \times 30 \times 12$  mm., which have been thinly coated with rubber solution on the contacting surfaces and dried, in such a position that the sections will be cut parallel to the grain. After pressing in a vice for several hours, the wooden mount is buffed on a grinding wheel so as to leave a projecting tongue of wood holding the rubber. The sections are then cut after liquid air has been applied to the sample by means of a brush.

D. F. TWISS.

See A., Mar., 357, Pyrolysis of rubber.

#### PATENTS.

(B) **Manufacture of compounds [accelerators] for treating rubber etc. and (A) treatment of rubber therewith.** L. MELLERSH-JACKSON. From NAUGATUCK CHEM. Co. (B.P. 340,573—4, 1.7.29).—(B) A nitroaryl compound containing reactive halogen or an aryl- or alkyl-substituted methyl halide is condensed with a dithiocarbamate, xanthate, or arylthiocarboxylate, to give rubber accelerators. (A) Compounds such as are obtainable according to (B) are used in rubber mixes as accelerators. Examples are products from: (A, B) 1-chloro-2 : 4-dinitrobenzene and sodium dimethyl- (m.p.

151—152°), diethyl- (m.p. 81°), di-*n*-propyl- (m.p. 84°), diisobutyl- (m.p. 85°), di-*n*-butyl-, diisomyl- (m.p. 52—54°), dibenzyl- (m.p. 106°), morpholine- (m.p. 125—130°), and dicyclohexyl- (m.p. 127°)-dithiocarbamates; benzylidene chloride and 2 mols. of sodium dimethyldithiocarbamate (m.p. 181—182°); triphenylmethyl chloride and sodium dimethyldithiocarbamate (m.p. 150—154°); 1 : 4-dichloro-2 : 6-dinitrobenzene and sodium diethyl- (m.p. 123°) and dimethyl- (m.p. 123°)-dithiocarbamates; benzotrichloride and 2 mols. of sodium dimethyl- (m.p. 110°) and diethyl- (m.p. 176—177°)-dithiocarbamates; allyl chloride and sodium diethyldithiocarbamate (m.p. 110—111°); triphenylmethyl chloride and sodium ethylxanthate (m.p. 138°); (B) 1-chloro-2 : 4-dinitrobenzene and 2-thiolbenzthiazole (m.p. 167°), and other products of B.P. 340,951 (*cf.* following abstract).

C. HOLLINS.

**Manufacture of compounds [accelerators] for treating rubber and the like.** L. MELLERSH-JACKSON. From NAUGATUCK CHEM. Co. (B.P. 340,951, 1.7.29. *Cf.* B.P. 340,574; preceding).—2-Thiolbenzthiazole salts are condensed with aromatic nitro-compounds containing reactive hydrogen or with an arylated or alkylated methyl halide. Examples are: products from 1-chloro-2 : 4-dinitrobenzene (m.p. 162.5°), picryl chloride (m.p. 152°), 1 : 4-dichloro-2 : 6-dinitrobenzene (m.p. 167°), allyl bromide (b.p. 145—148°), diphenylmethyl chloride (m.p. 108°). The products from the two last-mentioned compounds are poor accelerators; the others accelerate vulcanisation.

C. HOLLINS.

**Manufacture of [rubber] tyres for vehicles.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,681, 30.10.29).—For the production of the separate parts of tyres, polymerisation products of butadiene are used, the solutions of which, at equal concentration in the same solvents, have different viscosities. A polymerisation product of high viscosity is desirable for the treads so as to ensure good resistance to attrition and wear; for the fabric carcasses of the tyres a polymeride of low viscosity is preferable. Mixtures of two or more polymerisation products of butadiene may be used for either purpose.

D. F. TWISS.

**Vulcanisation of rubber to leather.** L. B. CONANT, ASSR. to STANDARD PATENT PROCESS CORP. (U.S.P. 1,769,943, 8.7.30. Appl., 1.11.26).—The leather, after dehydration at moderate temperature and then heating to vulcanising temperature, is brought into contact with the rubber composition; the latter is then vulcanised. The leather is subsequently rehydrated by immersion in water and finally dried to a normal moisture content.

D. F. TWISS.

**Polymerised compounds.**—See III. Fatty acid derivatives.—See XII.

#### XV.—LEATHER; GLUE.

**Mode of action [on pelt] of pickles of different compositions. II. Acid absorption and swelling of collagen in sulphuric and formic acid pickling solutions.** A. KÜNTZEL and W. PREISANTANZ (Collegium, 1930, 577—593; *cf.* B., 1930, 873).—Sulphuric

acid pickling solutions, with sodium chloride or sulphate, were observed to have effects similar to those of hydrochloric acid pickles. Only 0.6 equiv. of acid was absorbed by the pelt from a sulphuric acid-sodium chloride pickle as compared with 0.7 equiv. when the sulphate was substituted. The degree of swelling was found to vary in the order:  $\text{HCl}/\text{NaCl} > \text{H}_2\text{SO}_4/\text{NaCl} > \text{H}_2\text{SO}_4/\text{Na}_2\text{SO}_4$ . Formic acid and pickles with weak organic acids differed in their action. The absorbed acid could not be calculated, as the acid-adsorption curve was not a straight line for weak organic acids and the amount of adsorbed acid was diminished by the salt in the case of lactic acid and increased with acetic and formic acids. The swelling curves were of the same form as for the mineral acid pickles and the pickling action of salt/formic acid was the same as that of mineral acid/sodium formate, although the  $p_{\text{H}}$  of the former is unaffected, whilst that of the latter is greatly influenced by the addition of the salt. The results disprove the Procter theory of pickling solutions, according to which all sodium chloride pickles act like hydrochloric acid pickles. The theory that deliming liquors must be buffered to  $p_{\text{H}} > 4$  is disproved. The prevention of swelling on the addition of salt is due solely to its pickling effect, and is independent of any buffering action.

D. WOODROFFE.

**Meaning of the basicity of one-bath chrome-tanning liquors.** E. STIASNY (Collegium, 1930, 574—577).—The free as well as the combined acid are both determined when making acidity titrations for the purpose of calculating the basicity of the chrome liquor (cf. Schorlemmer, B., 1920, 155 A). Much more free acid is present with "verolten" chromium complexes than exist in a chrome liquor of the same basicity containing chromium complexes, which have not been "verolten." The degree of basicity of the chromium salts present in a chrome liquor can be determined by titrating the free acid with 0.1N-sodium hydroxide in a comparator, using the potassium salt of *p*-sulphobenzeneazobenzylaniline and 0.05N-acetic acid as the standard ( $p_{\text{H}}$  3.0) to which the titration is to be carried. The chrome liquor should be diluted to contain 1 g. of chromium per litre before the titration. Results are given to show the differences obtained between the basicity figure of the liquor and that of the chrome-tanning salts. It is shown also that as the liquor is rendered more basic, the difference between the two figures diminishes. No difference is shown by many technical chrome liquors, since they have  $p_{\text{H}} > 3$ .

D. WOODROFFE.

**Theory and application of vegetable tanning.** M. MEUNIER (Halle aux Cuirs, Tech. Suppl., 1930, 33—54; Chem. Zentr., 1930, ii, 3107—3108).—A lecture.

A. A. ELDRIDGE.

**Photomicrographic methods for determining the kind of and faults in leather.** F. FEIN (Collegium, 1930, 593—598).—Photomicrographs are given of the grain of a number of goatskins, calfskins, and sheep and horse leathers which have been finished in different ways. A Zeiss camera and a microscope ( $\times 28$ ) were used.

D. WOODROFFE.

**Furs.**—See VI. Lacquers.—See XIII.

## PATENTS.

**Soaking of hides.** J. V. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,151, 16.10.29).—Dried hides and skins are soaked for 6—48 hrs. in 0.01—1.0% solutions of a water-soluble hydroxyalkylamine.

D. WOODROFFE.

**Treatment of scraps of skins and leather.** (Miss) A. ENGEL (B.P. 342,913 and 343,314, [A, B] 17.2.30. Fr., [A] 10.9.29, [B] 11.9.29).

**Softening of leather.**—See VI. Rubber-leather vulcanisation.—See XIV.

## XVI.—AGRICULTURE.

**Character of the colloidal materials in the profiles of certain major soil groups.** M. S. ANDERSON and H. G. BYERS (U.S. Dept. Agric. Tech. Bull., 1931, No. 228, 24 pp.).—The physical and chemical nature of the colloidal constituents of soils produced by three principal processes of soil formation is examined. The process of calcification involves the formation of colloids, of which the major constituents are uniform. The ratio silica: sesquioxides is relatively high, as also are the heat of wetting, moisture absorption, moisture equivalent, exchangeable-base content, and base-exchange capacity. The colloids of the different horizons of podsol profiles have very divergent characteristics. In A-horizons the silica: sesquioxide ratio is high, in B-horizons low, and in C-horizons intermediate. Physical values examined differed from those usually associated with the above ratios. Organic matter plays an important part in these colloids. Lateritisation produces colloids of low silica: sesquioxide ratio, low physical characteristics, and low base-exchange values. The kaolinised horizon resembles a mineral powder rather than a normal soil. Variations in the  $p_{\text{H}}$  of electrolysed clays are considerable and bear no relationship to chemical composition, but have a general direct association with silica: sesquioxide ratios. The  $p_{\text{H}}$  of electrolysed colloids increases on long keeping.

A. G. POLLARD.

**Variations of the colloidal material extracted from soils of the Miami, Chester, and Cecil series.** R. S. HOLMES and G. EDGINGTON (U.S. Dept. Agric., Tech. Bull., 1930, No. 229, 23 pp.).—Analyses of the colloidal matter separated from the various soils are recorded, together with data of base-exchange capacity,  $p_{\text{H}}$ , and water-adsorptive capacity. Interrelationships are discussed. The range of variation in composition of colloids in the various horizons of individual profiles is characteristic of the soil type.

A. G. POLLARD.

**Nature of the base-exchange materials of bentonite, soils, and zeolites, as revealed by chemical investigation and X-ray analysis.** W. P. KELLEY, W. H. DORE, and S. M. BROWN (Soil Sci., 1931, 31, 25—55).—X-Ray examination shows soil colloids to consist of crystalline particles. Grinding of soil colloids increased their content of exchangeable magnesium and potassium and, to a less extent, of sodium. The replaceable bases of colloids are located not only on the surface, but in the interior of the crystals. Inorganic soil colloids differ in chemical and physical properties from zeolites and from permutits. The



proportion of replaceable bases in soil colloids and in bentonite was not appreciably affected by heating to 350°, but it decreased at higher temperatures. Natural zeolites were much less stable to heat.

A. G. POLLARD.

**Soil sampling with a compressed-air unit.** H. F. BLANEY and C. A. TAYLOR (Soil Sci., 1931, 31, 1—3).—A sampling tube, working to 18 ft. depth, is driven into the soil by means of a compressed-air hammer.

A. G. POLLARD.

**Nitrate assimilation in soils.** F. B. SMITH and P. E. BROWN (Iowa Agric. Exp. Sta. Res. Bull., 1931, No. 135, 36—60).—Data are recorded of the assimilation of nitrates by soil organisms in a number of soils under various conditions of cropping and manuring.

A. G. POLLARD.

**Determination of available nitrogen, phosphorus, and potash in soils.** G. H. BUTLER (J. S. African Chem. Inst., 1931, 14, 10—21).—Current analytical methods are discussed.

A. G. POLLARD.

**Determination of the potash requirement of soils by means of *Aspergillus niger*.** H. NIKLAS, H. POSCHENRIEDER, and I. TRISCHLER (Ernähr. Pflanze, 1930, 26, 339; Bied. Zentr., 1931, 60, 4—5).—Precautionary measures in the application of the method are discussed, together with the significance of the results obtained.

A. G. POLLARD.

**Determination of the fertiliser requirements of soils.** GERLACH (Ernähr. Pflanze, 1931, 27, 73—77).—Comparative discussion of current methods.

A. G. POLLARD.

**Solubility of soil potash by the repeated extraction method of Saidel.** H. LIESEGANG (Landw. Versuchs-Stat., 1931, 111, 261—269).—Comparison is made in a number of soils of the assimilable potash (Neubauer) and the water-soluble potash determined by repeated extraction (Saidel and Cernescu, Ernähr. Pflanze, 1927, 321). The latter method may serve as a qualitative test of the easily-soluble potash in soil. Calculations of the relative solubility of potash do not provide figures utilisable in practice, although they may offer information of theoretical interest.

A. G. POLLARD.

**Increased root-solubility of the potash contained in soil as a result of phosphoric acid fertilisation.** C. DREYSPRING and F. HEINRICH (Superphosphate, 1931, 4, 46—59).—Examination of numerous soils by the seedling method showed that phosphate manuring increased the root-assimilable potash content. The proportionate increase varied widely with soil type and, in general, increased with the solubility of the phosphate fertiliser used; the relative effects of superphosphate and basic slag were of the order 2:1. The percentage increase in root-soluble potash following applications of phosphate increased as the original root-soluble content of the soil decreased, and was the greater in soils having the higher proportions of difficultly soluble potash, since the latter only is affected. The ability of phosphates to "activate" the difficultly soluble potash of soils is ascribed to the action of hydrogen ions derived from phosphoric acid or primary calcium phosphate, rather than to the effect of gypsum.

A. G. POLLARD.

**Influence of various fertilisers on the [crop] yield and reaction of sandy soils exhibiting strong exchange acidity.** H. RÖSSLER and L. SCHMITT (Landw. Versuchs-Stat., 1931, 111, 293—326).—Results of 5-year field trials are recorded. Potassium chloride or sulphate or kainite did not affect soil acidity. Kainite produced considerably increased yields of rye even on unlimed soil. Superphosphate and dicalcium phosphate did not increase soil acidity. Ammonium sulphate was definitely injurious. Farmyard manure caused a temporary reduction of acidity. Calcium cyanamide reduced soil acidity to a greater extent than sodium nitrate or basic slag. Relatively small dressings of calcium carbonate improved crop yields and reduced acidity, the effect being most marked in the season following that of its application.

A. G. POLLARD.

**Ammonium-calcium balance: a concentrated fertiliser problem.** L. G. WILLIS and J. R. PILAND (Soil Sci., 1931, 31, 5—23).—In pot cultures with a soil of low absorptive capacity diammonium phosphate used in conjunction with potassium nitrate and chloride proved toxic to cotton seedlings. The toxicity is attributed mainly to the formation of free ammonia by hydrolysis of the phosphate. Ammonium sulphate, chloride, and nitrate, in similar concentrations, were not toxic. Applications of calcium salts reduced ammonia toxicity in the order of effectiveness: sulphate > chloride > oxide > carbonate. Magnesium salts had a similar effect, but were less active than the corresponding calcium compounds. Monoammonium phosphate was less toxic than the diammonium salt, but the beneficial effect of calcium salts in this case was smaller.

A. G. POLLARD.

**Combined action of mineral fertilisers and stall manure on potatoes.** K. BÖRESCH and R. SCHREIBER (Ernähr. Pflanze, 1931, 27, 82—85).—In potato trials applications of farmyard manure with potassium salts proved the most successful. The nutrient value of farmyard manure is largely attributed to its potash content. A portion of the customary dressing of farmyard manure may be replaced by an equivalent amount of potassium salts without impairing the yield or quality of the crop.

A. G. POLLARD.

**Technique of the "Edelmist" process in practice.** K. (FHR.) VON HARSDOERFF (Ill. Landw. Ztg., 1930, 2, 34; Bied. Zentr., 1931, 60, 21—22).—Practical details of the preparation of Edelmist are recorded and its manurial value is confirmed.

A. G. POLLARD.

**Preparation and action of "Edelmist."** K. J. TSCHÉKALOV and A. T. KIRSSANOV (Mitt. Ackerbau, Leningrad, 1930, No. 31; Bied. Zentr., 1931, 60, 21—22).—In the preparation of Edelmist by the Krantz process, losses of organic matter, phosphorus, and nitrogen are smaller than by the usual method or by loose stacking without pressure. The germinating power of weed seeds is destroyed by the Krantz process. In field trials with cereals, Edelmist produced 30—50% greater yields than did yard manures, but showed little superiority with potatoes.

A. G. POLLARD.

**Decomposition of green manures in soil.** A. SOKOLOV (Arb. Poless. Landw. Versuchs-Stat., 1928, 2, 151; Bied. Zentr., 1931, 60, 22—23).—The moisture

contents of soils largely govern the rates of decomposition of green manures in them: The optimum moisture content for the nitrification of green manures is greater than that for unmanured soil. The low water-holding capacity of sandy soils retards nitrification sufficiently to cause a temporary accumulation of ammonia after green manuring. Addition of sulphuric acid to soil, whether green manured or unmanured, increases the accumulation of ammonia and retards nitrification. The ploughing-in of chalk with green manures accelerates the nitrification process.

A. G. POLLARD.

**Influence of inorganic iodine compounds on the occurrence and viability of some important soil bacteria.** H. KARNAHL (Zentr. Bakt. Par., 1931, II, 82, 494—518).—The effect of additions of the iodates and iodides of sodium and potassium on the activity of soil organisms was examined. Carbon dioxide production by the organisms was decreased by applications (up to 1 g. per kg. of soil) of each of the salts. No difference in the actions of iodide and iodate was observed. Parallel experiments with soil extracts gave similar results, but in this case the iodates had the greater depressive action. Bacterial numbers (on soil extract-agar) were reduced only by the heavier applications of iodine. In cultures, 1 g. of the salts per litre retarded denitrification and with 10 g. per litre only moulds survived. Nitrogen fixation and urea decomposition were retarded in cultures containing 1 g. of the salts per litre; iodates had a far greater effect than iodides. No beneficial effects of iodine applications were observed.

A. G. POLLARD.

**Contact insecticides. I. Surface tension, surface activity, and wetting ability as factors in the performance of contact insecticides.** W. C. O'KANE, W. A. WESTGATE, L. C. GLOVER, and P. R. LOWRY. II. **Lime-sulphur in relation to San José and Oyster Shell scales.** W. C. O'KANE and J. G. CONKLIN (New Hamps. Agric. Exp. Sta. Tech. Bull., 1930, Nos. 39 and 40).—I. Inter-relationships between the toxicity of numerous organic insecticides and the surface tension, surface activities, absorption, and ionisation of their solutions are examined.

II. Parallel measurements of the wetting power and angle of contact of lime-sulphur solutions on tree barks and scall insects are recorded. The angle of contact, surface tension, and wetting power of lime-sulphur were practically identical in effect with those of distilled water. Lime-sulphur solutions remained alkaline for 18—24 hrs. after spraying. Hydrogen sulphide was evolved for a few hours after application, but in quantity far too small to explain the toxic action of lime-sulphur. Sulphur dioxide was not found. Sulphide was not detected in the bodies of insects exposed to lime-sulphur.

A. G. POLLARD.

**Chemical nature and origin of the humus of soil.** S. A. WAKSMAN and H. W. REUSSER (Cellulose-chem., 1930, 11, 209—220).

**Ammonium nitrate.**—See VII. **Metals for dairies.**—See XIX.

See also A., Mar., 332, New York soils. 399,

**Plant feeding. Cotton leaves. Acidity of maize sap. 400, Mung beans. 401, Irradiation of plants.**

#### PATENTS.

**Raising of rice.** B. STANSEL (U.S.P. 1,770,867, 15.7.30. Appl., 2.8.29).—The addition of a film of crude oil (1 gal. per acre each day) to the water in rice fields from planting to sprouting of the seed is claimed to control insect pests; after sprouting, the field is drained and the process repeated to control weeds, which, at that stage, are not so aggressive as the young rice.

E. HOLMES.

**Immunisation of grain.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,500, 30.9.29).—A quinoneimide or derivative, *e.g.*, *p*-benzoquinone chloroimide or dichlorodimide, *o*-tolu-*p*-quinone chloroimide, naphthaquinone chloroimides, etc., is applied, diluted with talc, dextrin, etc. for the disinfection of seed grain.

C. HOLLINS.

**Immunisation of seed grain.** I. G. FARBENIND. A.-G. (B.P. 341,366, 1.5.30. Ger., 6.5.29).—Seed grain is disinfected with alkylthioarsines or alkylthioarsinic acid derivatives, *e.g.*, methylthioarsine (MeAsS) or the product from methylarsenious oxide and thio- $\beta$ -naphthol.

C. HOLLINS.

**Seed disinfectants.** M. S. KHARASCH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,770,886—7, 15.7.30. Appl., [A] 16.6.27, [B] 15.5.29).—Two complementary and comprehensive claims cover the use for the treatment of seed and plant diseases of compounds of the type R·Hg·X, where R is an alkyl or aryl radical and X an acid radical other than iodine. The materials may be mixed with spreaders and wetting agents and used as sprays, or with fillers such as bentonite, clay, infusorial earth, talc, or hydrated lime and employed as dusts.

E. HOLMES.

**Manufacture of [manganese arsenate] insecticides and the like of low water-solubility.** IMPERIAL CHEM. INDUSTRIES, LTD. From GRASSELLI CHEM. CO. (B.P. 340,882, 18.3.30).—The reaction between manganese dioxide and arsenious acid in aqueous medium is carried out at 100° under a steam pressure of 80 lb./in.<sup>2</sup> with agitation and in presence of an alkaline agent, such as lime. The product contains less than 1% of water-soluble arsenic (calc. as As<sub>2</sub>O<sub>3</sub>).

W. J. WRIGHT.

**Fungicide [with sulphur base].** H. A. LEE (U.S.P. 1,770,001, 8.7.30. Appl., 22.1.27).—Finely-divided sulphur, preferably passing 200—300-mesh, mixed with small amounts of active oxidising reagents such as 1% of potassium permanganate or 0.25% of nitric acid is claimed to be converted rapidly on the plant into the fungicidal polythionic acids.

E. HOLMES.

**By-products from gas.**—See II.

#### XVII.—SUGARS; STARCHES; GUMS.

**Filtration of crude juice in sugar manufacture.** C. W. SCHONEBAUM (Chem. Weekblad, 1931, 28, 162—163).—Results of large-scale trials with colloid filters proved satisfactory. It was found necessary to preheat the liquor for a few minutes at 80—85°, and to reduce the acidity by addition of alkali.

S. I. LEVY.

**Determination of the starch content of potatoes by the Reimann balance.** B. LAMPE (*Z. Spiritusind.*, 1931, 54, 36).—For the determination of the sp. gr. of the potatoes from which the starch content is calculated, the Reimann balance need not be corrected. The measurements are independent of the relation of the arms of the balance, and deviations from the customary 10 : 1 ratio do not vitiate the result, provided the deviation is not corrected by the use of the rider, and provided the weights are absolutely accurate. C. RANKEN.

See A., Mar., 306, **Starch gels.** 337, **Dextrins.**

PATENT.

**Lactose.**—See I.

### XVIII.—FERMENTATION INDUSTRIES.

**Composition and determination of proteins in Danish barley.** G. HOFMAN-BANG (*J. Inst. Brew.*, 1931, 37, 72—80).—The percentage of glutelin in samples of the same variety of Danish barley is constant, no matter what the soil conditions are. The content of salt-soluble protein decreases and that of hordein increases in proportion as the total protein content increases. Storage of immature grain at 20° causes a transformation of salt-soluble protein into glutelin, whereas the hordein content remains constant. This change is rapid at 35°, but slow or negligible at 10°. When barley is dried the change is in the reverse direction and the salt-soluble and alcohol-soluble proteins increase at the expense of the glutelin. The salt-soluble protein content of the malted barley is highest if the malting is carried out at a low temperature and the carbon dioxide is allowed to accumulate, and lowest with warm malting in an atmosphere free from carbon dioxide. C. RANKEN.

**Fractionation of the proteins of barley and malt.** K. MYRBÄCK and S. MYRBÄCK (*Woch. Brau.*, 1931, 48, 43—47).—From a re-investigation of the methods it is concluded that the precipitates obtained with stannous chloride and ferric acetate are not sufficiently reproducible to form a trustworthy basis for defining Schjerning's fractions "albumin I" and "propeptone," and doubt is thrown on the existence of any definite fraction corresponding to the latter term. Saturation with magnesium sulphate and precipitation by mercuric chloride and uranyl acetate give reproducible fractions: I, the "protein fraction"; II, III, corresponding to Schjerning's "denuclein" and "peptone" fractions, respectively; IV, consisting of simpler peptides and amino-acids. This method of fractionation shows no noteworthy difference between the water-soluble nitrogenous constituents of good and poor quality barleys. In making cold water extracts of malts, fine grinding, short extraction time, and low temperature are necessary to minimise enzyme action; violent shaking must be avoided since it denatures part of the protein. The composition of the nitrogenous constituents of cold water extracts of malt is much more variable than that of "congress" worts. In the latter the soluble nitrogen is generally about 33% of the total nitrogen in the malt, and it contains about 28, 7, 13, and 52% of fractions I—IV, respectively. F. E. DAY.

**Removal of head during [bottom] fermentation, and its effect on beer and yeast.** A. HALLERMANN (*Woch. Brau.*, 1931, 48, 31—38).—Following a discussion of the effects of contact between the fermenting wort and the head, which contains undesirable impurities, several possible devices for minimising this contact are described. That adopted by the author consists of two shallow trays, apparently about 6—7 cm. deep, each fitting loosely into half of the fermenting vessel, and supported on floats 1 cm. above the surface of the wort. Shallow conical rims, dished upwards at about 17 cm. distance from centre to centre, in the bottom of the trays surround large holes through which the head works, the wort carried by it draining off through small (1 cm. diam.) holes in the floor of the trays. The trays may be constructed of rustless steel or of aluminium. It is claimed that the flavour of the beer and the purity of the yeast are greatly improved. F. E. DAY.

**Measurement of surface tension of worts and beers.** J. DE CLERCK (*Bull. Assoc. anc. Etud.*, Louvain, 1930, 30, 136; *Woch. Brau.*, 1931, 48, 22—26).—The flow-adjusting capillary of Traube's stalagmometer is replaced by a longish glass tube (5—6 mm. in diam.) fixed parallel to the instrument, their upper ends being joined by an india-rubber tube. Near its upper end the glass tube carries a side tube with stopcock and just below this another stopcock. With the latter closed, liquid is drawn into the stalagmometer till the bulb is about three quarters full, when the side stopcock is closed. A vessel of water is placed under the lower end of the vertical tube, the stopcock in which is now opened, and the liquid in flowing from the stalagmometer draws a column of water into the tube. When the desired rate of outflow is attained the water vessel is withdrawn, and air bubbles are drawn through the water column, their resistance maintaining a constant rate of outflow from the stalagmometer. The addition of graduations to the regulating tube is of advantage in reproducing previous conditions. Some determinations of the effect of varying conditions on the results are quoted and discussed. It is shown that reproducible results may be obtained on worts and beers by comparing the weights of 25 drops falling at a rate of 10 drops/min. at 17.5° with that (taken as 100) of 25 drops of water at the same rate and temperature. If the outflow rate is not exactly 10 drops/min., a small correction, determined for each stalagmometer, must be applied. F. E. DAY.

**Determination of fixed acids in wine.** P. BRUÈRE (*J. Pharm. Chim.*, 1931, [viii], 13, 77—81).—An apparatus is described by which the non-volatile acids may be determined on 4—5 c.c. of wine placed in a boiling tube which is fixed in a corked, wide-necked, conical flask used as a steam generator. Tubes are so arranged that the steam generated is released into the atmosphere until the wine is brought to the b.p., when it is passed through the wine, carrying over the alcohol and volatile acids. Excessive increase in volume, or loss of fixed acid due to concentration, is thereby avoided.

T. McLACHLAN.

**Determination of glycerol in wines and fermented beverages.** L. SEMICHON and M. FLANZY (*Ann. Falsif.*, 1930, 23, 583—602).—Various methods of extracting and

determining glycerol have been studied and the following slightly modified method of Ferré and Bourges (Proc. 7me Cong. Chem. & Ind., Bordeaux, 1928, 775) is recommended. Gums, acids, and sugars are precipitated with 95% alcohol followed by baryta, excess of the latter is then removed by carbon dioxide, and the liquid is concentrated to remove alcohol. The liquid is distilled at about 80° *in vacuo* under a pressure of 30–40 cm. Hg, the distillate passing into chromic acid mixture maintained at 100°. The residue is distilled in steam at 100–115° for 3 hrs. so that 600 c.c. of distillate are collected, the vapours also passing through the chromic acid mixture. Excess of chromic acid is determined by titration with ferrous ammonium sulphate, using ferri-yanide as indicator.

E. B. HUGHES.

See A., Mar., 333, Absolute alcohol. 392, Glyoxalase. 393, Beer yeasts. 394, Fermentation citric acid.

## PATENTS.

**Purification of solutions of enzymes.** A. R. BOLDIN and I. A. EFFRONT (B.P. 315,877, 18.7.29. Fr., 21.7.28).—The raw enzymic solution, which may be prepared by the fermentation of an amylaceous medium by *B. mesentericus*, is freed from fatty matters by a solvent of fatty acids and salts. Proteins and colloids are precipitated by the addition of a heavy-metal salt or by the adjustment of the  $p_H$  of the solution, and removed with the micro-organisms from the solution by filtration or centrifuging. The amylolytic and proteolytic enzymes may be separated by treatment with various adsorbents.

C. RANKEN.

**Manufacture of hopped, alcohol-reduced, fermented beverages.** C. A. MITCHEL, Assr. to H. E. FREES Co. (U.S.P. 1,769,749, 1.7.30. Appl., 30.7.28).—After removal of the essential oils and volatile constituents from hops by steam-distillation, the hop residue is boiled with a cereal wort. The major portion of the hopped wort is dealcoholised after fermentation, and is concentrated by boiling to 45–60% of its original volume. The minor portion of the wort, to which the hop volatile constituents are added, is diluted and fermented, and is mixed with the dealcoholised portion in such proportions that the alcohol content of the mixture does not exceed the legal limit.

C. RANKEN.

**Manufacture of absolute alcohol.** R. B. CROWELL, Assr. to AMER. SOLVENTS & CHEM. CORP. (U.S.P. 1,761,779, 3.6.30. Appl., 9.12.26).—Benzene or an equivalent substance is added to aqueous alcohol in amount less than 9.5 (preferably 3.5) times the weight of water present, and the mixture is distilled to separate an azeotropic mixture of alcohol, benzene, and water. During distillation sufficient benzene is maintained in the still until the alcohol is completely dehydrated. The condensed ternary mixture is separated into two layers, and the benzene layer is returned to the still during distillation.

C. RANKEN.

**[Fermentation] treatment of sulphite-liquor spirit.** ZELLSTOFFFABR. WALDHOF, and M. GADE (B.P. 341,358, 15.4.30. Ger., 24.4.29).—The aldehyde first runnings from the distillation of sulphite-liquor spirit is

conducted, either continuously or periodically, to a fresh mash which is fermenting. The spirit obtained by distilling a mash so treated shows no increase in its content of aldehyde, but yields more alcohol.

C. RANKEN.

## XIX.—FOODS.

**Diastase and wheat flour.** E. BERLINER and R. RÜTER (Z. ges. Mühlenw., 1930, 5, 134–140, 156–162; 7, 63–66; Chem. Zentr., 1930, ii, 2319–2320).—Comparative experiments have been performed on the action of diastase of various origin on soluble starch and wheat starch under different conditions, the  $p_H$ ,  $\alpha_D$ , viscosity, and iodine reaction being observed during various periods of time. Increase in the percentage of diastase and in the concentration of starch accelerates decomposition of the starch. A soft Swedish wheat flour was less readily saccharified than a Manitoba flour; a similar difference was observed with the corresponding starch. Erythro-dextrin was determined colorimetrically by means of iodine, using an ammoniacal copper sulphate filter. Germinated flour affords small and diminishing quantities of erythro-dextrin; ungerminated flour affords larger quantities. Determinations of the soluble matter, maltose, and nitrogen in aqueous suspensions of wheat flour were made in order to elucidate autolytic processes in dough. The liability of wheat flour to diastatic action appears to depend more on the equal distribution of saccharification during the whole period of reaction than on the absolute quantity of sugar.

A. A. ELDRIDGE.

**Viscosity of alkaline [extracts of] pure wheat flours and its relation to practical bakery problems.** T. BIÉCHY [with R. MÜLLER, GRAMS, and NÜCHTER] (Kolloid-Z., 1931, 54, 194–210).—Viscosity measurements have been made with a 0.05*N*-sodium hydroxide extract of wheat flour, using Lavaczek's viscosimeter. The procedure is recommended as a means of characterising flour, and graphs have been constructed to enable the necessary data to be obtained from one determination carried out with a known amount of the sample. A method, based on displacement of a liquid, has been developed for determining the amount of gas evolved from the dough during fermentation. The experimental data demonstrate that for doughs of constant fermentative power, the volume of the bread produced increases with the viscosity of the alkaline extract of the flour.

E. S. HEDGES.

**Metals in the dairy.** POPP. I. Review of literature. T. LIEPUS and OSTERBERG. II. Examination of disinfectants and cleansers. OSTERBERG. III. Comparative bactericidal effects of cleansers and disinfectants. RICHTER, ESPE, and BAADE. IV. Action of disinfectants and cleansers on metals and alloys. MOHR, OSTERBERG, GUERTLER, and T. LIEPUS (Landw. Versuchs.-Stat., 1931, 111, 271–291).—III. Mechanical cleansers did not destroy milk bacteria on metallic surfaces. Washing with water after treatment with alkali did not remove bacteria adhering to metallic surfaces. Addition of water-glass or soap to cleansers did not improve their bactericidal action. Of the disinfectants examined, inorganic and organic chlorine compounds were the most effective.

IV. Corrosion tests with numerous commercial cleaners and disinfectants on various metals and alloys are recorded. In general, chromium-nickel steels, pure nickel, and certain copper-nickel alloys containing small proportions of zinc, manganese, etc. were the least affected. Corrosion of pure metals was in the order  $\text{Ni} < \text{Cu} < \text{Fe}$ ,  $\text{Zn}$ , and  $\text{Al}$ . Chromium-plated metals were very satisfactory. A. G. POLLARD.

**Retained milk.** O. LAXA (Ann. Falsif., 1930, 23, 609—610).—A complete analysis of milk from cows which had not been milked for three days is given. The proportion of lime and chlorine in the ash was normal, but the ratio of chlorine to lactose was very high. E. B. HUGHES.

**Composition and analysis of milk-and-egg cream.** G. PANOPoulos and J. MEGALOIKONOMOS (Praktika, 1927, 2, 10 pp.; Chem. Zentr., 1930, ii, 3097).—The total fat ( $a$ ), determined by the Röse-Gottlieb method, should be 5.3% ( $\pm 10\%$ ); the butyrefractometric value ( $b$ ) of the ethereal extract of the dried material is also determined. Then the milk fat  $x = 0.5(63a - ab)$ . The value of  $b$  should be 49—50, corresponding with 65% of milk fat. A. A. ELDRIDGE.

**Spanish cheese.** K. TEICHERT and W. STOCKER (Milch. Zentr., 1931, 60, 33—34).—The favourite Spanish cheeses are "Jdiazabal" and "Montana," which are made from ewe's milk. Proximal analyses of samples of these cheeses are given. B. W. TOWN.

**Green vegetable cheese.** K. TEICHERT (Milch. Zentr., 1931, 60, 17—19).—A description of the manufacture of this cheese is given. Sweet separated milk is warmed in tinned copper vessels to 75—78° and stirred. Lactic acid is then slowly sprayed into it, with care, so that the curds are not tough or hard. The presence of either iron or copper spoils the curd for cheesemaking. The curd is allowed to ripen for 4—6 weeks, and salted; the cheese is now ground in stone mills with blue Melilot (*Melilotus caerulea*), to which the peculiar taste and green colour of the cheese are due. The cheese is packed and stored for drying. B. W. TOWN.

**Cheese possessing the nature of "laitage."** M. FOUASSIER (Ann. Falsif., 1930, 23, 611—613).—A legal definition of the term "laitage" as applied to cheese, for purposes of taxation, is given. E. B. HUGHES.

**[Significance of] sorbitol in the analysis of fruit preparations.** C. F. MUTTELET (Ann. Falsif., 1930, 23, 602—605).—The author makes use of his previous observation (cf. B., 1930, 482), that the sorbitol content of jam or preserve may serve as an indication of the presence of apple juice, in a method for the determination of sorbitol in such products. The method consists in removing sugars by fermentation with yeast and then precipitating the sorbitol in acid solution as dibenzylidenesorbitol by addition of benzaldehyde. The weight of the dibenzylidenesorbitol is twice that of the sorbitol. E. B. HUGHES.

**Proximate composition of fresh vegetables.** C. CHATFIELD and G. ADAMS (U.S. Dept. Agric. Circ., 1931, No. 146, 24 pp.).—Analytical data for numerous vegetables are recorded. A. G. POLLARD.

**Artificial removal of astringency in kaki.** K. KAKESITA (Proc. Imp. Acad. Tokyo, 1930, 6, 397—398).—The astringency is due to a constituent, shibuol, which contains a phloroglucinol and a gallic acid nucleus in its molecule (cf. Komatsu and Matsunami, B., 1925, 611). Treatment with acetaldehyde, or other aldehyde, causes its polymerisation and removes the astringency, and it is thought that this is responsible for its absence in sweet kaki and for its removal experimentally from the bitter variety. T. McLACHLAN.

**Iodine content of shrimp waste.** H. PARKER, JUN., and F. C. VILBRANDT (J. Amer. Chem. Soc., 1931, 53, 633—635).—Dry shrimp waste contains 16.9 pts. of iodine per million, the majority of which is organically combined. Thyroxine could not be detected in the hydrolysate of the waste. H. BURTON.

See also A., Mar., 320, Ergosterol. 381, Iron in milks. 385, Benniseed. 394, Cheese. 395, *B. coli* in milk. 398, Vitamins. 401, Japanese persimmon.

#### PATENTS.

**Treatment of milk.** R. L. HILL, Assr. to DARCO CORP. (U.S.P. 1,772,349, 5.8.30. Appl., 20.10.27).—Objectionable flavours and odours may be removed from milk by mixing with it activated carbon (for at least 5 min.) and then filtering. E. B. HUGHES.

**Preservation of milk.** R. HELLERUD (B.P. 340,622, 5.10.29).—To avoid separation of fat in prolonged heating for preservation the milk is first homogenised at 74°. E. B. HUGHES.

**Manufacture of cheese.** S. H. HARTMANN and S. C. WILSON (B.P. 340,485, 23.9.29. Austral., 28.11.28).—Dried or concentrated buttermilk is mixed with finely-divided matured or partly matured cheese. E. B. HUGHES.

**[Apparatus for] manufacture of whipped cream.** L. MELLERSH-JACKSON. From E. COHNITZ (B.P. 340,265, 22.6.29).

**Cooking or sterilising [of bottled or canned goods] by steam under pressure.** W. W. TRIGGS. From G. J. MEYER MANUFG. CO. (B.P. 342,397, 9.11.29).

**Cooling of milk. Desiccation of juices etc.**—See I. Waterproof products.—See VI. Beverages.—See XVIII. Vitamins.—See XX.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Identification of quinine in quinine syrup, F.P.** R. GUYOT (Bull. Soc. Pharm. Bordeaux, 1930, 68, 115—118; Chem. Zentr., 1930, ii, 2681).—The thalleioquinine reaction fails unless the ethereal extract is evaporated and the residue dissolved in dilute acetic acid. The syrup is preferably diluted with an equal quantity of water, and sodium hydroxide is employed instead of ammonia. A. A. ELDRIDGE.

**Detection of quinine in quinine syrup.** R. GUYOT (Bull. Soc. Pharm. Bordeaux, 1930, 68, 118—121; Chem. Zentr., 1930, ii, 2681).—When calcium hydroxide solution or solid potassium hydroxide is added to a solution of quinine containing bromine a red colour appears; the colour of the upper portion changes to

green when ammonia solution is cautiously added. The development of the colour is accelerated by copper sulphate or ferrous sulphate. The green colour obtained in the thalleioquinine reaction is intensified by subsequent addition of sodium hydroxide; finally, addition of excess of acetic acid affords a stable blue coloration which can be employed quantitatively by comparison with standard Fehling's solution ("cyanoquinine reaction").

A. A. ELDRIDGE.

**Determination of pyrethrin I.** R. C. VOLLMAR (Ind. Eng. Chem. [Anal.], 1931, 3, 110—112).—The following method for the determination of pyrethrin I in kerosene extracts of the flowers is an adaptation of the short acid method of Tattersfield and Hobson (B., 1929, 732). 100 C.c. of the extract are refluxed for  $1\frac{1}{2}$ —2 hrs. with 5 c.c. of *N*-methyl-alcoholic caustic soda and then as much as possible of the kerosene is poured off and refluxed with alcoholic soda again. The kerosene is again poured off and to the residue are added 11 c.c. of *N*-sulphuric acid and, after shaking, the mixture is transferred to the first flask. The second flask is washed with two 25-c.c. portions of light petroleum, which are added to the main bulk; this is then steam-distilled and the distillates are titrated with 0.02*N*-caustic soda in the manner described by Tattersfield (*loc. cit.*). Analyses of flowers and extracts are given. E. H. SHARPLES.

**Alkaloid content of sun- and shade-dried mydriatic *Solanaceæ* drugs.** E. KOPP (Pharm. Zentr., 1931, 72, 113—116).—No difference is observed between the alkaloid contents of sun-dried and shade-dried samples of leaves and stems of *Atropa belladonna* and of *Hyoscyamus niger*. Shade-dried leaves of *Datura stramonium*, *D. metel*, and *D. tatula* contain, however, about 10% more alkaloid than the corresponding sun-dried drugs.

H. E. F. NOTTON.

**Manufacture of benzonaphthol and salophen.** J. SCHWYZER (Pharm. Ztg., 1931, 76, 186—188).—Methods suitable for the manufacture on the 50-kg. scale of benzonaphthol ( $\beta$ -naphthyl benzoate) from  $\beta$ -naphthol, salicylic acid, and phosphoryl chloride are described. Phenyl and  $\beta$ -naphthyl salicylates are prepared similarly. In the manufacture of salophen (*p*-acetamidophenyl salicylate) *p*-nitrophenol is reduced by iron and water at 80—100° and the product quantitatively acetylated by acetic anhydride in glacial acetic acid. Salophen is formed almost quantitatively by distilling together iron-free *p*-acetamidophenol and phenyl salicylate at 210—225°/20 mm., but its preparation from salicylic acid, phosphoryl chloride, and the phenol is unsatisfactory.

H. E. F. NOTTON.

**Detection of juglone.** R. FISCHER and F. STAUDER (Pharm. Zentr., 1931, 72, 97—100).—Juglone (cf. Tunmann, A., 1912, ii, 1110) cannot be detected by the micro-sublimation method of the D.A.B. VI in leaves of the walnut, *Juglans regia*, fresh, or dried for three days, or in pericarps, sliced and dried for eight days. It is, however, found in all three, but not in leaves dried for longer periods by sublimation of a chloroform extract. Attempts to sublime  $\alpha$ - or  $\beta$ -hydrojuglone from fresh pericarps or their extracts were unsuccessful.

H. E. F. NOTTON.

**Determination of pantopon [omnopon] in syr. guaiac. co.** O. TOMÍČEK and O. PRČOKE (Casopis Českoslov. Lék., 1930, 10, 239—244; Chem. Zentr., 1930, ii, 2811).—The alkaloidal mixture gives with Marquis' reagent the same red colour as morphine, the colour becoming blue at 100°. Silicotungstic acid solution (10%) gives a voluminous precipitate, whilst syrup containing morphine remains clear or gives a slight turbidity. The determination is carried out by extraction from ammoniacal solution with chloroform and methyl alcohol.

A. A. ELDRIDGE.

**Preparation of hydrarg. cum creta and of finely-divided mercury.** G. VITTE (Bull. Soc. Pharm. Bordeaux, 1930, 68, 73—76; Chem. Zentr., 1930, ii, 2670).—Wood charcoal (3 pts.) is ground with mercury (9 pts.) and precipitated calcium carbonate (15 pts.); the mercury is thus more readily comminuted and reassociation of the particles when the calcium carbonate is attacked by the gastric juice is partly avoided. The use of kaolin instead of calcium carbonate is recommended.

A. A. ELDRIDGE.

**Composition of hydrarg. cum creta.** G. VITTE (Bull. Soc. Pharm. Bordeaux, 1930, 68, 77—80; Chem. Zentr., 1930, ii, 2670—2671).—Commercial samples, on extraction with 10% acetic acid, yielded up to 4 mg. of mercuric oxide per g. Mercuric oxide is formed by excessively long grinding.

A. A. ELDRIDGE.

**Occurrence of acetyleugenol in clove oil.** J. HEROLD (Riechstoffind., 1930, 5, 100—102; Chem. Zentr., 1930, ii, 3206).—After removal of acetic acid by shaking with dilute sodium carbonate solution, the total eugenol content is determined by the benzoate method; acetyleugenol is determined by hydrolysis with alcoholic potash and titration of the excess of alkali. Commercial clove oil ( $d_{4}^{15}$  1.0672) contained 18.93% of acetyleugenol.

A. A. ELDRIDGE.

**New constituents of the essential oil of *Chamaecyparis obtusa* wood.** G. TAKAHASHI, Y. YOSHIKI, and T. YAGINUMA (Proc. Imp. Acad. Tokyo, 1930, 6, 393—396).—Two constituents,  $C_{20}H_{32}O_2$  (m.p. 236°) and  $C_{22}H_{22}O_7$  ( $d_4^{25}$  1.337, m.p. 64°) have been discovered by means of the polarising microscope and their crystal constants determined. Bromine derivatives,  $C_{22}H_{20}O_7Br_2$  ( $d_4^{25}$  1.664, m.p. 134—135°) and  $C_{14}H_{16}O_4Br_2$  (m.p. 117°) have been obtained from the former and their crystal constants measured.

T. McLACHLAN.

See A., Mar., 320 and 399, Ergosterol. 326, Hypophosphites and arsenites. 330, Vacuum evaporation etc. 336, Benzoates. 350, Synthetic bases. 371, Alkaloids. 372, Arseno-organic compounds. 375, Determination of alkaloids. 387, "Larocain." Anæsthetic octyl alcohols. 388, Analgesics. Piperidine derivatives. Tincture of digitalis. 389, Therapeutic electrodes. 395, Antidiphtheria serum. 397, Insulin. 398, Male sexual hormone. Vitamins. 401, Korean *Ledum palustre*. *Patricia acuminata*. 402, Tobacco.

## PATENTS.

**Recovery of nicotine.** H. K. McCONNELL, Assr. to TOBACCO BY-PRODUCTS & CHEM. CORP. (U.S.P. 1,770,753,

15.7.30. Appl., 29.11.21).—A solution of a nicotine salt is incorporated with a hydroxide of a metal capable of forming a water-insoluble compound with the acid present in the salt, and then a water-soluble compound of an alkali metal is added in solid form and in an amount which will form an aqueous solution having a density greater than that of the nicotine solution and in which the nicotine is insoluble. Also it should be not more than sufficient to form a saturated solution with all the water finally present in the mixture. On the addition of water to this mixture the nicotine separates and is drawn off.

E. H. SHARPLES.

**Production of ephedrine inhalant.** E. B. CARTER, Assr. to E. LILLY & Co. (U.S.P. 1,762,128, 3.6.30. Appl., 23.7.28).—Ephedrine alkaloid, preferably containing not more than 3% of moisture, is dissolved in a mineral oil and to the filtered solution, containing about 1% of alkaloid, is added oil of rose.

E. H. SHARPLES.

**Manufacture of barbituric acid derivatives.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 339,937, 14.8.29).—The *N*-methyl or -ethyl derivatives of 5-phenyl-5-alkylbarbituric acids have reduced toxicity and prolonged hypnotic effect. Barbituric acids from ethyl phenylethylmalonate and methylcarbamide (m.p. 176–5°) or ethylcarbamide (m.p. 102°), from ethyl phenylmethylmalonate and methylcarbamide (m.p. 154°), and from ethyl phenyl-*n*-propylmalonate and methylcarbamide (m.p. 109°) are described. [Stat. ref.]

C. HOLLINS.

**Manufacture of (A) pyracridone, (B) derivatives of pyracridone.** I. G. FARBENIND. A.-G., Assees. of DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 319,794 and 339,932, 28.9.29. Ger., 29.9.98).—(A) Anthranilic acid is heated with a 2-halogenopyridine, in a solvent if desired, at 150–160°. Pyracridone, m.p. 210°, is isolated by dissolving in hydrochloric acid and precipitating with alkali. (B) Pyracridone is nitrated with mixed acid below 50° to give a dinitro-compound, m.p. 228°, which is reduced by stannous chloride to diaminopyracridone, m.p. 297°.

C. HOLLINS.

**Production of hormone preparations.** "PHARMAGANS" PHARMACEUTISCHES INST. L. W. GANS A.-G. (B.P. 315,709, 12.7.29. Ger., 16.7.28).—Stable hormone preparations, e.g., of insulin, capable of oral administration and not destroyed by the digestive juices, are made by the colloidal dispersion of hormones, compounds of hormones, etc., either alone or in presence of substances such as lipins, in vegetable oils and fats, especially those capable of producing reflux of bile in the stomach, such as butter, margarine, etc.

E. H. SHARPLES.

**Therapeutic substance [from suprarenal glands].** W. B. COFFEY and J. D. HUMBER (U.S.P. 1,771,976, 29.7.30. Appl., 12.3.30).—The outer or cortical layer of the suprarenal gland, either with or without the enveloping membrane, is progressively extracted with water at 60–85° and the aqueous extract, after cooling, is treated with alcohol. The mixture, which may be kept for a time at 10–18°, is filtered, the filtrate being set aside and then filtered again. Alcohol is removed from the filtrate, and the residue is kept at 12° to permit

clouding and settling, with occasional filtering until the solution remains quite clear for 36 hrs. A stable, non-irritating extract is obtained which, when injected subcutaneously at a point removed from the involved tissue, causes sloughing or necrosis of carcinoma, sarcoma, and other malignant tissue. E. H. SHARPLES.

**Manufacture of basic bismuth salts of organic mercury compounds.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 339,760, 15.11.29).—A simple or complex bismuth salt is caused to react with a soluble organic mercury compound, containing an acid group, in which the mercury is non-ionised. The products are useful against syphilis. Examples are: sodium *O*-carboxymethylmercurisallylallylamide with bismuth nitrate in glycerin and water, giving on neutralisation with sodium hydroxide a basic bismuth salt; basic bismuth salts of mercurated 3-acetamido-4-allyloxy-benzenearsinic acid, of mercurated benzthioiminazole-4-arsinic acid, or -4-carboxylic acid, or -4-sulphonic acid, and of mercurated *m*-allylthiocarbamidobenzoic acid.

C. HOLLINS.

**Production of antirachitic substances.** A. J. PACINI, Assr. to C. M. RICHTER (U.S.P. 1,771,343, 22.7.30. Appl., 10.12.27).—Substances are irradiated as described previously (cf. U.S.P. 1,681,120; B., 1928, 767) in presence of photocatalysts, halogens, or other catalysts. Electromagnetic radiation in presence of suitable catalysts may also be used.

E. H. SHARPLES.

**Synthesis of vitamins.** A. J. PACINI, Assr. to C. M. RICHTER (U.S.P. 1,762,105, 3.6.30. Appl., 20.8.28).—Vitamin-*A* (anti-xerophthalmic, as distinct from vitamin-*a*, growth-stimulating) is synthesised by treating suitable substances, such as cholesterol, with infra-red or ultra-violet irradiation in an inert atmosphere for a long enough period to produce the vitamin-*A*, the treatment being discontinued before a substantial amount of vitamin-*D* is produced (cf. U.S.P. 1,681,120; B., 1928, 767).

E. H. SHARPLES.

**Sterilisation of organic substances etc.** E. SCHULZE (B.P. 339,602, 22.7.29).—A 0.1–0.2% solution of benzoic acid, to which have been added a few drops of a 10% alcoholic solution of a quinoxaline, particularly an alkylated hydrocupreine having 5–9 carbon atoms in the alkyl group (e.g., eucupine and vucine), is suitable for permanently sterilising organic substances, dressings, etc. without heat.

C. HOLLINS.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic emulsion spots of biological origin.**

R. B. ARCHEY (Phot. J., 1931, 71, 19–30).—The desensitisation produced by certain types of bacteria is shown to be due to certain desensitising excrements of the living cells. Infection takes place by putrefaction of the parent growth, crenothrix and associates, which disintegrates into particles saturated with the bacteria body product and these are conveyed by air currents to raw paper, gelatin, or emulsion surface. Antiseptics which do not chemically alter the desensitiser will not prevent insensitive spots. Potassium permanganate



successfully destroyed the desensitising and fogging action of a bacteria filtrate, whereas potassium dichromate and hydrogen peroxide were useless and bromine water was only partly successful. J. W. GLASSETT.

**Secondary reactions in latent image formation. Influence of free alkali halide.** T. S. PRICE (Phot. J., 1931, 71, 59—69).—The action of gelatin as a halogen acceptor has been investigated by measuring the rate of absorption of hypochlorite (or, in experiments with a bromide emulsion, hypobromite) by gelatin sol at constant  $p_H$  (buffered solutions). The rate (amount used up after 2 min.) increases from a very small value at  $p_H$  6 to a comparatively high value at  $p_H$  10. Addition of a small amount of sodium chloride to the hypochlorite reaction mixture has no effect, but potassium bromide addition to hypobromite causes marked decrease of the rate of reaction. From considerations of the probable reactions and equilibrium equations, it is shown that the concentration of  $OCl^-$  ion varies with the  $p_H$  in an analogous manner to the reaction velocity. For hypobromite solutions the  $OBr^-$  concentration also rises with  $p_H$ , but free bromine is present in all solutions up to  $p_H$  9. It is concluded that halide liberated by the action of light on silver halide forms "hypohalide" ions which react preferentially with gelatin. Above  $p_H$  10, in both cases, the velocity of reaction falls continuously, but addition of alkali halide increases it. The reduction of the sensitivity of an emulsion by the presence of excess of salts is investigated and discussed. J. LEWKOWITSCH.

See A., Mar., 278, Recording electrons. 319, Latent images. Reversal process. Gelatin.

#### PATENTS.

**Treating derivatives of aqueous cellulose compounds for use in photographic and its allied arts etc.** F. W. HOCHSTETTER (U.S.P. 1,772,081, 5.8.30. Appl., 27.8.27).—To render them water-, alkali-, and acid-proof, films derived from aqueous cellulose solutions are steamed to make them absorbent, cooled, and treated at 30° with a gelatinous waterproofing solution containing gelatin, ammonium bromide, sodium chloride, potassium iodide, alum, and tannin. After successive cooling and heating, the films are further treated, first with a gelatinous non-inflammable compound (aqueous gelatin, magnesium sulphate, alum, ammonium carbonate), then with a medium sensitive to light (aqueous gelatin, potassium dichromate, and ammonia), and are finally cooled, dried, and exposed to strong light and formaldehyde gas. F. R. ENNOS.

**Manufacture of light-sensitive layers.** FRANGIALLI & CIE., Assees. of SOC. DES MAT. COL. & PROD. CHIM. DE ST. DENIS, and R. LANTZ (B.P. 340,166, 10.2.30. Fr., 9.2.29).—Naphthasultones, obtained, e.g., by heating diazotised 1:8-naphthylaminesulphonic acids in substance or on the substrate, are used to generate coupling components in diazo-types on development with alkali. Examples are: diazotised thioaniline and sodium 1:8-naphthasultone-4-sulphonate (red); diazotised 4-aminodiphenylamine-2-sulphonic acid and 1:8-naphthasultone-3-sulphonic acid (ammonia develop-

ment, violet-blue; reddish in presence of nickel sulphate); diazotised 4-aminodiphenylamine and diazotised  $\alpha$ -naphthylamine-4:8-disulphonic acid, heated at 80° to give the sultone (ammonia development, violet-black); diazotised 2R-acid and 1:8-naphthasultone-4-sulphonic acid (ammonia development, red-violet).

C. HOLLINS.

**Manufacture of light-sensitive layers for colour photography.** E. WOLFF-HEIDE (B.P. 340,278, 17.9.29).—A light-sensitive emulsion film is treated successively with colloidal solutions of green and red sensitisers such that the green sensitiser penetrates only as far as the intermediate layers, and the red sensitiser affects only the surface layers, the emulsion near the support remaining unsensitised. The film is then treated with an orange dye capable of absorbing blue and green. By exposure through the support the three-colour-separation negatives are obtained in successive layers of the emulsion film. J. W. GLASSETT.

(A, B) **Production of two-colour pictures by photographic dye-impression methods.** (c) **Dye-printing plate and sensitised material for producing it, for use in dye-impression colour photography.** J. E. THORNTON (B.P. 339,296, 339,319, and 339,321, [A—C] 3.7.29).—(A) Two-colour separation negatives are produced simultaneously upon suitably sensitised emulsion layers coated on either side of a transparent support, selective filters being introduced before the front layer and between the layers. After treatment in a non-tanning developer, the gelatin in contact with the silver is rendered insoluble by the action of an oxidising bath, and the negative is fixed, washed, and dried. (B) Sheets of the positive material, consisting of a support, with perforations for registration, coated with a thin layer of dye-absorbing colloid, are placed in firm contact with both sides of the negative, which have been inked up with suitably coloured dyes and, after stripping, are cemented together in register. A suitable duplex dye-printing and registering press is described. (c) Methods for marking duplex dye-plates for two-colour photography are described.

J. W. GLASSETT.

**Production of light-sensitive films on metal, wood, etc.** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 340,246, 20.9.29. Ger., 8.10.28).—Silver bromide- or chloride-gelatin emulsions are dried, ground to a fine powder, and incorporated in a nitrocellulose varnish, which is sprayed over the surface to be treated. To facilitate development and fixing, a small quantity of a solvent for the lacquer, e.g., amyl acetate or ether, is added to the baths.

J. W. GLASSETT.

**Mordanting of photographic silver images.** F. LIERG (B.P. 335,930, 2.7.29).—The silver image is converted directly into a transparent silver iodide-metal iodide mordant by treatment with a solution of calcium, rubidium, or lithium iodide containing potassium ferri-cyanide. For direct positives the silver chloride or bromide is similarly converted by solutions containing calcium or potassium iodide and urea or thiocarbamide followed by the removal of the developed silver by known methods. J. W. GLASSETT.

**Printing processes employed in colour photography.** D. A. SPENCER, and COLOUR PHOTOGRAPHS (BRIT. & FOREIGN), LTD. (B.P. 340,605, 3.10.29).—In processes of the "carbon" and "carbonyl" types, a sheet of regenerated cellulose, *e.g.*, "cellophane," is used as a developing support and final support for the pigmented gelatin images. J. W. GLASSETT.

**Preparation of multicolour motion pictures.** I. G. FARBERMIND. A.-G. (B.P. 335,432, 11.11.29. Ger., 12.11.28).—In the preparation of colour films for additive synthesis, partial colour-record images of reduced area ( $< 19 \times 25$  mm.) are produced in recurring sequence upon sub-standard film and developed to positives by a reversal method. J. W. GLASSETT.

**Photographic processes.** W. SMITH and J. E. BRAMWELL (B.P. 340,775, 28.12.29).—Negatives having an opaque base are glazed by known methods in order to increase the depth of image of positives prepared by reflection printing. J. W. GLASSETT.

**Reproduction of images from lenticulated films.** TECHNICOLOR MOTION PICTURE CORP., ASSEES. of H. T. KALMUS and J. A. BALL (B.P. 343,369, 2.4.30. U.S., 3.4.29).

**[Photographically] reproducing imitation wood-graining, marbling, or like markings on surfaces.** F. E. LANGDON (B.P. 342,765, 7.11.29).

## XXII.—EXPLOSIVES; MATCHES.

**Manufacture of nitrocellulose with the highest possible nitrogen content.** F. LENZE and E. RUBENS (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 4).—Bleached linters were nitrated with various proportions of mixtures of nitric and sulphuric acids, sulphur trioxide or sulphuric acid containing it, glacial acetic acid and acetic anhydride, nitrogen pentoxide, and phosphoric anhydride. With the last-named mixture, a nitrocellulose containing 13.8–13.9% N was obtained, and by suitable pretreatment of the cotton it is possible to reach 14%. Almost equally high nitrogen contents are obtained when certain collodions or guncottons are nitrated, especially if these are dissolved in nitromethane.

W. J. WRIGHT.

**High brisance [of explosives in relation to blasting efficiency and chemical constitution]. III. Dynamite and penthrinite in the plastic and frozen states.** A. STETTBACHER (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 8–10).—Nitroglycerin, nitroglycol, and penthrinite give deeper penetration on iron plates when tested in a frozen condition at temperatures not below  $-40^{\circ}$  than when tested in their normal state, but blasting gelatine shows no appreciable difference. At  $-75^{\circ}$  to  $-80^{\circ}$ , however, nitroglycol explodes with only a mild report and leaves no impression, nitroglycerin behaves the same as at  $-40^{\circ}$ , penthrinite gives an increased and blasting gelatine a retarded effect. The superiority, as regards brisance, of ammonium penthrinite over blasting gelatine, both being in the plastic state, is not very apparent on rolled iron plates, but is accentuated when steel plates are used. It is evident that

collodion, when mixed with glycerin or glycol nitrates, retains its macro-structure, even when the explosives are frozen, and retards the explosion wave, and the only way to ensure complete detonation is to incorporate a suitable amount of a crystalline compound, such as penthrinite. The high sensitiveness to detonation of a mixture of toluene and tetranitromethane is discussed.

W. J. WRIGHT.

**Water in glycerols.**—See III.

## PATENTS.

**Treatment of black powder.** H. E. ELLSWORTH, ASSR. to ENSIGN-BICKFORD Co. (U.S.P. 1,770,482, 15.7.30. Appl., 16.11.27).—The powder is treated with a solution of nitrocellulose and the volatile solvent then evaporated, so that combustion is retarded, but remains uniform. W. J. WRIGHT.

**Detonating firework composition.** W. F. GEHRIG, ASSR. to ESSEX SPECIALTY Co., INC. (U.S.P. 1,771,116, 22.7.30. Appl., 12.3.29).—A non-hygroscopic firework for producing successive explosions comprises a chlorate, a compound of sulphur and phosphorus, inorganic binding agents, and a dispersive agent, a suitable composition being potassium chlorate 25, phosphorus sesquisulphide 8, magnesium oxide 35, chromium potassium sulphate 5, ferric oxide 5, and gum arabic 2 pts. W. J. WRIGHT.

## XXIII.—SANITATION; WATER PURIFICATION.

**Behaviour of the catalyst in carbon monoxide filters.** G. STAMPE and F. BANGERT (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 24–26).—An investigation was made of the absorption by the Dräger filter of gases and vapours other than carbon monoxide, using an air current of 30 litres/min. and a gas concentration of 0.1%. The amounts absorbed were: phosgene 3.4, chlorine 7.2, hydrocyanic acid 3.3, sulphur dioxide 18.5, hydrogen sulphide 19.9, ammonia 2.3, carbon tetrachloride 8.0, and benzene 10.0 g. The corresponding figures for the life (min.) of the catalyst were: 27, 81, 98, 232, 467, 108, 41, and 103. In further tests with air of 30% humidity, containing small proportions of carbon monoxide and various "poison" gases or vapours, the effect on the catalyst was greatest with chlorine, followed by sulphur dioxide, hydrocyanic acid, hydrogen sulphide, and water vapour in decreasing order of magnitude, the proportional amounts that cause the same poisoning being 10, 23, 25, 58, and 150, respectively. The results are plotted on curves.

W. J. WRIGHT.

**Chromium-plating baths.**—See X. **Base-exchange material.**—See XVI.

See also A., Mar., 327, **Magnesium in water.** 396, **Thiocyanates.** **Oligodynamic dilutions.**

## PATENTS.

**Water-softening apparatus.** UNITED WATER SOFTENERS, LTD., R. T. PEMBERTON, and H. S. LAWRENCE (B.P. 342,772, 8.11.29).

**Carbon monoxide.**—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

APRIL 24 and MAY 1, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

#### High-speed dispersing machines [colloid mills].

W. GROHN (Chem. Fabr., 1931, 1—4, 13—15, 27—28).—The pioneer work of Plauson is described. Colloid mills fall into two classes: (1) beater mills, such as the original Plauson mill; (2) mills in which the dispersion is forced through a slit as a thin film by high-speed rotors. Several mills of the first class, including some operating by centrifugal force, are described. Those of the second class (Premier mill and others) are especially suited for the preparation of emulsions. The improvements made in the Plauson mill are chiefly concerned with the increase of speed and reduction of power requirements. The eccentric position of the beaters was retained, although it involved a circulating of the liquid within the mill, which absorbed power. In the Oderberger colloid mill, however, the liquid is immediately removed by a pump, the action of the beaters being merely dispersive. The various theories of the action of the beater mill, as proposed by Plauson, Block, and others, are reviewed and criticised. Travis' view, that colloid mills only deflocculate secondary aggregates without breaking up large primary particles, is not accepted. The "vacuum dryer colloid mill" effects improved results by forcing the liquid in counter-current to the direction of movement of the beaters, and by the use of subsidiary additional beaters. Probably dispersion is a function of relative velocity of beaters and liquid, and of the number of impacts. C. IRWIN.

**Viscous flow and surface films.** R. BULKLEY (Bur. Stand. J. Res., 1931, 6, 89—112).—From a repetition of the experiments of Wilson and Barnard on the flow of oils containing fatty acids through capillary tubes (B., 1922, 929), it appears probably that the clogging up of the capillaries observed by these authors was due to the presence of foreign particles in the oil, for when the liquids are efficiently filtered clogging does not occur. The flow of various liquids in platinum and glass capillaries with internal radii as small as 9.35 and 5.59  $\mu$ , respectively, has also been investigated. The radius of a given capillary, as calculated from the rate of flow by Poiseuille's equation, is found to be the same for all the liquids flowing through, and in the case of the smallest glass capillaries the calculated radii correspond with the radii measured directly. Further, for a constant pressure difference between the ends of the capillary, the rate of flow does not vary with the time, but is proportional to the pressure in all cases. It is therefore concluded that if there is any motionless adsorbed film on the walls of the capillary its thickness

cannot exceed 0.02—0.03  $\mu$ , and that the liquid has its bulk viscosity to within this distance from the wall.

R. CUTHILL.

**Theory of rectification in packed columns.** E. KIRSCHBAUM (Chem. Fabr., 1931, 38—40, 51—53, 63—65).—A mixture of two liquids of different b.p. undergoing distillation is considered. If  $x_v$  is the percentage number of mols. of the more volatile component present in the vapour and  $x_l$  that in the liquid at any point in the column, then  $x_v = vx(v+1) + x_D(v+1)$ , where  $x_D$  is the content in the product and  $v$  the ratio of condensate to distillate, if fractionation is complete. This line is plotted, and the effect of packing considered graphically. The case of partial condensation is similarly treated. A packed column is considered as providing continuous contact between liquid and vapour having temperature differences corresponding to the differences between the b.p. curve and condensation curve. If  $\delta$  is this difference,  $\alpha$  the heat transmission between liquid and vapour,  $R$  the latent heat of evaporation, and  $F$  the heat-exchange surface, then the rate of condensation  $dC = dF\alpha\delta/R$ . The value  $F$  depends on the dimensions of the packing and the height of the column, and may be replaced by  $KdH$ . From this an integral equation is obtained, which gives a relation between the height of the column and yield of distillate, and is capable of graphical solution. The quantity  $\alpha$  is dependent on the form of packing, vapour velocity, and reflux ratio, and requires determination for each case. It also, of course, varies with the vapour concentration. The case of a benzene-toluene mixture is taken as an example, and the necessary curves are worked out both for a simple column and dephlegmator, and for continuous still with distillation and rectification columns. C. IRWIN.

**Behaviour of moulds on expansion [insulating] corks.** J. GREGER (Z. Unters. Lebensm., 1930, 60, 532—536).—Crude cork (e.g., outer bark and stopper-manufacturers' refuse) used as an insulator may be infected by bacteria and moulds (*Penicillium*, *Mucor*, *Aspergillus*, *Dematium*) which can develop subsequently under favourable conditions owing to the presence in the cork of the necessary nutrient substances (primary infection). The action of hot air for 2 hrs. at 100° or 30 min. at 150° destroys the spores, but secondary infection may occur subsequently if temperatures below 250° are used. Hot air at 260° for 10 min. results in complete sterilisation, owing to the destruction of the nutrient substances. J. GRANT.

**Boiler water.**—See XXIII.

\* The remainder of this set of Abstracts will appear in next week's issue.

## PATENTS.

**Apparatus for preparing [roasting] granular or like materials.** J. L. ROSENFELD (U.S.P. 1,773,202, 19.8.30. Appl., 21.2.27).—Material, such as coffee, peanuts, etc. is roasted while in contact with a heat-carrying agent such as sand. The sand is preheated, and the drum which conveys the material is also heated throughout its length. B. M. VENABLES.

**Tunnel drying ovens.** AKTIEBOLAGET SVENSKA FLÄKTFABRIKEN (B.P. 342,225, 7.5.30. Swed., 7.5.29).—The drying air is circulated transversely to the goods by means of a number of fans, but its general motion is longitudinally countercurrent to the goods. When approaching the inlet (for the goods) the air passes over heaters during the transverse circulation, so that it arrives at the inlet end in its hottest and moistest condition; thence it is led back about half way, and passes over heat exchangers (in the place of the positive heaters) moving concurrent to, but out of contact with, the goods and giving up considerable heat of condensation to fresh air circulating transversely in the cooler and drier end of the kiln. External heat exchangers and fans are also placed at one or more alternative points. B. M. VENABLES.

**Introducing gaseous, vaporous, or liquid agents into a melting bath of high temperature.** F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 341,873, 21.10.29. Ger., 11.6.29).—Gaseous or liquid addition agents are supplied continuously through a pipe which is periodically raised out of the molten bath before its critical period is reached. A protective layer of the molten mass is produced by cooling, and this is aided if the pipe is wrapped with asbestos cord. C. A. KING.

**Vertical water-tube boiler for waste heat, especially of water-gas.** DAMPFKESELSFABR. VORM. A. RODBERG A.-G. (B.P. 342,201, 22.3.30. Ger., 23.3.29).—A nest of vertical tubes substantially fills the space within a vertical cylindrical stack, the tube ends being connected to steam and water drums arranged horizontally at the top and bottom. B. M. VENABLES.

**Apparatus for recovery of chemicals and heat from waste liquors.** C. L. WAGNER, Assr. to C.L.W. PATENTS CORP. (U.S.P. 1,771,829, 29.7.30. Appl., 24.1.25).—A vertical retort with an outer airtight casing and an inner refractory lining is divided into upper and lower communicating zones, each having adjustable inlets for air. After heating the retort to the requisite temperature by burning downwardly-directed jets of atomised oil, the oil supply is replaced by the waste liquor (e.g., paper pulp waste products), which is then atomised and burned partly in the upper and partly in the lower zone, whilst the waste gas, the heat of which is utilised in a suitable manner, and the non-gaseous residue (e.g., sodium carbonate) are withdrawn through outlets in each zone. F. R. ENNOS.

**Steam-heated heat exchangers.** SCHMIDT'SCHE HEISSDAMPF-GES.M.B.H. (B.P. 341,767, 7.3.30. Ger., 25.4.29).—A re-superheater or other type of apparatus comprising a number of U-tubes arranged lengthways in a drum has the outlet or lower header rigidly connected to the drum. The steam is led to the upper header by a

pipe of some length and having bends to introduce flexibility. B. M. VENABLES.

**Centrifugal dryer.** L. B. GREEN, Assr. to BORDEN CO. (U.S.P. 1,772,863, 12.8.30. Appl., 25.3.27).—A detachable perforated basket is placed within an imperforate, slightly conical one, the latter being provided with heavy truly balanced weights and attached to the driving shaft by means of a hollow conical boss jammed on to a lump of rubber or other resilient material on the upper end of the shaft. B. M. VENABLES.

**Drying apparatus.** L. N. LLOYD, and PHILLIPS ENG. CO., LTD. (B.P. 341,407, 3.8.29).—A form of air-lock, suitable for charging a drying chamber operating under vacuum or pressure, is described. (Cf. B.P. 336,602; B., 1931, 2.) B. M. VENABLES.

**Drying apparatus.** O. PFEIFFER (B.P. 341,587, 31.10.29).—Hot gases are deflected upwards from a flue into the drying apparatus and down again into the flue by suitable flap doors. The dryer comprises deep narrow pockets alternately for goods and flue gases. B. M. VENABLES.

**Steam dryer, or grit and dust extractor from air or gases and the like.** F. K. O. MOYNAN (B.P. 341,532, 18.10.29).—A stationary apparatus of the whirl-type is described. B. M. VENABLES.

**Dryers and the like [for sheet material; e.g., wall-board].** COE MANUFACT. CO., Assees. of R. C. MOORE (B.P. 340,510, 20.8.29. U.S., 16.2.29).—A dryer of the type described in B.P. 314,013 etc. (B., 1931, 139) is provided with extensions forming sealing chambers at the inlet and outlet ends, the atmosphere within which is maintained by fans exactly equal in quality and pressure to that within the dryer at the respective ends. B. M. VENABLES.

**Drying of hides, skins, leather, plywood, etc.** J. J. BURTON (B.P. 341,932, 19.8.29).—Heated and conditioned air is admitted to a drying chamber, from a longitudinal duct underneath, through pipes which are kept swinging transversely. The air is exhausted from the apex of the roof by means of a fan. A suitable heater for the air is described. B. M. VENABLES.

**Crusher.** G. E. CHAMBERLAIN (U.S.P. 1,773,476, 19.8.30. Appl., 7.3.25).—The apparatus comprises two chain belts running towards each other down the sides of a V. B. M. VENABLES.

**Hammer crusher.** W. A. BATTEY, Assr. to PENNSYLVANIA CRUSHER CO. (U.S.P. 1,772,533, 12.8.30. Appl., 25.8.28).—The upper part of the grid of a crusher of the disintegrator type is formed of spaced bars through which the hammers intercalate; the bars also form the bottom of the feed hopper and pivot on a strong pin at one end, and are secured by weak shear pins at the end to which the rotor runs upwards, so that they will yield to uncrushable matter. B. M. VENABLES.

**Hammer mills.** C. NIELSEN (B.P. 341,824, 15.5.30).—Swing hammers for disintegrators are formed as hollow castings hollowed out more at the pivot end so as to reduce scrap material. The pivots are placed in eccentric bushings to restore the radius of worn hammers. B. M. VENABLES.

**Ball mill.** A. A. WISE and G. GATES (U.S.P. 1,772,737, 12.8.30. Appl., 28.5.28).—The cylindrical shell has one closed end to which is attached the driving shaft running in a spherical bearing. The mill has one tyre, and the distance between the supporting rollers is variable, to alter the inclination of the mill to the horizontal. B. M. VENABLES.

**Grinding and separating mill.** E. JÜNGELS, Assr. to SCHNELLPRESSENFABR. A.-G. HEIDELBERG (U.S.P. 1,773,228, 19.8.30. Appl., 5.11.28. Ger., 8.11.27).—A disintegrator is provided with means to create a strong air current, and below it is a collecting chamber for coarse material. The air-borne fine material passes sideways through a screen to a bag filter or other collecting device, whence the air is returned to the mill. B. M. VENABLES.

**Apparatus for disintegrating ore and other material.** HARDINGE CO., INC., Assees. of H. HARDINGE (B.P. 342,130, 27.1.30. U.S., 30.1.29).—A mill of the Hardinge type, instead of being cylindro-conical, is formed of three conical portions. The feed end is as previously constructed; next, abutting base to base, is the main portion, of length greater than the maximum diameter, which has a comparatively slow taper; and lastly, there is the outlet cone of about the same angle as, but of lesser diameter than, the inlet end. The angles indicated in the figure are  $60^\circ$ ,  $7\frac{1}{2}^\circ$ , and  $60^\circ$ , respectively, with respect to the axis. B. M. VENABLES.

**Production of granular substances.** O. H. BUSE, Assr. to GRASELLI CHEM. CO. (U.S.P. 1,773,257, 19.8.30. Appl., 13.2.28).—The substance while in a liquid condition is sprayed upwards into a relatively stagnant body of air which is surrounded by rapidly moving air currents. The process is particularly suitable for the granulation of sodium bisulphate. B. M. VENABLES.

**Mixing of granular or like materials.** F. KRUPP GRUSONWERK A.-G. (B.P. 341,821, 10.5.30. Ger., 11.5.29).—A rotating drum contains a receiving chamber and a discharge chamber in line around the shaft, the former being of large capacity and effecting preliminary mixing so that in continuous work comparatively large portions of each constituent may be added; outside these chambers are annular passages having internal blades inclined alternately from the opposite walls. Only one entrance port is provided from the preliminary chamber into the bladed chambers so that, with slow rotation of the drum, the final mixing is effected in small batches. B. M. VENABLES.

**Mixing of powdered material.** P. GOEBELS, Assr. to G. POLYSIUS A.-G. (U.S.P. 1,773,619, 19.8.30. Appl., 3.1.29. Ger., 9.12.27).—The constituents are stored in separate pressure-tight containers; a conveying pipe is connected through individual valves to the lower end of each container and runs round to the top of every container, again with a valve to each; there is also a valved outlet to an external place situated where the conveyor pipe turns upwards. The contents of a group of containers, after loosening, are blown by compressed air into another group, and the mixture thus formed is blown to the external place by means of the residual pressure. B. M. VENABLES.

**Filtering liquids.** N. E. NILSSON (B.P. 341,645, 3.12.29. Swed., 4.12.28).—The water or other liquid is passed through a preliminary strainer which hangs more or less vertically over a collecting chamber for coarse material and is kept clean by means of scraping laths. The strained water then passes horizontally through a finer filter which is hinged at the top end and at the lower end is given a motion, by means of a cam and spring, which is slow in the direction counter-current to the water and rapid in the opposite direction, overtaking the water; any collected matter is thereby dislodged and falls into the collecting pit. B. M. VENABLES.

**[Spray separator for] evaporators.** S. VON LE JUGE (B.P. 341,751, 17.2.30).—The dome for separation of entrained liquid from vapour is entirely external to the evaporator and is provided with a separator, comprising filling material sandwiched between two perforated plates, which is capable of being flushed with live steam, the washings being led to waste or back to the evaporator feed. B. M. VENABLES.

**Apparatus for treating liquids.** A. JENSEN (U.S.P. 1,772,898, 12.8.30. Appl., 20.4.26).—A heater or cooler of the rotating-coil type is described. B. M. VENABLES.

**Countercurrent contacting apparatus.** C. L. CAMPBELL, Assr. to E. B. BADGER & SONS CO. (U.S.P. 1,773,258, 19.8.30. Appl., 2.2.27).—In a bubble tower the trays are provided with baffles (for the liquid) across the greater part of a diameter and the downflows to and from any plate are at opposite sides of the closed end of the baffle, so that the liquid has to take a U-shape or rotary course. Successive trays are assembled at a slight angle to each other so that the direction of rotation of the liquid is the same on every tray. B. M. VENABLES.

**Catalytic converter.** D. F. SIRDEVAN, Assr. to GEN. CHEM. CO. (U.S.P. 1,773,322, 19.8.30. Appl., 12.3.26).—The apparatus comprises a vessel provided with spaced, perforated, and firmly secured diaphragms upon which is placed a medium, such as asbestos, that must not become compacted. B. M. VENABLES.

**Adsorption of gases.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, Assees. of SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (B.P. 342,242, 16.6.30. Fr., 17.6.29).—In a process involving the adsorption by solids of the less volatile constituents of a gaseous mixture, the gases are placed in contact with the adsorbent while still under pressure, but after cooling by heat exchange with the expanded remaining gas. B. M. VENABLES.

**Absorption apparatus.** A. GODEL, Assr. to SOC. DE RECHERCHES & D'EXPLOIT. PÉTROLIFÈRES (U.S.P. 1,773,224, 19.8.30. Appl., 19.8.27. Fr., 21.3.27).—The absorbent material is contained in an annular space within a cylindrical container, the axial space being occupied by a bundle of tubes with a jacket. The entering gases pass in one direction through the tubes and return through the absorbent, a fan being provided to draw the gas through and, if desired, to recirculate a portion. In the jacket is a heating or cooling medium for exact regulation of temperature. B. M. VENABLES.

**Apparatus for liquefying air.** M. HAZARD-FLAMAND (B.P. 341,537, 18.10.29. Fr., 19.10.28).—A system of rectification involving two sections of rectifier and one or two additional compressors is used in conjunction with the liquefaction apparatus described in B.P. 336,798 (B., 1931, 3). B. M. VENABLES.

**Hot-joint compound.** J. C. WASSON, Assr. to PENNSYLVANIA LUBRICATING Co. (U.S.P. 1,770,187, 8.7.30. Appl., 1.2.27).—The composition contains an oil-soluble sodium sulphonate derived from a hydrocarbon oil; materials such as graphite, mineral fillers (zinc dust, red lead, etc.), and sulphite-cellulose waste liquor, paraffin oil, water, etc. may be added as desired. L. A. COLES.

**Checker-work for heat exchangers in furnaces.** F. H. LOFTUS and T. N. KURTZ (B.P. 343,969, 27.9.29. U.S., 31.1.29).

**Refrigerant evaporator for cooling brine.** J. C. and C. R. HOVEMAN and H. L. F. MOUZET (B.P. 343,643, 12.12.29. Fr., 13.12.28).

**Vibrating sifting screens.** F. PIRLOT and H. ELTGES (B.P. 343,616, 29.11.29. Belg., 3.8.29).

**Drum [without corners], more particularly of stainless steel and intended for liquids easily acquiring bad taste.** F. KRUPP A.-G. (B.P. 344,307, 30.5.30. Ger., 7.6.29).

**Rectification of the mixed vapours issuing from the generator of an absorption refrigerating machine.** B. LEHMANN (B.P. 343,931, 25.11.29. Ger., 28.11.28).

**Rotary kilns.**—See IX. **Continuous extraction.**—See XII. **Boiler water.**—See XXIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Continuous verticals.** W. A. CURRIE (Gas J., 1931, 193, 280—281).—The influence of temperature on the carbonisation process and the relation of throughput to rate of carbonisation are discussed. The rate of carbonisation is largely determined by the swelling power and size of the coal, and segregation of the coal of different sizes may occur during discharge of the bunkers. The swelling powers of coals may be rapidly compared by heating  $\frac{1}{3}$  g. of the coal (through 60-mesh) in a vertical silica tube (10 cm. long  $\times$  8 mm. bore, and provided with an asbestos plug to support the coal) to 900°, above the coal being placed a carbon rod 6 mm. in diam. and weighing 2 g. The volume of the coal is observed, the coal heated for 5 min. at 900°, cooled, the resultant coke extracted, and the change in volume expressed as a percentage of the coal volume. The figure obtained represents the swelling power of the coal. Various methods of estimating the throughput of a retort are briefly discussed and an approximate equation enabling it to be calculated is given. H. E. BLAYDEN.

**Humic acid from lignite.** H. STACH (Z. angew. Chem., 1931, 44, 118—120).—Freshly prepared, moist humic acid, carefully freed from electrolytes by dialysis, is completely methylated by a small excess of diazomethane in aqueous alcohol, in contrast to the dried product, which needs repeated treatment. Its carboxyl content may be determined by esterification with methyl-

alcoholic hydrochloric acid, or alkaline hydrolysis of the fully methylated product, the results obtained being concordant with each other and with the value for etherification obtained with methyl iodide and alkali. Slightly lower values are obtained by complete methylation of the debituminised lignite, from which the humic acid is prepared, by diazomethane in ether, or by esterification with methyl-alcoholic hydrochloric acid. Slightly lower values still are obtained by complete methylation or esterification of the residue left by extraction of the humic acids from the debituminised lignite by cold aqueous potassium hydroxide. A number of lignites from different localities are shown to be capable of methylation by diazomethane to a degree but little inferior to that attainable with humic acid; somewhat lower values were given by two samples of bituminous lignite with a superficial resemblance to coal, but a Westphalian coal was almost unacted upon. It is suggested that the behaviour on methylation may serve as a test to distinguish between lignite and coal. This reactivity towards diazomethane is difficult to reconcile with lactonic or anhydride structures, and it is suggested that the ageing process that leads to the change of humic acid into lignite is of the nature of an irreversible dehydration of the humic acid gel. H. A. PIGGOTT.

**Development of Dakota lignite. V. Extraction and study of the benzene-soluble portion.** E. E. HARRIS, C. F. BELCHER, and A. W. GAUGER (Ind. Eng. Chem., 1931, 23, 199—204; cf. B., 1931, 323).—Previous work on the solvent extraction of coal is briefly reviewed. Lignite from the Velva district (N. Dakota) was extracted with benzene under pressure by the methods of Bone and of Fischer. Extraction up to at least 4.3% of the dry ashless coal substance was effected. The composition of the extract, particularly when obtained under pressures up to 17 atm., is analogous to that of montan wax obtained from German brown coal. When treated with aqueous caustic potash as in Bone's method, the soluble portions were found to consist of aliphatic acids and not of phenols; the whole extract was resolvable into waxy alcohols and acids, resins, and compounds of unknown composition. It is concluded that the Velva lignite differs from the Estevan lignite examined by Bone (provided that no decomposition of the lignite occurred with the Estevan lignite) in that the acid portion of the benzene extract consists of aliphatic acids instead of phenols.

H. E. BLAYDEN.

**Density and proximate composition of some Indian durains.** L. L. FERMOR (Rec. Geol. Survey India, 1930, 63, iii, 358—377).—Caking and non-caking durain from various coalfields of Gondwana age contained, respectively: moisture 0.67, 3.76; volatile matter 20.18, 23.45; fixed carbon 44.63, 38.00; ash 34.52, 34.79%, and had  $d$  1.628, 1.656, fuel ratio 2.21, 1.62. Average values for the ash-free caking durain and vitrain were, respectively: moisture 1.03, 1.50; volatile matter 31.05, 32.64; fixed carbon 67.92, 65.86; fuel ratio 2.19, 2.01%;  $d$  1.286, 1.267. Those for non-caking durain and vitrain were: 5.68, 9.46; 36.55, 34.07; 57.77, 56.47; 1.58, 1.68; 1.309, 1.298. Support is afforded for the theory that

vitrain acts as a dispersion medium with ash as the disperse phase.

CHEMICAL ABSTRACTS.

**Spontaneous ignition of Hungarian brown coal.** K. GÄRTNER (Mat. Nat. Anz. Ungar. Akad. Wiss., 1929, 46, 378—406; Chem. Zentr., 1930, ii, 3486).—The ignition depends on the particle size and on the content of unsaturated carbon compounds, and not on the water and sulphur contents. The humus content is not related to the rise of temperature.

A. A. ELDRIDGE.

**Variation of release by gases by preheating coals of different rank.** M. LEGRAYE (Chim. et Ind., 1931, 25, 18—21).—The contents of volatile matter of Liège coals containing approximately the same proportions of vitrain and clarain, and with 7.3—27.6% of volatile matter, were determined with and without an initial heating during which the temperature was raised to 400° within a period of 20 min. Four samples of vitrain (10.2—29.0% of volatile matter) obtained from a lump coal were examined in the same way. Preheating resulted in an increased yield of volatile matter with coals containing less than 15% of volatile matter and a decreased yield with coals containing more than 18%. The vitrain containing less than 20% of volatile matter gave an increased yield of volatile matter after preheating. The influence of oxygen during the preheating and the explanation of the results are briefly discussed with reference to the rank of the coal.

H. E. BLAYDEN.

**Swelling of coal during coking.** K. BAUM and P. HEUSER (Glückauf, 1930, 66, 1497, 1538; Fuel, 1931, 10, 51—64).—To avoid distortion of the coke-oven walls and difficulties in discharging the oven, the swelling pressure developed by the coal during coking should not exceed a certain limit, and should be followed by some shrinkage. A useful laboratory method of studying these qualities was proposed by Korten (B., 1920, 713A), and has been developed by Damm (B., 1929, 382), Hofmeister (B., 1930, 495), and Koppers. In Koppers' apparatus, which is simpler to operate than the others, the swelling takes place under a constant pressure of 1 kg./cm.<sup>2</sup>, whilst Damm and Hofmeister keep the volume of the coal constant by suitably varying the load. A number of tests have been made by Koppers' method, and a new apparatus has been designed based on that used by Damm and Hofmeister. With closer packing of the coal, *i.e.*, with a higher bulk density, the swelling pressure increases. It is not influenced, however, by varying the water content of the coking coal while keeping the bulk density, calculated on the dry coal, constant. Examination of the single-sieve fractions of a coking coal in Koppers' apparatus shows a rise of the swelling curve with increasing grain size; with all grain sizes above 2 mm. the coking qualities appear to be nearly uniform. Fusain shows no swelling tendencies. The expansion curves of the dull coal show very varied values; no real expansion pressure was observed. Clarain develops a dangerous expansion pressure only with a high bulk density. The vitrain proved in all cases to be the source of the expansion pressure. The extent of shrinkage is very different with the various bright coals. An attempt is being made to

correlate the results obtained with Koppers' apparatus and with the modified Damm-Hofmeister apparatus.

A. B. MANNING.

**Complete gasification of small coke.** L. M. WILSON (Fuel, 1931, 10, 69—71).—When working a rotary gas machine on unscreened coke (2½ in. to 0 in.) trouble was experienced due to clinker formation near the walls. The feed arrangement was then modified by providing a shoot having one side lower than the other, and a vertical plate hinged just above the centre line of the bells and capable of being swung across the exit of the shoot, so that the fines, leaving the lower side of the shoot, could be concentrated against the walls of the machine. The walls were also water-cooled and corrugated on the inner surface. With this arrangement the coke was successfully gasified.

A. B. MANNING.

**Formation of fractures in coke.** R. G. DAVIES and R. V. WHEELER (Gas World, 1931, 94, Coking Sect., 20—21).—A miniature oven built for the study of the formation of fractures in coke is described. The coal used is crushed to pass ½-in. mesh and conditions in the oven approximate to those of rapid-coking practice. Photographs of the coke next to the oven walls obtained after coking for 2½, 3½, and 5½ hrs. confirm Thau's observations; the cracks appear immediately after the beginning of the coking process and follow the movement of the plastic layer, their lengths increasing proportionately.

H. E. BLAYDEN.

**Determination of thiocyanates in coke-oven liquors.** A. TRAVERS and AVENET (Compt. rend., 1931, 192, 52—53).—Sulphides are precipitated with cadmium or zinc, and in the presence of several g. of thiosulphates (resulting from oxidation of polysulphides) per litre, the thiocyanates are precipitated in the cold by a 10% excess of copper sulphate, in a medium saturated with sulphur dioxide to ensure dissolution of the double thiosulphates of sodium and copper. After 3 hrs. the precipitate is filtered off, washed, dissolved in an excess of ammonia, and the copper oxidised by boiling with hydrogen peroxide. The cold mixture is titrated in the presence of a 15% excess of sulphuric acid with potassium permanganate solution, whereby the CNS' ion is converted into cyanide, which does not react further, and the sulphur is oxidised (6KMnO<sub>4</sub> ≡ 5CNS). The error is 1%.

J. GRANT.

**The carbon-black flame.** W. B. WEIGAND (Ind. Eng. Chem., 1931, 23, 178—181).—The basic difference between carbon black and lampblack is defined in terms of the method of collection, and possible causes of the unique properties of impingement blacks are discussed. By reference to two types of carbon-black flames (round and flat), the conception of "combustion quotient,"  $s/v$ , where  $s$  is the total combustion surface and  $v$  the volume of gas contained within  $s$ , is developed and applied to variations in flame size and shape, and draught. The position of the impingement surface, the effect of flame, shape of gas flow, and position of channel are factors of importance in large-scale operations that are discussed in relation to the quality and quantity of black obtained.

H. S. GARLICK.

**The tar index in the presence of copper (Kupfer-verteerungszahl).** K. TYPKE (Petroleum, 1931, 27;



Motorenbehr., 4, 3—7).—Investigation of the factors influencing the tar index (Kissling's method) shows that the results are slightly affected by the time elapsing between oxidation of the oil and subsequent testing, and markedly so by the method of extraction of the acidified alkali extract, the amount of oxygen used, and the extent and nature of the copper surface used. The amount of sludge produced is also affected by these factors. The following method is recommended for technical use. The oil (150 g.), contained in a 300-c.c. Erlenmeyer flask, is immersed to a definite and fixed depth in an oil-bath maintained at 120°. Oxygen, purified by passage through caustic soda solution and sulphuric acid, is passed at the rate of 2 bubbles per sec. through a delivery tube opening at 1—2 mm. above the bottom of the flask. An open spiral of clean (unprepared surface), rolled, electrolytic copper foil (made from a strip 100 mm. long, 10 mm. broad, and 0.1 mm. thick) is placed loosely around the delivery tube. After 70 hrs.' continuous heating in the presence of the copper and oxygen, the oil is set aside overnight, well shaken, and 50 g. of it are refluxed for 20 min. with 50 c.c. of a solution made by dissolving 75 g. of pure caustic soda in 1 litre of distilled water and mixing with 1 litre of 96% alcohol. Then 40 c.c. of the filtered alcohol-water layer obtained by separation are acidified with hydrochloric acid, mixed with 50 c.c. of distilled water, and the mixture is thrice extracted with 50 c.c. of pure, residue- and thiophen-free benzene. The combined benzene extracts and benzene washings from the funnel are extracted with 50 c.c. of distilled water, and the benzene fraction is evaporated, the water eliminated from the residue by distillation with absolute or 96% alcohol, dried in the oven at 105° for 10 min., and the cool residue weighed. The amount of tar (in g.) multiplied by 2.5 gives the percentage tar index ("Verteerungszahl"). The amount of sludge formation may be determined gravimetrically by mixing 10 g. of the oxidised oil with 30 c.c. of standard petrol, filtering, washing the separated sludge with cold standard petrol till free from oil, dissolving the sludge in benzene, and weighing the benzene solution as above. Some results of the methods applied to American and Russian oils are given.

H. E. BLAYDEN.

**Wax from low-temperature tar.** J. D. DAVIS and K. M. IRCY (Ind. Eng. Chem., 1931, 23, 186—189).—Wax isolated from low-temperature tar derived from Utah coal proved to consist of a series of hydrocarbons in which penta- (m.p. 54°) and hepta- (m.p. 60°) -cosane predominated. Small amounts of soft wax and unsaturated compounds were also present. The m.p. of the wax (a superior grade of dry wax) was higher than those of marketable petroleum waxes. The physical constants of the crude and refined wax are given. Bituminous coals were found to yield a tar of low wax content as compared with that of the Utah coal and the sub-bituminous coals. Carbonisation with steam tends to increase the wax in a tar.

H. S. GARLICK.

**Distilling naphthenic acids.** I. GUTT, L. IOANESYAN, and G. NOVRUSHEANOV (Azerbaij. Neft. Choz., 1930, No. 9, 138—139).—Crude oils, when distilled in a vacuum or in steam, afford distillates containing naph-

thenic acids having b.p. up to 50° above the b.-p. range of the respective petroleum fractions.

CHEMICAL ABSTRACTS.

**New type of Surakhani crude oil.** L. GUKHMAN and A. PLOTKO (Azerbaij. Neft. Choz., 1930, No. 9, 102—103).—Characteristics of a monoparaffinic crude oil which has a low asphalt content are recorded.

CHEMICAL ABSTRACTS.

**Cracking of hydrocarbons at temperatures higher than critical temperatures.** R. H. MCKEE and A. SZAYNA (Ind. Eng. Chem., 1930, 22, 953—956).—Gasolines, b.p. 120—160°, together with a number of pure hydrocarbons, have been cracked at 405—450°, the process being followed by determination of the critical temperatures before and after cracking for several periods. For paraffin hydrocarbons and straight-run gasolines the change of critical temperature indicates that the cracking process is a linear function. Ordinary gasolines crack at one rate, saturated hydrocarbons somewhat more rapidly, and unsaturated hydrocarbons still more rapidly. The results obtained are in disagreement with a common theory of cracking and with some common beliefs on the mechanism of cracking.

H. S. GARLICK.

**Action of the catalysts ferric oxide and ferric chloride in the reactions of simple hydrogenating cracking of Ragusa oil.** E. SALMOIRAGHI (Annali Chim. Appl., 1931, 21, 27—38).—The Ragusa oil used in the experiments described had  $d_{15}^{20}$  0.9667, flash point 107°, and on analysis gave C 83.46, H 9.14, (O + N) 4.55, S 2.85%; it was subjected to both simple and hydrogenating cracking with and without ferric oxide or chloride as catalyst. The influence of catalysts is similar to that observed by Kling and Florentin (cf. B., 1929, 968). With simple cracking, cracking in presence of ferric oxide, and hydrogenating cracking in presence of ferric oxide, the residue at the first distillation remains identical with the residue of the Engler distillation of the original oil. The cyclic hydrocarbon fraction undergoes condensation and resists the action of the heating. The results obtained on hydrogenation in presence of non-hydrogenating catalysts reveal the influence of the quantity of the catalyst, part of which is consumed by the sulphur and so excluded from the reaction; when the residue is washed with solvents and treated with acid, hydrogen sulphide is evolved. The optimum effect is obtained by hydrogenation in presence of ferric chloride, the percentage of light products obtained being 77.5% and that of residue 11.07%, which is probably able to undergo further hydrogenation. The analytical figures show that the hydrogen tends to displace a large part of the sulphur from the lighter to the heavier fractions. The residues from the hydrogenations in presence of ferric chloride contain no coke and, after being washed with organic solvents, consist solely of catalyst converted into sulphide or oxide.

T. H. POPE.

**Action of alkali hydroxides on elementary sulphur and mercaptans dissolved in naphtha.** V. VESSELOVSKY and V. KALICHEVSKY (Ind. Eng. Chem., 1931, 23, 181—184).—Potassium and sodium hydroxides in the anhydrous state and in solution or suspension in various organic solvents, e.g., absolute ethyl and isopropyl

alcohols, absolute ether, and commercial isopropyl alcohol, remove quantitatively sulphur and mercaptans from petroleum oil. Sodium hydroxide is the more effective for the removal of mercaptans and potassium hydroxide is the more reactive towards elementary sulphur, potassium thiosulphate, mono- and poly-sulphides, and water being the products of reaction between potassium hydroxide in alcoholic solution and elementary sulphur dissolved in naphtha. Higher polysulphides are formed by the direct addition of sulphur to the lower sulphides, the trisulphide being the highest polysulphide found if the reaction mixture has not been agitated. When the reagent is kept in intimate contact with the sulphur solution, potassium penta- and possibly hexa-sulphides are also to be found. The products of reaction between sodium hydroxide in alcoholic solution and mercaptans dissolved in naphtha are sodium mercaptide and water. Keeping any of the precipitated substances in contact with naphtha for a considerable length of time results in some of the sulphur being returned to the oil in elemental form, due to the oxidising effect of dissolved air in the polysulphides and thiosulphate and sodium mercaptide, respectively. H. S. GARLICK.

**Adsorption and base exchange.** P. G. NUTTING (J. Washington Acad. Sci., 1931, 21, 33—36).—Adsorption by sand and clay is discussed, particularly in relation to the filtration of crude mineral oils. Oil sands consist of quartz grains coated with ferrous or ferrous aluminium silicates and finally with hydrocarbons. It is possible to remove the two layers successively. Sea sand may be activated by treating with alkali, acid, and water successively and then drying at 200°. The activated product adsorbs all kinds of positive ions and colloidal aluminium hydroxide. A dry filtering clay gives a white filtrate from black petroleum, but a filter which is not thoroughly dried gives a yellow product.

E. S. HEGGES.

**Synthesis of petroleum hydrocarbons from hydrogen and carbon monoxide at ordinary pressure.**

II. K. KOBAYASHI, K. YAMAMOTO, and H. ISHIKAWA (Mem. Fac. Sci. Eng., Waseda, 1930, No. 7, 26—27).—The synthesis is carried out using cobalt, cobalt-manganese oxide, and cobalt-copper-manganese oxide catalysts. It is considered that cobalt and iron are primary catalysts, manganese oxide and alkalis acting as promoters eliminating carbon dioxide. In addition to the formation of methane carbon monoxide is considered to form carbon dioxide and active carbon, the latter forming ethylene and acetylene by hydrogenation, and, by polymerisation and hydrogenation of the two latter compounds, higher hydrocarbons. The formation of methane cannot be prevented in favour of that of hydrocarbons, as the temperatures of reaction are similar (230—250°).

E. A. RYDER.

**Decolorisation of petroleum with acid clay.**

I—III. K. YAMAMOTO and H. ISHIKAWA (Mem. Fac. Sci. Eng., Waseda, 1930, No. 7, 28—31).—I. The quality and fineness of an acid clay are shown to affect its decolorising power, air elutriation being suggested for use in preparation of the clay. Improved decolorisation results from finer sieving, but fine sieving alone will not render an inferior clay equal in performance to a

good natural clay. Decolorisation is not proportional to the acidity of the clay, but a relation may exist between acidity and fineness.

II. The acidity of acid clay decreases gradually on heating from 100° to 1000°, being practically zero at the latter temperature. The decolorising power of an acid clay is little affected by heating from 100—250°, but decreases from 250° to 600° and falls off much more rapidly from 600° to 1000°.

III. The acidity of clays is determined by direct neutralisation with standard alkali, and also by mixing the clay with standard neutral salt solution, such as an alkali chloride, followed by determination of the hydrogen-ion concentration of the filtrate potentiometrically or by titration with standard alkali. Results obtained by the two methods are identical and the  $p_H$  values of many clays is found to be from 4 to 6. The acidity of clays is not found to increase when treated with various concentrations of hydrochloric acid, but the decolorising power does increase somewhat. The acidity decreases when treated with sodium hydroxide in proportion to the concentration of the alkali up to the point at which the clay becomes neutral, whilst the decolorising power of the clay is decreased by this treatment. The acidity of the clay is reduced to the point of neutrality by treatment with alkali chloride, but the decolorising power is approximately unchanged. E. A. RYDER.

**Determination of the carbon residue from petroleum products.** (Carbon residue by Conradson's method.) B. ROSENBAUM (Chem.-Ztg., 1931, 55, 106; cf. B., 1931, 101).—Conradson's method (of 1927) is modified by the A.S.T.M. specifications of 1930. The deviation of the present values of carbon residues of the standard Gargoyle Mobiloils from values found previously (cf. *loc. cit.*) amounts to about 37% of the experimental figure. The decrease is chiefly due to improvement in quality of the oils and not only to the modified method of determination. A proportionality exists between increase of viscosity and the amount of carbon residue from mineral oils; comparisons of oils on the basis of determination of carbon residue should therefore be made only with oils of similar viscosity. H. E. BLAYDEN.

**Establishing the identity and purity of a hydrocarbon obtained from petroleum.** E. W. WASHBURN (Ind. Eng. Chem., 1930, 22, 985—988).—The evidence in support of the identity and purity of most of the hydrocarbons reported in literature as having been isolated from petroleum is critically examined and found to be inadequate. Suitable tests are described and a procedure suggested in which at least the five properties—refractive index, density, b.p., f.p., and halogenation behaviour—should be reported on all petroleum fractions for which identification is claimed. H. S. GARLICK.

**Composition and recovery of crude benzol produced under different carbonising conditions.** S. TWEEDY (Gas World, 1931, 94, Coking Sect., 15—18).—Analyses, by Colman's method, of benzols obtained from various systems of carbonisation show that variations in composition occur with coke-oven benzol, but to a smaller extent than those occurring in gasworks' practice. Comparison of the composition of crude benzol produced at

different carbonising times on individual plants, and of the product from different types of ovens, shows that the coke-oven benzol is of varying composition, that short carbonising times on the same battery increase the benzene content, and that similar carbonising times on different batteries produce crude benzols of varying composition. Investigations of the factors influencing scrubbing efficiency show that, when using the theoretical quantity of scrubbing oil required to absorb the benzene, satisfactory absorption of benzene, toluene, and solvent naphtha is obtained at temperatures up to 20° with efficient scrubbing. At 30° absorption of benzene becomes inefficient, but absorption of toluene and solvent naphtha is still good.

H. E. BLAYDEN.

**Determination of lead tetraethyl in anti-knock petrols by Vareton's method.** G. WEISS (Petroleum, 1931, 27, 113—114).—The substances in use as anti-knock materials are reviewed and the methods of detecting and determining lead tetraethyl (cf., e.g., Toms and Money, B., 1928, 559) are discussed. Lead tetraethyl may be detected by soaking a filter paper in the petrol, exposing it until dry under the mercury-vapour lamp, treating with dilute acetic acid and then with hydrogen sulphide or potassium iodide. For its determination a method depending on its decomposition by acetyl chloride is recommended. The petrol (100 c.c.) is placed in a glass-stoppered flask, 2—3 c.c. of acetyl chloride and 1—2 drops of water are added, and the mixture is well shaken and allowed to settle. After 4—5 hrs. the lead tetraethyl is thereby completely decomposed, and the precipitate is collected, washed with light petroleum, dissolved in nitric acid, and the lead determined as sulphate.

H. E. BLAYDEN.

**[Mineral oil] sludge determination.** L. GUKHMAN (Azerbaij. Neft. Choz., 1930, No. 5, 97—100).—Errors arise from the high solubility of sulphuric acid in the diluent gasoline; the use of gas oil is recommended.

CHEMICAL ABSTRACTS.

**Air in [Russian mineral oil] refineries.** I. LAPKIN and G. EGOROVA (Azerbaij. Neft. Choz., 1929, No. 12, 76—78).—Still-house air contained 16.7 mg. (max.) of hydrocarbons per litre.

CHEMICAL ABSTRACTS.

**Knock-rating of motor fuels.** SUB-COMMEE. OF INSTITUTION OF PETROLEUM TECHNOLOGISTS (J. Inst. Petroleum Tech., 1931, 17, 69—71).—The report of the Sub-Committee on knock-ratings for 1929 showed that the gasolines sold on the British market gave comparative results on 3 different types of test engines. The extension of this work to cover more types of gasoline and blends showed that fuels rated in terms of pure hydrocarbons gave somewhat poor agreement in the case of straight-run gasolines and pronounced disagreement in the case of non-straight-run fuels. The bouncing-pin method was the most satisfactory for measuring knock-intensity on small engines. On the Horning engines isooctane gives more consistent results than does benzene, and it is agreed that knock-ratings shall in future be expressed in terms of isooctane. Coil ignition is preferable to magneto ignition and, pending the adoption of standard engines, the testing equipment should be such that variable compression and throttling methods are available.

T. A. SMITH.

**Vapour-pressure curves of motor spirits.** P. MEYER (J. Inst. Petroleum Tech., 1931, 17, 42—68).—From collected experimental data it is observed that the vapour-pressure curves of air-free motor spirits are all of the same type, a single curve based on the Ramsay-Young b.-p. law fitting the data. The true vapour-pressure curves agree with an apparent value of Trouton's constant of 17.6. Vapour-pressure curves for pure hydrocarbons and the corresponding equations are given together with curves for air-free motor spirits as determined by four different methods. These results agree with the equation proposed by Bridgeman, Aldrich, and White, viz.,  $\log P = 6.76 - 3.834/T$ , and also with the value 17.6 for Trouton's constant. An alignment chart for use in connexion with evaporation losses, transport, and engine performance of light spirits is given. Available data for latent heat of vaporisation of petroleum fractions are graphed against the 50% distillation temperature. From a knowledge of the mol. wts. of their fractions, the values of Trouton's constant may be calculated; these give a mean of 20.4—the value for pure hydrocarbons.

T. A. SMITH.

**Following combustion in the gasoline engine by chemical means.** L. WITHROW, W. G. LOVELL, and T. A. BOYD (Ind. Eng. Chem., 1930, 22, 945—951).—Measurements have been made of the oxygen concentration in gases withdrawn from the cylinder of a gasoline engine having an improved type of sampling valve, which was located at different places in the combustion chamber and opened at different times during the combustion of the charge; the results are plotted against the respective angles of revolution at which the samples were removed. Under the conditions defined, a narrow combustion zone passes through the charge of gasoline and air in the engine at a finite velocity, which is faster in the middle portions than along the side walls of the combustion space. The speed of the combustion zone increases with engine speed. The forms of the steep parts of the oxygen curve are affected by several factors. Thus the longer the valve remains open the flatter is the curve, the slope of the curve varying with fluctuations in the rate of flame propagation between successive explosions. Other factors are: pressure changes in the combustion chamber during the interval the valve is open, time required for the combustion zone to pass by the sampling valve, rate of combustion of the gasoline in the burning zone, quantity of oxidation taking place in the expansion chamber of the valve itself. The progress of combustion is unaffected by a change in spark timing or by the addition of sufficient lead tetraethyl to the fuel to stop detonation until after it has travelled the greater portion of the distance across the combustion chamber. Knock or detonation is confined to that part of the charge which burns last.

H. S. GARLICK.

**Chemical structure of lubricating oils.** G. H. B. DAVIS and E. N. McALLISTER (Ind. Eng. Chem., 1930, 22, 1326—1329).—A correlation of data on paraffin and cycloparaffin hydrocarbons has indicated that the relation of mol. vol. to mol. wt. offers a means of estimating the relative proportions of these compounds in petroleum products. This relationship has been expressed in the

form of an equation, and is applied to the study of a series of closely cut fractions derived from Pennsylvania lubricating oils. Close agreement has been obtained, using the empirical formula in comparison with C:H analysis. It is shown that the viscosity index of a Pennsylvania oil is a good indication of the relative proportions of the hydrocarbon molecules that are in ring structures or in paraffinic side-chains. The results obtained show that oils of the Pennsylvania type (saturated hydrocarbons) are composed essentially of a grouping of paraffinic chains with naphthene rings.

H. S. GARLICK.

**Determination of the "goudron number" of lubricating oils.** F. CHIERER and J. PRIMOST (Przemysl Chem., 1931, 15, 49—52).—The usual method of determining the above value consists in shaking 10 c.c. of sulphuric acid ( $d$  1.83) with 50 c.c. of oil dissolved in 100 c.c. of light petroleum, and determining the increase in volume of the acid layer. Inexact results are obtained, as a result of differences in the hygroscopicity of the acid used, of occlusion of oil and petroleum in the precipitate formed, and of emulsification of acid in the hydrocarbon layer. These sources of error are largely eliminated by the following procedure: paraffin oil is substituted for light petroleum in the above method, the acid layer is extracted with light petroleum to remove oil and solvent, and this as well as the oil layer are washed with saturated sodium chloride to the disappearance of the reaction for sulphuric acid, which is then determined in the washings. The difference between the amount found and that introduced represents the quantity of sulphuric acid which has reacted with the oil, and this amount expressed as a percentage should be taken as the "goudron number."

R. TRUSZKOWSKI.

**Solubilities of [lubricating] oils and waxes in organic solvents.** II. J. W. POOLE and (in part) F. C. FAHNESTOCK, E. L. KRALL, W. C. MURRAY, and R. M. WILSON (Ind. Eng. Chem., 1931, 23, 170—177; cf. B., 1930, 46).—The solubilities of paraffin wax, m.p. 50°, in light naphtha, b.p. 88—138°, heavy naphtha, b.p. 157—231°, turpentine, *n*-butaldehyde, ethylene dichloride, *n*-amyl alcohol, "pentasol" (a technical mixture of amyl alcohols), paraldehyde, methyl ethyl ketone, and isopropyl alcohol were determined at various temperatures between 0° and 30°. The last two are not completely miscible with mineral oils, and the solubility of a typical petroleum in these is depicted graphically. Further data are given for the ternary system oil-wax-solvent at 0—30° in the cases of solvents having suitable properties for use as de-waxing agents, viz., *n*-butyl alcohol and acetate, chlorobenzene, pentasol, and paraldehyde. Ethylene dichloride also seems of potential value, but was not further studied. In the course of a general survey, approximate ("qualitative") determinations of the solubility of paraffin wax and two typical petroleum in a variety of organic liquids are given.

H. A. PIGGOTT.

**Lubricating oils.** Y. E. EMMUL (Neft. Choz., 1930, 18, 413—419).—The characteristics of a heavy Binagadi asphalt-base crude oil, and of various distillates, are recorded.

CHEMICAL ABSTRACTS.

**Preheating crude oil.** P. VARDIKYAN (Azerbaij. Neft. Choz., 1929, No. 12, 73—76).

**Rectification.**—See I. Friedel-Crafts reaction.—See VII. Absolute alcohol.—See XVIII. Disinfectants.—See XXIII.

#### PATENTS.

**Cleaning of carbonaceous materials.** CLEAN COAL Co., LTD., R. LESSING, and R. H. ALLEN (B.P. 341,863, 16.10.29).—The "float" and "sink" materials from the separation process (cf. B.P. 282,874; B., 1928, 179) are drained to remove surplus bath liquor, treated with dilute wash liquor, and washed by upward or downward displacement of water in a tower. It is claimed that such replacement of the bath liquor results in more uniform and quicker drainage.

H. E. BLAYDEN.

**Separation of mineral materials, especially coal.** R. LESSING (B.P. 341,949, 23.10.29. Addn. to B.P. 287,262; B., 1928, 356).—When coal to be cleaned is moist it is made to fall down a tubular separator against an upward-flowing current of hot air or hot waste gas (at about 400°), which may be introduced at several points in the separator. The coarse material falls to the lower parts of the separator and is removed; the dust and middlings are carried away with the gas stream to an analyser, where they are separated. The coarse material, with or without addition of middlings, is passed to a separator, where rapid cooling and final separation of dust are effected by means of a current of cold air and/or gravity separation.

H. E. BLAYDEN.

**Extraction of hydrocarbons from coal.** F. G. RENOU (B.P. 342,320, 1.10.29).—A mixture of coal and tar is heated in a retort at about 150°, so that only the lighter hydrocarbons present are volatilised. The coal is preferably finely powdered, and the charge is agitated vigorously during treatment. The residue is of an asphaltic nature and suitable for road-making and similar purposes, or for use in electrical insulation.

R. H. GRIFFITH.

**Extraction of resins from coal.** W. D. GREEN, ASSR. to COMBINED METALS REDUCTION Co. (U.S.P. 1,773,997, 26.8.30. Appl., 19.4.29).—Resinous coal is pulverised, pulped with water, and subjected to flotation with higher alcohols (e.g., amyl alcohol), turpentine, cresol, or pine oil as flotation agents, the proportion being 0.6 lb. per ton of coal used. The resin mixed with coal is separated with the froth, which is again subjected to froth flotation, e.g., with amyl alcohol (0.2 lb. per ton of resin-bearing coal) and potash alum (0.4 lb. per ton), to effect the separation of contained coal. The coal residue is used as fuel.

H. E. BLAYDEN.

**Production and utilisation of carbonaceous products for domestic and industrial fuel purposes.** C. G. WODEHOUSE-TEMPLE (B.P. 341,877, 12.7., 6.8., and 2.9.29).—Semi-coke is produced by mixing coal dust (other than anthracite), slack, coke breeze, etc. with not more than 20% of a binding and permeating material such as petroleum-distillation and tarry residues and heating in either static or continuous retorts at not above 550°. [Stat. ref.]

H. E. BLAYDEN.

**Compound fuel.** C. A. A. M. ROUX (B.P. 315,006, 6.7.29. Fr., 7.7.28).—A synthetic fuel for gas producers, more especially for those used in automobiles, is prepared by treating an intimate mixture of vegetable charcoal and mineral carbon (*e.g.*, peat charcoal) with a mucilaginous binding medium (*e.g.*, that obtained from seaweed), together with a suitable iron-containing catalyst, mixing the colloidal mass thus obtained with tar, and then drying, briquetting, and distilling it, the final product being quenched in an aqueous nitrate solution containing molasses, treacle, or syrup. The fuel may be enriched with hydrogen or any hydrocarbon such as methane by treating it under pressure with these gases, which are thereby retained by adsorption.

C. B. MARSON.

**Coke ovens.** KOKSOFENBAU U. GASVERWERTUNG A.-G. (B.P. 341,157, 31.10.29. Ger., 11.10.29).—Regenerators for coke ovens are designed and operated so that turbulent flow is obtained. A fan is used to supply the air for combustion, which is forced through the horizontal, smooth-walled, continuous passages of the regenerator at a speed greater than the critical velocity of about 0.55 m. per sec.; the free cross-sectional area of the regenerator is suitably reduced relatively to the speed of flow. The lower portion of each regenerator is replaced by a passage, which conducts air for combustion from the outside end of the regenerator to its inside end, where it passes through the regenerator and leaves at the outside end of the oven. C. B. MARSON.

**Apparatus for distillation of solid [carbonaceous] materials.** C. P. TOLMAN, ASSR. to PETROLEUM CONVERSION CORP. (U.S.P. 1,773,893, 26.8.30. Appl., 3.6.25).—Hydrocarbon gases, suitably preheated by passage through regenerative hot-blast stoves working in alternation, are passed through a distillation vessel containing the solid materials (*e.g.*, oil shale, coal, lignite), and the products of distillation are collected. The apparatus may be purged of air or other gases by the use of steam. H. E. BLAYDEN.

**Retorts for low-temperature distillation of carbonaceous material.** CARLTON MAIN COLLIERY CO., LTD., R. ADDY, S. GILL, and C. W. TOZER (B.P. 342,104, 7.1.30).—The retort is constructed of two vertical concentric cylinders suitably mounted within a refractory brickwork chamber. The space between the cylinders comprises the chamber in which the material (*e.g.*, coal) is to be carbonised, and is closed at the bottom by means of a door for discharge of the carbonised material. The inner cylinder may be raised to facilitate discharge of the retort, and normally rests upon a gastight seating at the top of the outer cylinder. Provision is made for the escape of the products of distillation through ports arranged above the space containing the charge. Heating of the charge is effected either by hot gases which circulate around the outer cylinder, or by hot gases passing around the outer and inside the inner cylinder. A third central and concentric cylinder may also be added so as to carbonise two charges simultaneously. H. E. BLAYDEN.

**Carbonisation of solid vegetation.** W. CROW, ASSR. to DITTLINGER-CROW PROCESS CO. (U.S.P. 1,773,959, 26.8.30. Appl., 22.6.25).—Carbonaceous products such

as sawdust, coal, plants, etc. are subjected to the action of superheated steam in a vessel allowing continuous descent of the material and ascent of the steam and continuous extraction of the resultant char. The hot gaseous products are exposed to the action of limestone and/or iron ore contained in the vessel, from which they are carried by the steam into a suitable condensing apparatus, where condensible fractions may be collected.

H. E. BLAYDEN.

**Destructive distillation of residues from pulp manufacture.** CORNSTALK PRODUCTS CO., INC., ASSEES. OF E. R. DARLING (B.P. 341,861, 14.10.29. U.S., 13.12.28).—Residual liquors derived from alkaline digestion of cornstalks and the like are evaporated to dryness and distilled under ordinary pressure at 300° to give a condensible distillate, combustible gases, and a residue suitable for the preparation of active carbon. To separate the pentosans present in the liquor, acid may be added to give  $pH$  4.

H. E. BLAYDEN.

**Destructive hydrogenation [of carbonaceous materials].** B. R. GOODFELLOW, L. PATRICK, K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 341,069, 4.10. and 14.12.29).—Hydrogenation is effected by using the hydrogenating gas in a circuit and preventing accumulation of methane, ethane, and nitrogen by their removal by cooling the gas to a low temperature, after the removal of the products condensable at ordinary temperatures. The make-up gas (additional hydrogen required to compensate for that used up in the course of the reaction), consisting of hydrogen in association with methane, nitrogen, and carbon monoxide (*e.g.*, purified coke-oven gas), may be added to the circuit prior to the low-temperature cooling. The gas may be cooled so that the nitrogen is liquefied or, alternatively, so that the ethane is liquefied, but not the methane or nitrogen, the liquid ethane being then used for washing the residual gas. After passing through the hydrogenating converter, and after removal of the easily condensable reaction products (petrol etc.), the pressure on the residual gas is reduced to 15–30 atm., so that at least 90% of the hydrogen can be obtained from the gas by cooling and liquefaction of the other constituents, the purified gas being finally recompressed to the reaction pressure of 200 atm. The make-up gas is added to the circuit after the pressure reduction and prior to cooling.

C. B. MARSON.

**Coking [of coal] at a low temperature.** PATENT-AKTIEBOLAGET GRÖNDAL-RAMÉN (B.P. 342,258, 26.10.29. Swed., 25.5.29).—Coal is heated at 250–300° to remove a portion of the volatile matter which it contains, and its temperature is then raised until coking takes place. Under these conditions a good coherent coke is obtained from a coal which ordinarily gives a friable or dusty residue. The process is carried out in a tunnel oven, through which an endless conveyor moves, with the coal compressed into a number of grooves. Heating is effected by means of preheated circulating gases.

R. H. GRIFFITH.

**Manufacture of granular active carbon.** I. G. FARBENIND. A.-G. (B.P. 341,233, 13.12.29. Ger., 13.12.28).—Carbonised material (*e.g.*, wood charcoal) is mixed with an organic binding agent (*e.g.*, tar or pitch),

a non-hydraulic inorganic binding agent such as clay or kaolin, and an inorganic substance which sinters or melts below 1000°, or which yields by chemical reaction with the non-hydraulic binding agent, during the ignition and activation process, a compound sintering or melting below 1000°, *e.g.*, an alkali, salts of the alkalis or alkaline-earth, or a heavy-metal chloride. The mixture is then shaped under pressure, ignited, and activated with gas or water vapour.

C. B. MARSON.

**Water-gas generators.** STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER, Assees. of SCHEIDHAUER & GIESSENG A.-G. (B.P. 342,405, 13.11.29. Ger., 14.11.28).—Two vertical retorts are arranged above a water-gas generator so that raw coal in the former is carbonised by heat supplied from the latter. During the blow period the producer gas obtained is burned in flues surrounding the retorts, and waste gases escape by way of a steam superheater. When water-gas is being made, part of it is diluted with waste gas so that its calorific value is about the same as that of the producer gas, and it is burned in the same flues. An automatic regulator controls the amount of waste gas that is added.

R. H. GRIFFITH.

**Production of water-gas.** T. MCGREGOR, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 342,229, 9.5.30).—Clinkering in make-and-blow gas generators is reduced or avoided by preparing the coke used from coal in which the natural ash content has been reduced so as to raise the m.p. to at least 1450° (preferably above 1470°, as determined in the presence of moist water-gas). The coal is cleaned by flotation to a predetermined sp. gr.

H. E. BLAYDEN.

**Manufacture of water-gas.** T. W. STONE, Assr. to WESTERN GAS CONSTRUCTION Co. (U.S.P. 1,771,067, 22.7.30. Appl., 24.10.23).—A water-gas generator is so arranged that a large volume of air may be blown downwards through the upper part of the fuel bed and a small volume upwards through the lower portion of the bed. The clinkering of the ash in the lower part of the bed is thereby avoided. After air-blowing, steam is passed upwards through the whole fuel bed.

T. A. SMITH.

**Preparation of acetylene and hydrogen [from methane and other hydrocarbons].** M. BANCK (U.S.P. 1,773,611, 19.8.30. Appl., 3.3.27. Ger., 15.3.26).—Acetylene is prepared by preheating a mixture of methane and an inert gas and passing the mixture (preferably at reduced pressure) over an extensive surface of porous carbon electrically maintained at 1400–2000°. The methane may be obtained by decomposition of gaseous or gasified hydrocarbons.

H. E. BLAYDEN.

**Heat treatment of hydrocarbon gases.** SOC. D'ÉTUDES ET RÉALISATION DITE "ÉREAL" (B.P. 342,034, 18.11.29. Belg., 23.11.28).—Gaseous mixtures containing hydrocarbons (*e.g.*, coke-oven gas) are passed through a vessel containing incandescent fuel (*e.g.*, coke at 800–1200°) into a separate chamber in which separation of the gaseous products and of the carbon produced (as carbon black) by the process is effected. The temperature of the fuel is maintained by periodically blowing air or other oxidising gases, suitably

preheated by heat-recuperative apparatus, through the fuel.

H. E. BLAYDEN.

**Diluting coke-oven gas with flue gas.** W. R. KNAPP, Assr. to SEMET-SOLVAY Co. (U.S.P. 1,772,101, 5.8.30. Appl., 4.12.25).—Part of the coke-oven gas obtained is burned in heating the charge; the flue gases, after removal of sulphur dioxide, are mixed with the gas to reduce its calorific value.

T. A. SMITH.

**Treatment of ammoniacal liquors obtained in scrubbing gases, and recovery of fertilisers and other products therefrom.** C. J. HANSEN (B.P. 342,318, 20.8.29).—Liquors obtained by scrubbing crude gases, such as coal gas, are treated with phosphoric acid for the direct recovery of ammonium phosphate. If sulphites are present, the liberated sulphurous acid is used to scrub a further quantity of gas; when the liquor contains thionates the usual treatment with sulphurous acid is omitted, and phosphoric acid is employed instead. During this stage of the process partial conversion into ammonium sulphate, or into sulphur and sulphurous acid, can be controlled by the amount of phosphoric acid that is added. If the crude gas is washed in two stages, cyanogen compounds that are first removed may be mixed separately with phosphoric acid, or added to the sulphite and thionate solution.

R. H. GRIFFITH.

**Working up spent gas-purification material to pure tar-free sulphur and tar-free purification material capable of being used again.** RUHRGAS A.-G. (B.P. 342,611, 29.4.30. Ger., 1.5.29).—Material contaminated with tarry impurities is treated with benzene or its homologues, in the cold or at a slightly raised temperature. This removes the tarry matter and leaves pure sulphur, to be subsequently extracted by a different solvent at a higher temperature.

R. H. GRIFFITH.

**Manufacture of aqueous dispersions of bitumen.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 342,296, 25.10.29).—Bituminous materials (after being liquefied) are agitated with aqueous solutions of gelatin or other water-soluble dispersing agents which are added in quantities sufficiently small to prevent redispersion of the water-free film derived from the dispersion. Gelation of the dispersion is inhibited by addition to the mixture, before or during dispersion, of water-soluble salts (*e.g.*, sodium or magnesium chloride, potassium iodide, ammonium thiocyanate or sulphate, sodium benzoate or acetate) in amounts of 25–100% of the weight of dispersing agent used.

H. E. BLAYDEN.

**Production of [bitumen-pitch] dispersions.** C. R. MACDONALD, Assr. to FLINTKOTE Co. (U.S.P. 1,774,205, 26.8.30. Appl., 22.7.27).—A suitable mixing apparatus for carrying out the processes described in U.S.P. 1,302,810 and 1,615,303 (B., 1919, 494 A; 1927, 301) is described.

H. S. GARLICK.

**Oil-cracking still.** L. B. CUDDY (U.S.P. 1,760,400, 27.5.30. Appl., 2.7.18).—A vertical cylindrical still, the lower portion of which is conical, is fitted with a vertical shaft carrying scraper blades which remove carbon from the sides of the still. These blades are



pivoted and make contact with the sides of the still by means of weighted levers. The conical portion of the still is fitted with a rigid scraper to advance deposited carbon to the outlet of the still. T. A. SMITH.

**Apparatus for treating hydrocarbon oils.** J. B. WEAVER, Assr. to GYRO PROCESS Co. (U.S.P. 1,763,608, 10.6.30. Appl., 15.6.25).—Crude oil is brought into contact with the vapours from a vapour-phase cracking unit, whereby it is preheated and the temperature of the vapours reduced. It is then passed through a pipe-still in which the temperature is raised sufficiently to vaporise those portions that it is desired to crack, the separation of liquid and vapour taking place in an expander. The vapours, mixed with steam, are then passed through the cracking unit, the tubes of which contain ferric oxide or other catalyst, which has been mixed with plastic material and worked up into bars of star-shaped cross-section. In the cracking tubes the temperature of the vapours is raised to about 555°.

T. A. SMITH.

**Apparatus for treatment of hydrocarbon oils.** W. W. TRIGGS. From PANHANDLE REFINING Co. (B.P. 340,949, 29.6.29. Cf. B.P. 337,380; B., 1931, 54).—A cracking still and a preheating still are arranged side by side in a chamber divided by a refractory partition, with an opening above to allow combustion gases to pass from the cracking still to the preheating still. Each still is separately fired and a stack is provided which communicates by means of a series of spaced adjustable flue openings with the preheating chamber, these openings being arranged to control the fraction of the combustion gases from the cracking still that is drawn over the partition into the preheating still chamber.

H. S. GARLICK.

**Treatment of hydrocarbon oil.** J. DELATTRE-SEGUY, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,774,183, 26.8.30. Appl., 28.1.25).—Oil is continuously passed through a coil disposed within a furnace, where it is heated to cracking temperature, to an enlarged reaction drum where vaporisation takes place. The vapours are passed to a dephlegmator and the condensate is collected. A portion of the hot vapours, equal to about 20% by wt. of the oil being treated, is withdrawn from the system, superheated, and injected into the oil in the reaction drum. A superatmospheric pressure is maintained on the oil undergoing conversion.

H. S. GARLICK.

**Treatment of hydrocarbon oils.** J. B. WEAVER, R. STAHL, and C. R. WAGNER, Assrs. to GYRO PROCESS Co. (U.S.P. 1,763,609, 10.6.30. Appl., 6.6.27).—Oil is heated to a dry vapour state and after the addition of steam is passed through a converter, consisting of a bank of tubes heated in such a manner that an almost instantaneous rise in temperature of 110° is obtained; without this sudden rise coke is produced. The converter tubes are provided with cores of catalyst and the time of contact of the vapours at the higher temperature (about 540°) is 2 sec. The gases enter the tubes at 2000 ft. per min., and from there pass at 5000 ft. to a cooler where the temperature is quickly reduced by means of a cold oil spray. Here again sudden cooling prevents coke formation. The vapours are fractionally

condensed, the heavy distillate being returned to the process. T. A. SMITH.

**Distillation of [hydrocarbon] oil.** J. C. BLACK (U.S.P. 1,771,705, 29.7.30. Appl., 26.10.26).—Crude oil is preheated indirectly in an analyser situated above a bubble-cap tower and is then heated in a heat exchanger, where it meets condensate from the tower. It is then passed through a residuum heat exchanger to a stripping still from which vapours are taken to the main bubble-cap tower. Residue from the stripping still is passed through a heating coil and delivered to the lower portion of the bubble-cap tower, where steam also is admitted.

T. A. SMITH.

**Distillation and cracking of hydrocarbon oils.** D. E. DAY (U.S.P. 1,764,391, 17.6.30. Appl., 21.8.22. Renewed 12.12.27).—The oil to be treated is introduced under pressure at high speed and in one direction through a number of pipes connected in series and mounted to rotate about a centre, the oil being passed progressively from the outer members farthest removed from the centre to the centrally located pipes and moving at a relatively increased speed as it passes lengthwise between a freely movable rod in each pipe. The oil is subjected to heat treatment applied directly to the exterior of the pipes sufficient to effect volatilisation within at least some of the pipes, the heat being applied more intensely to the outer pipes than to those positioned centrally, the increased speed of the movement of the oil in the region of the rod aiding to prevent deposition of carbon.

H. S. GARLICK.

**Cracking of hydrocarbon oils.** H. L. PELZER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,770,287, 8.7.30. Appl., 11.4.28).—Crude oil containing more than 1% S is mixed with caustic alkali and passed through a cracking tube at a velocity of more than 6 ft./sec. The hot oil is discharged into a supply of residual oil and the alkali and solid products are removed with the residual oil without being allowed to pass again through the cracking tube.

T. A. SMITH.

**Cracking of hydrocarbon oils.** E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,773,180, 19.8.30. Appl., 11.6.27).—A charge of oil is circulated in a pressure still from a bulk-supply drum through heating tubes and back to the drum, where a bed of finely-divided fuller's earth is maintained submerged in the oil and extends entirely across the path of the circulating oil. The oil is heated to cracking temperature in the heating tubes. The vapours are removed under pressure from the drum and refluxed in direct contact with raw oil containing finely-divided fuller's earth in suspension. The refluxed condensate and unvaporised raw oil are returned to the still charge circulating from the drum to the heating tubes, whereby the tendency toward the formation of a pitchy crust on top of the earth bed is inhibited.

H. S. GARLICK.

**Treatment of hydrocarbons.** S. C. CARNEY, Assr. to SHELL PETROLEUM CORP. (U.S.P. 1,768,827, 1.7.30. Appl., 23.7.25).—Petroleum mixtures containing low-boiling hydrocarbons are distilled in a rectifying column and the vapours cooled by water under pressure so that a portion of the vapours is liquefied. The residual gases



are refrigerated under pressure so that propane is condensed. The gases which are not liquefied in the refrigerator are partly absorbed in oil, methane and ethane being allowed to escape. The gases distilled from the absorption oil are returned for refrigeration.

T. A. SMITH.

**Treatment of petroleum oil** C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,761,622, 3.6.30. Appl., 21.12.21. Renewed 26.3.29).—Crude oil is passed through a coil in the top of a dephlegmator and is then used directly in the dephlegmator to cool the vapours from an expansion chamber. The oil from the base of the dephlegmator is pumped through a heating coil and thence to the expansion chamber. The vapours from the dephlegmator are passed to a second reflux column, the condensate from which is returned to the top of the first dephlegmator. Valves are interposed in the system so that each part may be maintained under the desired pressure.

T. A. SMITH.

**Preparation of hydrocarbon products.** M. B. HOPKINS, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,767,363, 24.6.30. Appl., 16.8.22).—Hydrocarbon vapours, mixed with air, are passed through a heating zone. The proportion of air is 5–20 cub. ft. per lb. of hydrocarbon. The temperatures used, 150–345°, are below cracking temperature and depend on the nature of the stock being treated, whilst the pressures are not allowed to rise above atmospheric. The products are rich in unsaturateds and of high anti-knock value.

T. A. SMITH.

**Cracking of hydrocarbon oil.** A. D. DAVID, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,774,228, 26.8.30. Appl., 31.5.23. Renewed 25.5.29).—Oil is continuously passed through a coil situated in a furnace, wherein it is heated to cracking temperature, and discharged in a thin film into an enlarged vaporisation zone. The vapours are passed to a dephlegmator into which preheated raw oil is introduced as dephlegmating medium. The uncondensed vapours pass to a primary condenser from which portions of the condensate are returned to the dephlegmator. The remaining condensate and vapours pass to a final condenser and the resulting distillate is collected. The mixed raw oil and reflux condensate from the dephlegmator are forced under pressure through a heat interchanger, wherein raw oil is preheated, to the heating coil. Heavy residue is continuously withdrawn from the conversion zone and passed to a second heat interchanger to preheat further the raw oil prior to its introduction into the dephlegmator. A superatmospheric pressure is maintained on the oil undergoing conversion.

H. S. GARLICK.

**Cracking of hydrocarbon oils.** E. C. HERTHEL and T. DE C. TIFFT, ASSRS. to SINCLAIR REFINING CO. (U.S.P. 1,760,357, 27.5.30. Appl., 5.1.28).—Raw oil is used to scrub the gases produced in cracking operations and is then introduced into the cracking cycle. The reaction chamber is fitted with two dephlegmator columns: through one of these the vapours from the reaction chamber are passed; in the other a portion of the heated oil has its pressure reduced so that the greater part is vaporised and the pitch residue is removed. The

vapours in each column are used to preheat the incoming stock by direct heat-exchange.

T. A. SMITH.

**Cracking of hydrocarbon oils.** E. C. HERTHEL and T. DE C. TIFFT, ASSRS. to SINCLAIR REFINING CO. (U.S.P. 1,773,181, 19.8.30. Appl., 7.6.28).—Hydrocarbon oil is distilled under pressure at cracking temperatures. A portion of the unvaporised oil is withdrawn during distillation and subjected to further vaporisation by its self-contained heat under a reduced pressure. The separated vapours are passed into a tower and condensed by direct contact with fresh oil. The condensate and unvaporised fresh oil are passed to a second tower where they make direct contact with the vapours from the still and from which the reflux and admixed unvaporised oil are returned for pressure distillation.

H. S. GARLICK.

**Vacuum system of [hydrocarbon] oil conversion.** A. E. HARNSBERGER, ASSR. to PURE OIL CO. (U.S.P. 1,763,604, 10.6.30. Appl., 31.5.28).—The oil is cracked in the vapour phase under reduced pressure, thus reducing the time the vapours are exposed to cracking temperature and resulting in the formation of less fixed gas. The vapours, mixed with steam, are passed through a heating element, part of which is exposed to radiant heat, the temperature being rapidly raised from 420° to above 550° and maintained at this temperature for about 2 sec. The vapours are then passed through an ejector where they are contacted with cool oil and thereby quickly cooled to 315°. They are then fractionally condensed.

T. A. SMITH.

**Distillation of [hydrocarbon] oil [from refinery gases].** E. W. ISOM, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,771,409, 29.7.30. Appl., 20.4.27).—The gasoline content of refinery gases is recovered by scrubbing with crude naphtha or crude cracked gasoline. The naphtha or gasoline is then redistilled to obtain an enriched gasoline.

T. A. SMITH.

**Treatment of hydrocarbons.** R. FLEMING (U.S.P. 1,764,296, 17.6.30. Appl., 29.10.23).—A stream of hydrocarbon oil is passed through a furnace where it is heated to about 430° under sufficiently high pressure to prevent decomposition or vaporisation. A portion of the heat in the liquid is then released by successively reducing the pressure and superheating the vapours evolved sufficiently to vaporise a wide range of products while still leaving a saturated hydrocarbon liquid mixed with any asphaltic residues. These are subsequently drawn off and the pressure is released while steam is injected to vaporise the lighter portions therefrom. The vaporised fractions are passed to a fractionating tower where an amount of the heavier vapours equivalent to about four times the amount of pressure distillate is condensed and recycled. The pressure container for the process has a passageway (leading from below liquid level) in which is a reducing-valve device which serves to remove carbon deposited therein as required.

H. S. GARLICK.

**[Heat-economy] treatment of hydrocarbons.** F. E. WELLMAN (U.S.P. 1,772,331, 5.8.30. Appl., 29.2.28).—The energy contained in the gases from pressure stills and similar apparatus is recovered by passing the gases through turbines or other motors. The power obtained

is used to generate electricity, which is used to heat the oil.

T. A. SMITH.

**Conversion of hydrocarbons of high b.p. range into products, especially those boiling at low temperatures.** I. G. FARBENIND. A.-G. (B.P. 335,522, 23.4.29. Addn. to B.P. 296,700; B., 1930, 179).—The initial materials are first subjected to a mild cracking operation so that a minimum amount of benzene is produced and the formation of carbon avoided. In a second stage the residue (oil and carbonaceous deposits) is hydrogenated, preferably in the liquid phase and in the presence of a catalyst (containing molybdenum and chromium). The product is then submitted to a further cracking operation.

T. A. SMITH.

**Control of pyrolytic conversion [of hydrocarbons to lower-boiling products].** F. A. HOWARD, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,760,136, 27.5.30. Appl., 21.5.23).—Oil is pumped through a cracking coil to a reaction chamber from which the vapours are taken to a dephlegmator tower. Condensate from this tower, either hot or cold as required for temperature control, is returned with the new stock to the cracking coil or to the reaction chamber.

T. A. SMITH.

**Cracking of hydrocarbons.** E. C. HERTHEL, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,761,340, 3.6.30. Appl., 27.10.27).—In the cracking of hydrocarbon oils of which not more than 50% boil off up to 315° at atmospheric pressure for production of gasoline, the oil which is difficult to crack is circulated through a cracking system consisting of a heating coil and reaction chamber, the reaction chamber being fitted with a dephlegmator column. An easily cracked oil is added at the dephlegmator column and also sufficient of the difficultly cracked oil to replace losses.

T. A. SMITH.

**Cracking of hydrocarbons.** L. LINK, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,756,563, 29.4.30. Appl., 28.5.23).—Oil is distilled to coking temperature in a shell still and the vapours are passed through a series of fractionally condensing towers. The heavy condensate from the first towers of such series and the lighter condensates from the later towers are collected in separate accumulator tanks and both condensates are passed through separate cracking zones, the effluents from which are returned to the bulk of oil undergoing distillation, through a perforated pipe in the bottom of the still.

H. S. GARLICK.

**Synthesis of hydrocarbon fuels, and cracking and hydrogenation of heavy hydrocarbons.** J. MERCIER (B.P. 312,916, 28.5.29. Belg., 2.6.28).—Hydrocarbon vapours, hydrogen, and carbon monoxide are passed through a retort at about 500° in the presence of a catalyst consisting of iron, nickel, or cobalt turnings. The vapours are then quickly cooled to 180–300° in a dephlegmator and packed reaction vessel, where more hydrogen can be added if necessary. Liquid products condensed in the dephlegmator and reaction vessel are returned to the cracking coil. The carbon monoxide and hydrogen required for the operation are obtained by treating the oil residues at 800–1100° with steam.

T. A. SMITH.

**Condensation of hydrocarbons [by pyrolysis].** IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, J. MCAULAY, and W. FRANCIS (B.P. 342,319 and 342,359, 21.8.29 and 5.6.30).—In the pyrolysis of mixtures of gases containing more than two carbon atoms to produce mixtures of unsaturated and aromatic hydrocarbons, the gaseous mixtures is passed through tubes heated at 1000–1200°. The optimum space velocities vary for the different constituents of the mixture and the best results are obtained by passing the gases through a series of reaction tubes so that the reaction is produced in stages. The first stage is preferably carried out at a lower temperature or higher space velocity than the succeeding stage. The walls of the reaction vessel consist of bonded silicon carbide, bonded graphite mixture, or heat-resisting alloys, and the vessels are lined with an aluminosilicate such as sillimanite to favour production of naphthalene. The liquid products, largely olefines or diolefines if the gases are passed through the reaction zones sufficiently rapidly, are separated after each stage. From a mixture consisting, e.g., of methane 45%, ethane 24%, propane 21%, and butane 10%, a yield of 0.32 gal. of light oil per 1000 cub. ft. was obtained when the treatment was carried out in one stage, but 1.08 gals. when a three-stage treatment was given.

T. A. SMITH.

**Production of aromatic hydrocarbons.** A. S. RAMAGE (B.P. 335,353, 3.9.29).—Gases rich in hydrocarbons of the acetylene series are passed, with a hydrogen-carbon monoxide mixture (e.g., water-gas), over a hydrogenating catalyst (iron) at 540–820° at atmospheric pressure. Paraffin hydrocarbons are first removed from the gases by passing them over heated iron oxide or copper oxide. The operation is carried out in 3-in. tubes, each 10 ft. long, the gas having to traverse 3 lengths, the last of which is at the lower temperature. A suitable mixture of gases per tube-hour is 600 cub. ft. of water-gas and 400 cub. ft. of olefinic hydrocarbon gases (average composition  $C_4H_6$ ). At higher temperatures more gas may be treated. The issuing gases are rich in benzene, toluene, and xylene, and contain less than 1%  $CO_2$ .

T. A. SMITH.

**Vaporisation of heavy oils in the manufacture of oil gas.** C. CHILOWSKY (B.P. 342,447, 4.12.29. Fr., 5.12.28).—An oil-water emulsion, with air, is atomised downwards into a vertical iron tube which is externally heated by means of the gas from a generator. The vertical tube is constructed of U-shaped members welded together, thereby giving a large heating surface.

T. A. SMITH.

**Oil-vapour separation and condensation.** J. E. BELL, ASSR. to FOSTER WHEELER CORP. (U.S.P. 1,764,190, 17.6.30. Appl., 27.8.24).—Heated oil is passed into a vaporising tower fitted with baffles, and the vapours are led through a further series of towers similarly fitted. The unvaporised oil from the bottom of each tower is passed through coils in the bases of the succeeding towers, whereby its sensible heat is used to reboil the condensate collecting in the tower. After passing through all the towers a portion of condensate is returned to the top of the tower preceding that in which it was liquefied for revaporisation.

T. A. SMITH.

**Absorption of gasoline from natural gas.** E. E. AYRES, JUN., ASSR. to B.A.S. Co. (U.S.P. 1,768,521, 24.6.30. Appl., 9.7.25).—Absorbing oil that has been thoroughly stripped is introduced into the second of a pair of absorber towers. Gasoline-bearing natural gas is introduced at the foot of the first absorber and flows in succession through the two absorbers. The oil is passed countercurrent to the direction of flow of the gas, but before passing from the second absorber to the first it is partly stripped to remove the lightest gases, which are unabsorbed by the partly stripped absorbing oil in the first tower, where the heaviest constituents are removed. Stripping is accomplished merely by reducing the pressure at which the oil was maintained in the absorber (usually about 30 lb.) to atmospheric and then advantageously to about 20 in. of vacuum without raising the temperature. The oil leaving the first absorber is saturated with the heavier hydrocarbons, for which it has the greatest selective capacity, and is reduced to atmospheric pressure before passing to the distillation apparatus. H. S. GARLICK.

**Separation of water from petrol and like liquids.** THOMPSON BROS. (BILSTON), LTD., F. S. THOMPSON, H. J. THOMPSON, and J. W. MEREDITH (B.P. 341,070, 4.10.29).—The liquid is fed into a container and caused to pass upwards under a relatively small pressure through a fine metal gauze arranged horizontally. Any particles of water are arrested and fall back into a sump at the bottom of the container, and are allowed to drain off through a valve system controlled by a float which will not float in petrol or other light liquid, but will rise to open the valve as soon as a given depth of water is attained. H. S. GARLICK.

**Manufacture of non-knocking motor fuels.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 341,068, 3.10.29).—Motor fuels having a tendency to knock are blended with hydrocarbons or hydrocarbon mixtures of b.p. not above 180°, obtained by the catalytic reduction of aldehydes or alcohols above C<sub>3</sub>, especially those containing side-chains or branched chains, e.g., the oxygen-containing oily by-products obtained in the synthesis of methyl alcohol from oxides of carbon and hydrogen. H. S. GARLICK.

**Distillation of shale.** R. H. MCKEE (U.S.P. 1,774,391, 26.8.30. Appl., 14.2.22).—Shale is continuously fed to a rotary gas producer by a hopper through a number of downwardly directed retort pipes having feet capable of being reciprocated; such feet are of relatively small cross-sectional area and depend from the hopper into the free space above the rotating fuel bed. Producer gas is withdrawn through the retort pipes in quantity sufficient, in conjunction with the heat imparted by conduction through the walls, to distil off all the volatile portion of the shale. Condensable matter is recovered from the resulting gases through a vapour line connected to the upper portions of the retort pipes, and producer gas is separately withdrawn through a separate exit. H. S. GARLICK.

**Treatment of shale.** J. D. ZIELEY, F. A. RUDOLF, and J. C. RYDER, ASSRS. to PETROLEUM DERIVATIVES, INC. (U.S.P. 1,773,839, 26.8.30. Appl., 15.10.20).—

Finely-divided oil shale and sodium chloride are continuously blown into an elongated chamber near the bottom of which is injected a gaseous fuel mixture proportioned to produce a flame sufficiently hot destructively to distil the oil shale. The vapours evolved during distillation are withdrawn at various levels, cooled, and returned with condensate to the chamber. The rate of flow of shale through the chamber is retarded by injecting countercurrentwise a stream of superheated steam. H. S. GARLICK.

**Coking of [petroleum] oils.** D. R. WELLER and L. LINK, ASSRS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,331, 24.6.30. Appl., 18.9.25).—Crude oil is distilled to coke in a coking still mounted in a furnace having an enlarged vapour dome provided with several vapour outlets, proportioned so as to permit the vapours evolved in the still to be removed as quickly as they are formed when the rate of formation is such as completely to eliminate all the volatile constituents of the oil in 10–18 hrs. With the usual cylindrical type of still, approx. 14 ft. in diam. by 40 ft. long, capable of holding a charge of 40,000–50,000 gals., the dome may be about 6 ft. in diam. and the vapour pipes each 12 in. in diam. The vapours are fractionally condensed to yield naphtha, kerosene, paraffin distillate, etc. H. S. GARLICK.

**Lye treatment of petroleum oils.** C. K. PARKER, ASSR. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,764,117, 17.6.30. Appl., 6.8.25).—The ash content is reduced and impurities are removed by continually mixing the oils with an aqueous alkaline solution and heating the mixture to above 100°, at a pressure sufficient to prevent ebullition of the water, until the required purification takes place. The mixture is then gravitationally separated, while maintaining the raised temperature and pressure, and the treated oil and spent alkali are separately removed. H. S. GARLICK.

**Stable mixture of petroleum hydrocarbons and alcohols.** M. D. MANN, JUN. (U.S.P. 1,774,180, 26.8.30. Appl., 8.6.22).—A mixture comprising a liquid petroleum product, b.p. below 232°, a primary alcohol, and a secondary alcohol as blending agent in proportion sufficient to prevent separation at temperatures below 10° is claimed. E.g., a mixture consisting of gasoline 80%, ethyl alcohol 15%, and sec.-butyl alcohol 5%, containing not more than 5% water, is suitable as a motor fuel, paint (etc.) thinner, degreasing agent, etc. H. S. GARLICK.

**Breaking of petroleum emulsions.** E. E. CLAYTOR, ASSR. to PETROLEUM CHEM. Co. (U.S.P. 1,773,517, 19.8.30. Appl., 31.12.28).—The emulsion is treated with a demulsifying agent comprising a water-soluble salt derived from the reaction for a few minutes at 80–90° between a non-hydroxylated aromatic hydrocarbon, e.g., naphthalene, a saturated aldehydic compound, e.g., formaldehyde, and sulphuric acid, intimately associated in the presence of water with a sulphonated fatty acid. H. S. GARLICK.

**Breaking of petroleum emulsions.** W. S. BARNICKEL & Co., ASSEES. of [A–L] M. DE GROOTE, [A–D, G–I, K, L] L. T. MONSON, [E, F] B. KEISER, and

[J, K] A. F. WIRTEL (U.S.P. 1,766,057—1,766,067 and 1,766,112, [A—L] 24.6.30. Appl., 21.1.29).—Petroleum emulsions of the water-in-oil type are caused to separate into two layers by agitation with (A) waste sulphite-cellulose liquors containing calcium hydrogen sulphite, (B) the magnesium salt of the product obtained by digestion of lignin with sulphite liquor under pressure, (C) a tannin compound, *e.g.*, sodium tannate, (D) a mixture of 15% of waste sulphite-cellulose liquors and 85% of the ammonium salt of the acid formed by sulphonating castor oil, (E) the sodium or ammonium salt of the water-soluble product formed by condensing formaldehyde, naphthalene, and castor oil with fuming sulphuric acid, (F) the ammonium salt of the acid formed by sulphonating a mixture of naphthalene and castor oil, or (G) the ammonium salt of the acid obtained by treating crude petroleum with sulphuric acid and propyl alcohol. Other separation agents are the ammonium salts of the condensation products of: (H) crude petroleum, formaldehyde, and sulphuric acid, (I) crude petroleum, dipropyl ketone, and sulphuric acid, (J) naphthalene, dipropyl ketone, and sulphuric acid, (K) naphthalene, ethylene glycol, and sulphuric acid, or (L) crude petroleum, ethylene glycol, and sulphuric acid.

A. R. POWELL.

**Apparatus for distilling lubricating oils.** A. E. PEW, JUN., Assr. to SUN OIL CO. (U.S.P. 1,761,151, 3.6.30. Appl., 29.5.26. Renewed 21.8.29).—Lubricating oil stock is vaporised under vacuum and passed through a tower packed with Raschig rings. Oil condenses on the rings, the lighter vapours, which are responsible for the bad odour of the oil, passing through the column. The condensed oil is run through a deodoriser consisting of a tower with inclined plates over which the oil flows in thin streams to facilitate the final disengagement of objectionable gases. The vaporiser is heated by mercury vapour.

T. A. SMITH.

**Distillation of [lubricating] oil.** A. E. PEW, JUN., Assr. to SUN OIL CO. (U.S.P. 1,761,153, 3.6.30. Appl., 3.1.27).—Topped oil from which lubricants are to be prepared is mixed with about 1% soda solution (*d* 1.35) under pressure. The pressure is then released so that the water is evaporated and the gas oil removed by distillation under reduced pressure. The lubricating fractions are then refined in apparatus such as that described in U.S.P. 1,761,151 (preceding abstract).

T. A. SMITH.

**Cleansing of [waste lubricating] oils.** J. F. STALLARD (B.P. 342,400, 11.11.29).—On to the surface of the waste oil is sprayed a solution containing 4 oz. each of caustic soda, sodium silicate, and talc powder in 8 oz. of water. The solution is maintained at 17° and circulated by pumping from the bottom of the treatment vessel to the spray above the oil. The impurities collect on the surface of the cleansing liquid.

T. A. SMITH.

**Refining of mineral oils, brown-coal tar oils, tars, waxes, etc.** ORANIENBURGER CHEM. FABR. A.-G. (B.P. 309,042, 3.4.29. Ger., 3.4.28).—The oils or molten waxes are treated with chlorosulphonic acid, only sufficient being used to react with the impurities. After separation the oil is washed and decolorised. The

sulphonic acids are separated from the tar and used for emulsification and other purposes. T. A. SMITH.

**Apparatus for hydrolysing acid sludge.** F. L. MAKER, J. L. COOLEY, and C. F. TUTTLE, Assrs. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,760,246, 27.5.30. Appl., 18.4.27).—Hydrolysis is effected at about 165° and under 50 lb./in.<sup>2</sup> pressure. The reaction is carried on in a vessel fitted with a baffle, from one side of which clear dilute acid can be removed. Tar suitable for fuel is removed from the upper layer on the other side of the baffle.

T. A. SMITH.

**Regeneration of [oil-cracking] aluminium chloride sludge.** E. B. PHILLIPS and J. G. STAFFORD, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,760,962, 3.6.30. Appl., 13.3.23).—In order to recover hydrochloric acid from residues of oil containing aluminium chloride, the sludge is first treated directly with superheated steam, whereby hydrochloric acid and volatile hydrocarbons are removed and a porous mass of aluminium oxide and coke is produced. This mass is dried and partly converted into fuel gas by heating it with air in a gas producer; it is then burned in contact with air and chlorine, whereby anhydrous aluminium chloride is produced. The chlorine is prepared from the hydrochloric acid recovered in the steaming operation.

T. A. SMITH.

**Apparatus for sweating wax or wax mixtures.** BURMAH OIL CO., LTD. From H. L. ALLAN (B.P. 340,993, 2.10.29).—A deep, upright, rectangular vessel having its external walls insulated is completely enclosed and equipped with means for controlling the admission or egress of air. Straight pipes for circulating heating or cooling fluid are arranged in the vessel in superposed transverse rows, each row being stepped in relation to the pipes of the subjacent and/or superjacent rows, the pipes being connected at their ends with inlet and outlet chambers or headers common to a group of pipes, thus dividing the vessel into a number of superposed compartments. Above each partition is a wax support composed of reticulated metal, metallic gauze, etc., supported by corrugations which are formed in and stiffen the bottom of the transverse partition. The bottom of the vessel is dished or cambered downward toward a central charging and discharging duct extending along the bottom of the underside of the vessel and having pipes for circulating heating fluid. The transverse row of pipes follow the camber. Means are provided for supercharging the vessel with wax or wax mixture to compensate for the shrinkage of the original charge on cooling.

H. S. GARLICK.

**Production of cleaning fluid.** B. B. FARRINGTON, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,759,155, 20.5.30. Appl., 1.6.25).—A petroleum distillate (boiling range 105—190°) is mixed with sufficient carbon tetrachloride to raise the flash point to above 43°.

T. A. SMITH.

**Apparatus [burner] for obtaining light and heat by combustion of liquid hydrocarbons.** SCOTT-SNELL LABORATORIES, LTD., and C. and E. SCOTT-SNELL (B.P. 342,749, 23.5.30).

**Burners for pulverised fuel furnaces.** F. KRUPP

GERMANIAWERFT A.-G. (B.P. 343,867, 30.7.30. Ger., 9.8.29).

Water-gas waste-heat. Hot-joint compound.—See I. Hydrocarbon derivatives.—See III. Bituminous pulps.—See V. Road stone etc.—See IX. Black [from peat].—See XIII.

### III.—ORGANIC INTERMEDIATES.

Catalytic synthesis of methyl alcohol under pressure. M. FLEURY (Mém. Poudres, 1931, 24, 10–48).—In a process adopted by the Service des Poudres, a mixture of 2 vols. of hydrogen and 1 vol. of carbon monoxide was subjected at high pressure to a temperature of 300–600° in presence of a suitable catalyst, the velocity of the gas mixture being controlled so as to prevent secondary reactions, *i.e.*, formation of methane and carbon dioxide. A small-scale plant is described. Of various catalysts investigated, the most favourable results were obtained with oxides of vanadium, chromium, and zinc; the effect of the catalyst on the production of higher alcohols is discussed. The yield of methyl alcohol was not affected by the presence of inert gases, such as nitrogen and methane, in the gas mixture. A semi-industrial installation was erected at the Poudrerie de Sevran-Livry, and is described. W. J. WRIGHT.

Gas-volumetric macro- and micro-determination of formic acid. A. HANAK (Z. Unters. Lebensm., 1930, 60, 403–407).—The sample (*e.g.*, fruit juice containing < 30 mg. of formic acid) is distilled in steam and 400 c.c. of distillate are collected, 200 c.c. of which are titrated and the volatile acidity is found, whilst 200 c.c. are neutralised and the esters hydrolysed by the action of an excess of hot alkali. The latter portion is then concentrated, any carbonates are precipitated by zinc sulphate and removed by filtration, and the filtrate is evaporated to dryness *in vacuo* in a boiling tube immersed in calcium chloride solution at 105°. The tube is then connected through a bulb tube to an eudiometer, 5 c.c. of sulphuric acid are pipetted on to the contents, and the mixture is heated slowly (*e.g.*, to 170° for sodium formate), the carbon monoxide evolved being collected over water and the volume measured after making the usual corrections. Then 1 c.c. of  $\text{CO} \equiv 2.056$  mg. of formic acid, the degree of accuracy being 0.1 mg. J. GRANT.

Nitration of diethylene glycol. W. H. RINKENBACH and H. A. AARONSON (Ind. Eng. Chem., 1931, 23, 160–163; cf. B., 1927, 555).—An 80.5% yield of pure diethylene glycol dinitrate (allowing for losses in recovery of spent acids) is obtained by direct nitration with nitric and sulphuric acids at 20–25°. A rise in temperature above 35° is to be avoided, and the product must be thoroughly freed from acid by washing before storage. It may be kept unchanged for a year if the latter precaution is observed. It is toxic when absorbed through the skin or inhaled, the symptoms resembling those of glycerol trinitrate. H. A. PIGGOTT.

Peractivin.—See VI. Organic acids in sulphuric acid.—See VII. o-Dichlorobenzene.—See XII. Quinic acid.—See XIX. Ketones.—See XX.

### PATENTS.

Recovery of oxygen-containing derivatives of unsaturated hydrocarbons from mixtures thereof with other substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 340,984, 30.7.29).—The complex compounds of zinc chloride (*etc.*) with alcohols or organic acids (*e.g.*, ethyl alcohol, acetic acid) form additive products with unsaturated alcohols *etc.* (carveol, crotonaldehyde, cinnamaldehyde in the case of the zinc chloride-alcohol complex; terpineol and the less unsaturated alcohols in the case of zinc chloride-acetic acid complex), and may thus be used for the isolation of these unsaturated oxygenated compounds from brown-coal tar oils *etc.* (*e.g.*, the fraction of b.p. 180–280° or 160–200°, freed first from acids and bases).

C. HOLLINS.

Manufacture of 1 : 1 : 2-[ $\alpha\alpha\beta$ ]-trichloroethane. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 341,781, 21.3.30. Ger., 21.3.29).—Vinyl chloride and chlorine are passed into  $\alpha\alpha\beta$ -trichloroethane (or other solvent) containing a chlorine carrier, especially 1% of antimony pentachloride. C. HOLLINS.

Manufacture of organic esters and acids. IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 341,730, 31.1.30).—The alkyl ester of a carboxylic acid, A, is treated with a carboxylic acid, B, in presence of an esterification catalyst (*e.g.*, sulphuric acid, zinc chloride), whereby the alkyl ester of B and the free acid, A, are produced. The more volatile product may be distilled off as formed. Examples are ethyl acetate with formic acid, ethyl propionate or carbonate with acetic acid. C. HOLLINS.

Manufacture of aliphatic [acetic] acids [from alcohols]. H. DREYFUS (B.P. 341,031, 6.9.29).—Ethyl alcohol (*etc.*) is passed with oxygen, preferably in presence of steam, over an oxide, hydroxide, or carbonate of a metal, other than an alkali or alkaline-earth metal, at 280–320°; or, if an alkali or alkaline-earth oxide, hydroxide, or carbonate be also present, at 230–280°.

C. HOLLINS.

Manufacture of esters [of higher fatty acids]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,158, 31.10.29).—The acid and alcohol are caused to react in the form of mist or vapour; *e.g.*, vapours of olive oil acids and steam meet glycerol vapour in a tube at 220°/50 mm., or olive oil acids and glycerol in equivalent proportions are sprayed with steam into a tubular vessel at 220°/50 mm. C. HOLLINS.

Manufacture of ethers of vinyl alcohol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,074, 7.10.29. Addn. to B.P. 332,605; B., 1930, 981).—The vinyl halide used in the prior process may be produced *in situ*, *e.g.*, from ethylene dichloride or ethyldiene dichloride in presence of sodium ethoxide or alcoholic alkali. C. HOLLINS.

Manufacture of nitrogen compounds [nitriles] having hydrogenated ring-systems. I. G. FARBENIND. A.-G. (B.P. 341,047, 9.10.29. Ger., 15.10.28. Addn. to B.P. 300,130; B., 1930, 549).—An  $\alpha\beta$ -unsaturated nitrile is condensed with aliphatic or hydroaromatic compounds containing a system of conjugated

carbon double linkings. Crotononitrile heated with cyclopentadiene at 150–160° under pressure gives the dicyclic nitrile,  $\text{CH}_2 \begin{matrix} \diagup \text{CH}(\text{CHMe})-\text{CH} \\ \diagdown \text{CH}(\text{CH}\cdot\text{CN})\cdot\text{CH} \end{matrix}$  b.p. 87–88.5°/12 mm. With dimethylbutadiene at 160° crotononitrile yields  $\Delta^3\text{-2:4:6-trimethyltetrahydrobenzonitrile}$ , b.p. 93–95°/13.5 mm. C. HOLLINS.

**Production of imino-ethers.** K. F. SCHMIDT and P. ZUTAVERN (B.P. 341,714, 18.1.30. Addn. to B.P. 331,947; B., 1930, 939).—The intermediate or end-products of the Beckmann transformation of oximes or of *O*-acylated oximes are treated with alcohols. *cyclo*-Hexanoneoxime *O*-toluene-*p*-sulphonate, *e.g.*, when heated with alcohol at 75° for 1 hr., undergoes the Beckmann change and yields the cyclic imino-ether, 2-ethoxy-1:2-dihydrohexamethyleneimine, b.p. 161–165°.

C. HOLLINS.

**Production of wetting, cleansing, and dispersing agents.** J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 341,053, 7.8.29).—Amides of higher aliphatic or cycloaliphatic acids, made in the absence of a metal halide condensing agent from such acids and secondary amines free from alcoholic groups, and in which any aryl or aralkyl groups contain no substituents other than sulphonic groups, are sulphonated. Examples are oleic diethylamide, oleic ethylanilide, oleic diphenylamide, oleic *m*-sulphoethylanilide, and stearic ethyl- $\alpha$ -naphthylamide.

C. HOLLINS.

**Manufacture of [alkylated] indole derivatives.** IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and E. H. RODD (B.P. 341,554, 22.10.29).—Indoles are alkylated in the pyrrole ring by heating at 135–160° with an alcohol and hydrogen halide. 2-Methylindole with methyl-alcoholic hydrogen chloride yields 2:3-dimethylindole and 2-methylene-1:3:3-trimethylindoline.

C. HOLLINS.

**Preparation of pure *i*-menthol.** RHEINISCHE KAMFER-FABR. G.M.B.H. (B.P. 341,654, 6.12.29. Ger., 28.1.29).—The mixture of isomerides is partly esterified (cf. B.P. 297,019; B., 1928, 837) and the esters, preferably after removal of unesterified menthols, are purified by crystallisation, *e.g.*, from alcohol, and hydrolysed.

C. HOLLINS.

**Production of thymol and menthone.** ORBIS PRODUCTS TRADING CO. INC. (B.P. 341,650, 5.12.29. U.S., 5.12.28).— $\Delta^1$ -Menthen-3-one or piperitone is heated with reduced nickel in the absence of hydrogen, whereby an equimolecular mixture of thymol and menthone is produced, from which thymol is removed by dissolution in caustic alkali.

C. HOLLINS.

**Manufacture of 6-arylamino-2-naphthols.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 341,120, 15.10.29).—An *N*-arylated Brönner acid is fused with alkali. 6-Anilino- (m.p. 134°) and 6-*p*-toluidino- (m.p. 144°) - $\beta$ -naphthols, and 6-hydroxy-2:2'-dinaphthylamine, m.p. 168–170°, are thus prepared.

C. HOLLINS.

**Manufacture of diphenyl.** F. X. GOVERS (B.P. 341,024, 7.10.29. U.S., 5.11.28).—Benzene vapour, preheated to 650°, is passed into molten cryolite or

other non-metallic liquid, at 750°. The conversion into diphenyl is about 8%.

C. HOLLINS.

**Hydrocarbons.**—See II. Anthranols.—See IV. Urea.—See VII. Lactic acid for baking etc.—See XIX.

#### IV.—DYESTUFFS.

**Friedel-Crafts reaction.**—See VII. **Pigment fastness.**—See XIII.

#### PATENTS.

**Manufacture of a [vat] dye of the thionaphthen-indoleindigo series.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 341,224, 5.12.29).—6-Chlorothioindoxyl is condensed with 5-bromoisatin  $\alpha$ -chloride (anil etc.) to give a grey vat dye of great fastness to light.

C. HOLLINS.

**Manufacture of vat dyes [from naphthalenetetracarboxylic anhydride and *o*-diamines].** I. G. FARBEIND. A.-G. (B.P. 341,357, 15.4.30. Ger., 15.4.29).—The isomerides produced by the processes of B.P. 237,294, 265,232, and 265,964 (B., 1925, 840; 1928, 225) are separated by dissolution in sulphuric acid or chlorosulphonic acid and fractional precipitation by dilution (*e.g.*, with ice or with sulphuric acid). The *o*-phenylenediamine product gives an orange and a blue-red vat dye; that from 2:4-diaminophenetole gives a red-brown and a violet-brown vat dye.

C. HOLLINS.

**Manufacture of vat dyes from dibenzanthrone and isodibenzanthrone.** S. PERCIVAL. From (A.C.N.A.) AZIENDE CHIMICHE NAZIONALI ASSOCIATE, and BELLONI & COLLI (B.P. 341,229, 10.12.29).—Green to green-blue vat dyes are obtained by treating dibenzanthrone or isodibenzanthrone with sulphur chloride in chlorosulphonic acid or oleum at 60–80°, preferably in presence of boric acid.

C. HOLLINS.

**Dyes, including intermediates, and dyeing [sulphuric esters of anthranols; azo dyes from diazotised aminoanthranol sulphuric esters].** D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 340,983, 1.7.29).— $\beta$ -Acetamidoanthranol, obtained, *e.g.*, by reduction of  $\beta$ -acetamidanthraquinone with copper powder and oleum, is converted into its sulphuric ester with pyridine-sulphuric anhydride and pyridine, and hydrolysed with sodium hydroxide to remove the acetyl group. The resulting  $\beta$ -aminoanthranol sulphuric ester may be diazotised and coupled, *e.g.*, with  $\beta$ -naphthol to give a bluish-red azo dye for cotton, which on oxidation with ferric chloride changes to bright orange; or may be oxidised to a blue vat dye of the indanthrone sulphuric ester type, from which the ester groups may be removed by chromic and dilute sulphuric acids.  $\alpha$ -Acetamido- and 3-chloro-2-acetamido-anthranol behave similarly. The aminoanthranol sulphuric esters dye wool brown from an acid bath and may be oxidised on the fibre to deep brown. Anthrone, 4-chloro-9-anthrone, and 2-sulphoanthrone are also converted into sulphuric esters.

C. HOLLINS.

**Manufacture of trisazo dyes [for cotton].** E. I. DU PONT DE NEMOURS & Co. (B.P. 318,146, 9.8.29. U.S., 28.8.28).—Blue direct dyes of the type: 1:8-amino-naphtholdisulphonic acid (having only one sulphonic

group in each ring)  $\rightarrow$  middle component (having no hydroxyl, sulphonic, or second amino-group)  $\rightarrow$  J-acid  $\rightarrow$  a naphthol or aminonaphthol other than a 2:5-aminonaphthol, are claimed. Examples are: H-acid  $\rightarrow$   $\alpha$ -naphthylamine or cresidine  $\rightarrow$  J-acid  $\rightarrow$  N.W.-acid or H-acid.  
C. HOLLINS.

Lakes.—See XIII. Anti-halation layers.—See XXI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Portable instrument for measuring air-permeability of fabrics. H. F. SCHIEFER and A. S. BEST (Bur. Stand. J. Res., 1931, 6, 51—58).—In the apparatus described, an orifice meter is used to determine the rate at which air flows through a disc or fabric under the influence of a known difference in pressure (up to 1 in. of water) between the two sides. The maximum uncertainty in the value obtained for the permeability is less than  $\pm 2\%$ .  
R. CUTHILL.

Determination of tendering of cotton. R. ELMQUIST (J. Home Econ., 1931, 23, 62—66).—Hägglund's method has been modified. CHEMICAL ABSTRACTS.

Recovery of viscose spinning bath. III. Properties of sodium sulphate. IV. Dehydration of Glauber's salt. V. K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 330—331 B, 332—333 B, 333—334 B; cf. B., 1930, 1022).—III. [With S. MIYOSHI.] The properties of sodium sulphate, *e.g.*, heats of dissolution, hydration, and fusion, sp. heat, equilibrium at the transition point, lowering of the transition point by addition of solute, and the three-component systems  $\text{H}_2\text{SO}_4\text{--Na}_2\text{SO}_4\text{--H}_2\text{O}$  and  $\text{Na}_2\text{SO}_4\text{--H}_2\text{O--(Mg, Zn, Mn, Fe, Cu)SO}_4$ , which have some relation to the new method for the recovery of the viscose spinning bath, are summarised. Sulphuric acid lowers the transition temperature of the decahydrate, whilst its crystallisation temperature is lowered both by sulphuric acid and magnesium sulphate. In normal viscose spinning baths the salt crystallising out on cooling is always the decahydrate, and neither anhydrous sodium sulphate nor bisulphate forms double salts with zinc, iron, manganese, or copper sulphates, these accumulating in the spinning bath.

IV. [With S. MIYOSHI and M. YOSHIDA.] Possible economical methods for the dehydration of Glauber's salt include dehydration by efflorescence, with ethyl alcohol, with concentrated caustic soda solution (using the transition point), and by addition of solid caustic soda at the transition point. Those involving the use of caustic soda require further study owing to the difficulty of separating the concentrated alkali from the anhydrous salt.

V. [With S. MIYOSHI.] Instead of recovering the sodium sulphate by evaporation and crystallisation, which causes attack of the evaporating pans, evolution of sulphur dioxide and hydrogen sulphide, and caramelisation of the dextrose present, the spinning bath is concentrated by adding anhydrous sodium sulphate, which removes water by formation of the decahydrate, cooling the solution, and allowing it to crystallise.  
F. R. ENNOS.

Effect of mild heat-treatment on the chemical composition of wood. L. F. HAWLEY and J. WIERTELAK (Ind. Eng. Chem., 1931, 23, 184—186).—Heating of white ash and Sitka spruce in closed iron tubes at  $138^\circ$  for periods of 2, 4, and 8 days causes alterations in the chemical composition of the wood. With both woods the loss consists chiefly of carbohydrates: in the case of ash, pentosans (72% of the total), and of spruce, the readily hydrolysable hexosans (66% of the total). There is a greater increase in the amount of lignin formed (33%) and a smaller one of alcohol-benzene-soluble material in the case of spruce than of ash, in which the content of alcohol-benzene-soluble constituents is doubled.  
T. McLACHLAN.

Purified wood fibre. G. A. RICHTER (Ind. Eng. Chem., 1931, 23, 131—139).—A study of the physical and chemical properties of purified wood fibre, describing modifications of the various tests for its examination and showing the results obtained on 53 samples. The so-called  $\beta$ -cellulose figure is purely arbitrary, as it contains pentosans, resins, and lignin, and cannot be replaced by the figure representing the 7.14% soda-solubility, since the latter combined with the  $\alpha$ -cellulose figure frequently totals well over 100%. The various cellulose figures require intelligent interpretation if they are to be of assistance; cellulose figures are higher in unbleached than in bleached fibres, due to the insolubility of the ligneous matter in 18% soda, and the proportions of  $\alpha$ -,  $\beta$ -, and  $\gamma$ -cellulose vary according to the pretreatment of the fibre.  
T. McLACHLAN.

Determination of pentosans in pulps by the hydroxylamine method. A. NOLL and W. BELZ (Papier-Fabr., 1931, 29, 33—34; cf. A., 1930, 1304).—The reaction described previously is extended to the determination of furfuraldehyde. Air-dry pulp (5 g.) is treated with 13% hydrochloric acid in the usual way. The distillate is neutralised, 25—50 c.c. of 7% hydroxylamine hydrochloride solution (according to the amount of furfuraldehyde present) are added to the distillate, the mixture being shaken frequently during 1 hr. at room temperature, and the liberated hydrochloric acid is titrated with 0.1N-caustic soda (1 c.c.  $\equiv$  0.0096 g. of furfuraldehyde). It is considered that the usual method of calculating pentosans from the furfuraldehyde content is unsound, and it is suggested that the weight of aldehyde obtained as above from 100 g. of material shall be termed the "furfural number" of that material. The yields of furfuraldehyde obtained by the given method are higher than those by the phloroglucinol method, it being held that the latter is inaccurate owing to incomplete precipitation.  
T. T. PORTS.

Process water in the pulp and paper industry. A. SPLITTGERBER (Papier-Fabr., 1931, 29, 81—84).—Literature relating to the effect of various impurities in process water, permissible limits, and the current methods of filtration and chemical treatment is summarised. Special attention is given to organic impurities, and the oxygen-absorption values of a number of commonly occurring organic substances are tabulated. A bibliography is appended.  
T. T. PORTS.

Manufacture of alcohol from waste sulphite [pulp] liquors. W. SEMBRITZKI (Papier-Fabr., 1931,



29, 86—92).—An historical review of the production of alcohol from waste sulphite liquors in Germany during the Great War. A typical plant is briefly described, and statistics regarding heat-balance and output are given.

T. T. POTTS.

**Mechanical control in the sulphite [pulp] process.** E. M. LEINO (Papier-Fabr., 1931, 29, 55—56).—An apparatus is depicted and described for the semi-automatic determination of the Björkman number of pulp during digestion. Pulp is treated with potassium permanganate solution for a standard time, the excess being automatically titrated with ferrous sulphate.

T. T. POTTS.

**Causes, effects, and prevention of paper-mill slime.** D. K. PATILLO (Cellulose, 1930, 1, 280—284).—Controlled chlorination is advocated as the means of slime prevention, chlorine being admitted at the points of highest chlorine demand, stock and water supply being separately treated. It is claimed that a residual chlorine content of 0.1—0.2 p.p.m. has no deleterious effect on plant. Brief mention is made of the "chlor-amine" process, i.e., treatment with ammonia followed by chlorine, in cases where pure chlorination affects the shade of coloured papers.

T. T. POTTS.

**Alkali-cellulose etc.**—See VI. **Sulphuric acid.**—See VII. **Wool in boards etc.**—See IX.

#### PATENTS.

**Viscose complex.** W. MENDEL, Assr. to S. A. NEIDICH (U.S.P. 1,773,923, 26.8.30. Appl., 23.5.25).—Normal viscose is mixed with less than 5% of trisodium phosphate in order to accelerate ripening.

F. R. ENNOS.

**Diminishing the viscosity of highly viscous cellulose ethers.** DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 342,391, 8.11.29. Ger., 15.4.29).—Diminished viscosity, without change in solubility, is attained by heating the ethers with dilute mineral acids, with aqueous solutions of acid salts, or with acetic acid, in the absence of swelling agents and solvents.

F. R. ENNOS.

**Manufacture of cellulose acetate composition of low inflammability.** KODAK, LTD., Assees. of E. S. FARROW, S. E. SHEPPARD, and H. T. CLARKE (B.P. 342,444, 3.12.29. U.S., 3.12.28).—Phosphoric acid (0.05—0.5 pt.) is incorporated with 100 pts. of cellulose acetate in a solvent or mixture of solvents.

F. R. ENNOS.

**Recovery of cellulose acetate from acetic acid solutions thereof.** KODAK, LTD. From EASTMAN KODAK Co. (B.P. 342,596, 28.3.30).—The solution, e.g., a cellulose acetate reaction mixture, is vigorously stirred into an aqueous precipitation bath containing initially at least 20% by wt. of acetic acid, the water therein being not less than 50% throughout the precipitation; the precipitated ester is separated and washed.

F. R. ENNOS.

**Manufacture of cuprammonium silk by the stretch-spinning process.** HÖLKENSEIDE GES.M.B.H. (B.P. 341,590 1.11.29. Ges., 16.11.28).—The pre-

cipitant is partly de-aerated by passing, while heated above the spinning temperature, over sharp-edged material having a large superficial area, e.g., coke.

F. R. ENNOS.

**Manufacture of artificial silk threads, films, etc. from viscose.** I. J. FRENKEL (B.P. 341,138, 21.10.29. Addn. to B.P. 338,318; B., 1931, 109).—The addition of 0.5% of nitrocellulose (11% N) to viscose solution containing 0.3—0.4% of nitrite increases the oxidising activity of the nitrite.

D. J. NORMAN.

**Production of artificial silk threads from cellulose esters and ethers.** O. VON KOHORN (O. KOHORN & Co.), and H. SCHUPP (B.P. 318,151, 16.8.29. Ger., 28.8.28).—Dispersively acting substances, e.g., water, alcohols, formaldehyde, toluene, ethers, acetic esters, are added to a secondary solution of a cellulose derivative, whereby a solution of uniform and minimum particle size is obtained, which after preheating to 40—50° is dry-spun.

F. R. ENNOS.

**Manufacture of artificial filaments and the like.** BRIT. CELANESE, LTD. From C. DREYFUS (B.P. 342,340, 29.10.29).—Heavy filaments, such as bristles, horsehair, straw, etc., are produced by extruding a solution (with 30—40% of solid constituents) of an organic derivative of cellulose containing 1—12%, relative to the cellulose derivative, of a resin, particularly a synthetic resin, together with a plasticiser, into an evaporative atmosphere or into a liquid coagulating bath.

F. R. ENNOS.

**Manufacture of artificial threads and other products from cellulose.** L. LILIENFELD (B.P. 341,843, 341,899, and 341,930, [A] 20.7.29, [B] 19.7.29, [C] 26.7.29. Addns. [A—C] to B.P. 335,906; B., 1931, 153).—Viscose or a derivative thereof is made to react with (A) monohalogenated fatty acids or (C) trithiocarbonic acid esters, particularly those of di- or poly-hydric alcohols, instead of with halogen derivatives of di- or poly-hydric alcohols as in the parent patent, and the solution of the product is spun as before into sulphuric acid of at least 50% concentration. According to (B) threads produced by the original or later processes are decolorised by treating with a warm or hot solution containing at least 5% of alkali sulphide (calc. as Na<sub>2</sub>S.9H<sub>2</sub>O) or with a hydrosulphide solution of equivalent concentration.

F. R. ENNOS.

**Translucent artificial silk.** H. A. GARDNER (U.S.P. 1,768,401, 24.6.30. Appl., 26.11.28).—Translucent (low-lustre) cellulose ester (nitrate or acetate) artificial silk is manufactured by dry-spinning of solutions of cellulose esters containing dissolved aldehyde condensation products of *p*-toluenesulphonamide in such a way that during the formation of the silk filaments these products deposit or crystallise within the filaments and thus render them opalescent; it is advantageous to add to the spinning solution an organic solvent (other than the chief solvent for the cellulose ester) which may be more or less volatile than the chief solvent, and in which the condensation product is soluble or insoluble, respectively.

A. J. HALL.

**Production of fibre of high  $\alpha$ -cellulose content.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,774,403,

26.8.30. Appl., 16.9.26).—Raw pulp, after treatment with a weak oxidising agent, is digested at a relatively low temperature with a bleaching liquor ("calcium bleach" or sodium peroxide) and an alkali, until the bleach is largely consumed. It is then further digested at a raised temperature in the same liquor, after which the product requires only a final mild bleaching with chlorine.

F. R. ENNOS.

**Refining of alkaline pulps.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,773,320, 19.8.30. Appl., 25.11.25).—Kraft pulp is treated at a consistency of about 5–6% with 100–175% of caustic soda and 5–10% of bleaching powder (or sodium hypochlorite), calculated on the weight of dry pulp, for 5–6 hrs. at the ordinary temperature, and is then washed and bleached in the usual way at a stock consistency of 10–16% with 5–10% of bleaching powder. The resulting bleached pulp contains 93–97% of  $\alpha$ -cellulose, has high strength characteristics (particularly tearing strength), and is suitable for high-grade bond and ledger papers or for the manufacture of cellulose derivatives.

D. J. NORMAN.

**Production of wood pulp.** G. E. RICE, Assr. to CONSERVATION CORP. OF AMERICA (U.S.P. 1,769,189, 1.7.30. Appl., 24.1.24).—Wood is boiled in water containing one or more carbohydrates (sugar, molasses, etc.), sodium fluoride, soda ash, and sodium sulphite, and, after grinding, is bleached, preferably in the dried condition.

F. R. ENNOS.

**Manufacture of pulps comprising fibrous material and heat-plastic waterproofing material [e.g., bitumen].** RICHARDSON Co. (B.P. 341,542, 18.10.29. U.S., 1.11.28).—At least 70 pts. of molten bitumen are mixed with wet paper, equivalent to not more than 30 pts. of dry fibre, until homogeneous; more water is then added partly to break up the adhesive mass, and the pulping is completed with further addition of water in a beating engine.

F. R. ENNOS.

**Manufacture of hollow articles from fibrous pulp.** PULP INDUSTRIES, LTD. (B.P. 341,785, 25.3.30. Ger., 6.7.29).—The heated, gaseous pressure medium, e.g., steam, used in moulding is removed partly through the walls of the mould and partly through an adjustable opening in the latter.

F. R. ENNOS.

**Production of [sulphite] paper pulp [and recovery of acid during the process].** R. B. WOLF (U.S.P. 1,772,792, 12.8.30. Appl., 26.6.29).—Each digester in the system is connected by a separate relief line with a small relief accumulator which is, in turn, connected through a pressure-regulating device to two or more recovery tanks containing raw sulphite liquor. Cooling coils are provided to maintain the liquor in the recovery tank at a definite temperature, e.g., 49°, so that the gauge pressure will give an exact indication of the concentration of the acid. This procedure gives a uniform back pressure in the relief lines of the digesters during the cooking period, and enables the relief liquor and gas to be used in adjusting the fresh acid to a uniform concentration.

D. J. NORMAN.

**Manufacture of paper and the like.** A. E. WHITE. From MEAD RES. ENG. Co. (B.P. 341,226, 7.12.29).—

The raw pulp obtained by the process of B.P. 339,334 (B., 1931, 344), shows reduced chlorine consumption if it is pretreated, preferably during disintegration in the rod mill, with cold dilute ( $\frac{1}{4}$ –1%) caustic soda.

D. J. NORMAN.

**Manufacture of paper.** MEAD PULP & PAPER Co., Assees. of J. J. O'CONNOR and D. F. MORRIS (B.P. 341,733, 4.2.30. U.S., 4.2.29).—Fibrous material is cooked with caustic alkali and the spent liquor therefrom is evaporated to dryness, incinerated, lixiviated, filtered, and causticised with lime; the "lime sludge" precipitate is separated, ground with water in the presence of alum (which neutralises the residual alkali and improves the colloidal character of the resulting suspension), and used as a filler for the paper.

F. R. ENNOS.

**Manufacture of paper products.** E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,771,138, 22.7.30. Appl., 8.7.27).—The fusible resinous reaction product of xylene and formaldehyde produced without the aid of a catalyst or hardening material other than furfuraldehyde is incorporated with paper pulp during the manufacture of the paper-like material, which is subsequently shaped and hardened by heat and pressure to render the resin infusible.

F. R. ENNOS.

**Manufacture of transparent paper.** F. K. WICKEL (B.P. 342,522, 30.1.30).—Highly calendered sulphite paper is impregnated with anhydrous glycerin, sodium lactate, etc., and the surface is coated with albumin or another colloid (coloured, if desired). The coating after being dried is subsequently hardened by means of formaldehyde and treated with ozone to eliminate odour; the product is finally calendered or goffered.

F. R. ENNOS.

**Decorative treatment for paper and like lamp-shades.** J. E. BROWN and E. C. H. COOPER (B.P. 342,269, 28.10.29).—The dried material is coated thickly with a slow-drying varnish and heated for a short time at about 160° to produce wrinkling; the coating is then dried and hardened at 60°.

F. R. ENNOS.

**[Cap-spinning] apparatus for manufacture of artificial threads.** BRIT. CELANESE, LTD., W. J. CROSSE, and W. I. TAYLOR (B.P. 343,965, 26.11.29. Addn. to B.P. 198,023; B., 1923, 765 A).

**Electrically driven spinning centrifuges.** MASCHINENFABR. OERLIKON (B.P. 344,200, 21.2.30. Switz., 15.3.29).

**[Drive for] artificial silk-spinning centrifugal apparatus.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 341,952, 24.10.29. Ger., 29.10.28).

**Drying apparatus for cellulose films and the like.** E. BLEIBLER (B.P. 343,814, 17.4.30. Ger., 2.5.29).

**Twisting artificial silk threads.** ACETA GES.M.B.H. (B.P. 343,791 and Addn. B.P. 343,817, [A] 28.3.30, [B] 22.4.30. Ger., [A] 28.3.29, [B] 22.4.29).

**Waste liquors.**—See I. Petroleum emulsions. **Pulp residues.**—See II. Films. **Compressed fibre.**—See XIII. **Rubber-paper.**—See XIV. **Sugars from cellulose.**—See XVII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Bleaching of [wood] pulp.** H. WENZL (Papier-Fabr., 1931, 29, 49—55).—The reactions involved in hypochlorite bleaching are discussed. Side reactions giving rise to oxidation of fibre are shown to take place during the early part of the process, and the attendant damage is overcome by bleaching in two stages. The "wet end" of a *presse-pâte* machine or concentrator is used to concentrate the pulp in web form, in which state it is given the first bleach, reducing the concentration from 30—33% to 17—20%; the stuff is again concentrated by pressing and the second stage of bleaching carried out. Steam may be used to "activate" the second stage. The process is claimed to be very rapid, and yields products of lower ash, higher  $\alpha$ -cellulose content, and lower copper number than does the bollander method. Chlorine consumption is also lower. The effects of the method on the various constants of a number of pulps are illustrated graphically.

T. T. POTTS.

**Peractivin.** R. FEIBELMANN (Chem.-Ztg., 1931, 55, 191—192).—The name "peractivin" is given to the white powder obtained by mixing toluenesulphondichloroamide with sufficient anhydrous sodium carbonate to remove the unpleasant bleaching powder odour of the amide. Bleaching solutions are prepared by dissolving the powder in 10% sodium carbonate or 3—5% sodium hydroxide solution to yield a 10% solution of the amide; the solutions may be diluted as required immediately before use, but slowly hydrolyse on keeping with the separation of the amide in a crystalline form. The bleaching reaction is due to the liberation of oxygen as follows:  $\text{Me} \cdot \text{C}_6\text{H}_4 \cdot \text{SO}_2 \cdot \text{NCl}_2 + 2\text{NaOH} = \text{Me} \cdot \text{C}_6\text{H}_4 \cdot \text{SO}_2 \cdot \text{NH}_2 + 2\text{NaCl} + 2\text{O}$ ; boiling does not cause this action to take place, hence the solution is very stable in the absence of oxidisable compounds.

A. R. POWELL.

**Behaviour of dye particles within cellulose materials.** A. J. HALL (Cellulose, 1930, 1, 275—280).—A correlation of the penetration and fixation of dyes with the micellar theory of fibre structure is indicated, with particular reference to viscose. Penetration is shown to depend on the disposition of intermicellar spaces. Strips of viscose film cut at right-angles from the sheet absorb a given dye in differing degree owing, it is claimed, to the difference in the orientation of the micelles in the two directions. The fixation of dyes and the behaviour of dyed cellulose under various treatments such as drying, soaping, and boiling in water are held to be accounted for by the properties of the dye particles relative to the nature of the intermicellar spaces.

T. T. POTTS.

**Improvement of furs.** A. SCHAEFER (Chem.-Ztg., 1930, 54, 297—298, 315—317).—The methods used industrially for production of more valuable furs from the cheaper kinds by dyeing, bleaching, and mechanical treatment are described.

W. J. BOYD.

**Alkali-cellulose and cellulose xanthate.** B. RASOW and K. SCHWARZE (Papier-Fabr., 1930, 28, 746—751, 832—836, 869—874; 1931, 29, 34—39, 56—60).—The products of mercerisation of cellulose by means of sodium hydroxide solution vary in composition according

to the conditions of mercerisation and method of analysis. When washing with 92.4 vol.-% alcohol, using phenolphthalein as indicator, the product of mercerisation with 35 vol.-% caustic soda has the composition  $[\text{Na}(\text{C}_6\text{H}_{10}\text{O}_5)_2]\text{OH}$ . If 92% alcohol is used for washing, mercerisation with 39% caustic soda is necessary to produce a product of the same composition, whilst it is found impossible to obtain a similar product if 90% alcohol is used, the higher water content causing decomposition of the alkali-cellulose. Alizarin Yellow R is preferred to phenolphthalein as an indicator. The best method of analysis is found to consist in pressing the cellulose between porcelain plates in a screw-press, the product being reduced to air-dry condition, and the alkali in the filtrate titrated. This method enables a mercerisation product of the above constitution to be obtained by treatment with 21% caustic soda. Use of 38.5% caustic potash produces an alkali-cellulose of similar constitution. It is shown that charcoal, asbestos, and capillary glass-wool when treated with alkali solutions of mercerising concentrations are completely freed from alkali by pressing and washing with absolute alcohol. Celluloses mercerised in aqueous alcoholic solutions of alkali contain a higher proportion of alkali the higher is the water content of the mercerising solution. It is held to be untenable that cellulose xanthate can exist as based on a  $\text{C}_6$  cellulose complex, the evidence being in favour of a formula based on a  $\text{C}_{12}$  complex.

T. T. POTTS.

**Testing the resistance of acetate silk to boiling.** W. FERMAZIN (Chem.-Ztg., 1931, 55, 93—94).—After boiling acetate silk for 1 hr. in water and drying for 2 days at ordinary temperature, the breaking strain and elasticity are diminished, but the extensibility is increased. Treatment with certain dyes (Textile-test H and Picrocarmine K) affords a valuable test for the varying effect of boiling on the threads, depending on the extent of the surface hydrolysis; the amount of acetic acid formed, however, is too small to be determined. Decrease in lustre is also employed quantitatively to determine the action of boiling liquids. The resistance of acetate silk to boiling is improved by the addition of calcium chloride to the liquor.

F. R. ENNOS.

Colours for rubber.—See XIV.

## PATENTS.

**Coloration [without creasing] of fabrics made of or containing cellulose esters.** BRIT. CELANESE, LTD. (B.P. 339,429, 26.10.29. U.S., 26.10.28).—Woven and knitted fabrics containing cellulose ester fibres may be dyed in rope form without the formation of permanent crease marks (cf. B.P. 338,190; B., 1931, 244) provided that they are first partly (3—5%) saponified.

A. J. HALL.

**Production of pattern effects on materials made of or containing cellulose esters or ethers.** BRIT. CELANESE, LTD. (B.P. 314,501, 28.6.29. U.S., 28.6.28).—Pattern effects are produced on cellulose acetate (etc.) materials by forming an oxidation coloration thereon before or after application of an agent which inhibits the formation of the coloration. Such colorations may be produced by oxidation of amines, e.g.,

aniline (B.P. 337,746; B., 1931, 198), *p*-aminodiphenylamine, and 2:4-diaminodiphenylamine (B.P. 258,699; B., 1926, 976), and suitable inhibiting agents are sodium and potassium hydroxides and carbonates, and sodium phenoxide.

A. J. HALL.

**Production of pattern effects on materials made of or containing cellulose esters or ethers.** BRIT. CELANESE, LTD., G. H. ELLIS, and W. B. MILLAR (B.P. 342,333, 26.10.29).—The discharge (in printing) of dyed cellulose acetate silk materials by means of reducing metallic salts such as stannous and titanous chlorides is much facilitated by the presence of a thiocyanate (*e.g.*, potassium thiocyanate), so that complete discharge of very deep shades such as navy-blue, black, and brown may be obtained, and also basic dyes may be used for securing coloured discharges without the possibility of their destruction.

A. J. HALL.

**[Discharge] preparations for treating textile materials.** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 341,909, 18.10.29).—In discharge-printing processes, use is made of discharge pastes containing a finely-divided metallic formaldehyde-sulphoxylate (*e.g.*, the zinc compound); such pastes are efficient and more stable than those in which a dissolved formaldehyde-sulphoxylate compound is present. Methods of working with such dispersions are described.

A. J. HALL.

**Manufacture and application of printing pastes.** SOC. CHEM. IND. IN BASLE (B.P. 318,978, 13.9.29. Switz., 13.9.28).—In printing with vat dyes, urea is added instead of glycerin to the printing paste in order to secure fixation of the dyes; marking-off difficulties are thereby avoided between printing and steaming.

A. J. HALL.

**Production of fabrics having ornamental effects thereon.** BRIT. CELANESE, LTD. (B.P. 342,075, 12.12.29. U.S., 20.12.28).—Fabrics having a cockled or pebble appearance are produced by treating with nitric acid (*d* 1.14—1.30) for 1—5 min. fabrics woven from cellulose acetate and cotton yarns suitably distributed; during the treatment the cellulose acetate yarns shrink more than the cotton yarns.

A. J. HALL.

**Production of artificial [cellulose ester and ether] filaments, threads, films, etc. [having reduced lustre].** BRIT. CELANESE, LTD., R. II. J. RILEY, R. H. PARKINSON, and H. H. SIMS (B.P. 341,897, 17.7.29).—Dull-lustre cellulose acetate silk is produced by spinning the usual acetone solutions of cellulose acetate to which has been added 0.5—4.0% of finely-ground titanium oxide (average particle size 0.0001—0.00035 mm.); zinc oxide and barium sulphate may also be added. The opacity of the resulting silk is increased by adding 2—6% (calc. on the wt. of titanium oxide) of boric acid or a borate (*e.g.*, barium borate) to the titanium oxide.

A. J. HALL.

**[Permanent] filling, loading, or weighting of cotton, linen, and other woven cellulosic fabrics.** R. W. R. MACKENZIE, E. H. ROBINSON, D. LUMSDEN, and M. FORT (B.P. 338,654, 23.9.29).—Fabric is brought into intimate contact with an insoluble mineral loading agent such as china clay, barytes, or talc, while in a plastic condition as produced by treatment with a plasticiser for cellulose such as sulphuric acid, cuprammonium

solution, or a mixture of caustic soda and carbon disulphide; it is then soured or otherwise treated to destroy the plastic condition, whereby the loading agent adheres tenaciously to the fabric and is fast to repeated washing. Fibrous cellulosic material may be incorporated with the mineral loading.

A. J. HALL.

**[Weighting of nitrocellulose] yarn, fabric, plastic, etc.** C. DREYFUS (U.S.P. 1,773,966, 26.8.30. Appl., 30.3.26).—The fibres (etc.) are weighted in the usual manner with insoluble metallic salts, *e.g.*, tin phosphate; they are thereby rendered less inflammable, so that denitration is unnecessary, and show less tendency to "ladder" and slip.

F. R. ENNOS.

**Dyeing and bleaching machines.** F. KENYON (B.P. 344,110, 24.12.29).

**Machine for use in dyeing or otherwise treating hanks or skeins of yarn and thread.** G. P. S. RICCARDI (B.P. 344,263, 10.4.30. It., 30.4.29).

**Cleaning fluid.**—See II. Wetting etc. agents.—See III. Anthranols.—See IV. Impregnating composition.—See XIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Determination of small quantities of volatile organic acids in sulphuric acid solutions.** D. N. CRAIG (Bur. Stand. J. Res., 1931, 6, 169—182).—A procedure suitable for the determination of amounts of volatile organic acids of the order of 0.1% in the sulphuric acid from accumulators and 30—40% sulphuric acid solutions of wood-separator extracts is described. An amount of sodium hydroxide equal to about 70% of the quantity required to convert all the sulphuric acid into normal sulphate is added and the volatile organic acids are distilled off, a portion of the distillate being titrated potentiometrically with alkali. Another portion of the distillate is treated with permanganate to oxidise the formic acid and then distilled, when the acetic acid passes over and is titrated.

R. CUTHILL.

**Microdetermination of iodide in common salt.** H. WERNER (Z. Unters. Lebensm., 1930, 60, 495—500).—The removal of added iodine from common salt by extraction with alcohol is not quantitative, and the author's modification of Winkler's method (*cf.* Leitch and Henderson, A., 1926, 1284) is recommended. The iodine in a filtered, acid solution of the salt is oxidised to iodate by addition of 1 c.c. of chlorine water, the excess of which is removed by boiling. The iodine liberated on addition of 1 g. of potassium iodide is the titrated immediately with 0.002*N*-sodium thiosulphate (1 c.c.  $\equiv$  0.042 mg. I) to a starch indicator. The reaction is upset by the presence of manganous (1.4 mg. Mn) or ferrous (0.01 mg. Fe) sulphates, but addition of oxalic or phosphoric acid, respectively, enables the normal accuracy of the method (determination of 5—27  $\gamma$  I per 50 g. of salt, with a maximum recorded error of  $\pm$  3  $\gamma$ ) to be attained.

J. GRANT.

**Determination of nitrite and sulphite in the presence of one another in salt mixtures and in meat products.** E. SZABÓ (Z. Unters. Lebensm., 1930, 60, 389—395).—(1) The sulphite is precipitated from a solution of the salt in boiled water by means of cold

lead acetate solution in the presence of sodium carbonate, and the nitrite determined colorimetrically after filtration by means of *m*-phenylenediamine. The sulphur dioxide is then removed from the precipitate by acidification and distillation in a stream of carbon dioxide in the usual way. (2) The sulphite is determined in the presence of sodium bicarbonate by addition of excess of 0.1*N*-iodine and back titration with 0.1*N*-sodium thiosulphate. Air is then removed in a stream of carbon dioxide, and the free iodine liberated from the hydriodic acid formed in the first reaction ( $2\text{HNO}_2 + 2\text{HI} = 2\text{H}_2\text{O} + 2\text{NO} + \text{I}_2$ ) titrated after acidification with 1 c.c. of 0.1*N*-sulphuric acid. The maximum error is  $\pm 1$  mg. in both cases for mixtures containing 100 mg. or less of either salt. Meat extracts should be freed from substances which interfere with the reactions.

J. GRANT.

**Volatilisation of potash from wyomingite.** S. L. MADORSKY (Ind. Eng. Chem., 1931, 23, 78–84).—Heating of small samples of the mineral containing 12–13%  $\text{K}_2\text{O}$  together with alumina, silica, and other oxides in an electric furnace at 1510° results in the volatilisation of 25–30% of the potash content in about 40 min.; volatilisation is practically independent of temperature above 1100°, but from 1100° to 1310° it is a little greater than from 1310° to 1510°. The time required for volatilisation is short, but will depend on the physical condition of the wyomingite and on the furnace used. The volatilisation is considerably helped by the presence of calcium carbonate to form a fluid slag with the alumina and silica present, and by a salt promoter such as an alkali chloride, the reactions of these substances with the mineral being exothermic. Calcium chloride is found to be the most effective salt promoting the volatilisation of the potash, as chloride, 95–98% being thus easily removed when a 1:1 mixture of wyomingite and calcium carbonate with 1 potash equivalent of calcium chloride is used. The use of increasing amounts of calcium fluoride as a promoter leads to a sudden increase in the loss of weight by volatilisation, pointing to the formation of silicon fluoride. The commercial recovery of potash from wyomingite by a smelting process is considered feasible. E. A. RYDER.

**Smelting of wyomingite and phosphate rock in the blast furnace.** T. P. HIGNETT and P. H. ROYSTER (Ind. Eng. Chem., 1931, 23, 84–87).—Mixtures of wyomingite and phosphate rock were treated with a hot blast in an experimental blast furnace, potash being volatilised and the  $\text{P}_2\text{O}_5$  of the phosphate reduced. The silicate in the wyomingite was more than enough to flux the phosphate present, so that limestone or burnt lime was added to flux the excess silicate. The reduction of phosphate is not rapid below 1300°. The use of limestone leads to a decrease in this reduction, due to the endothermic decomposition of limestone to give carbon dioxide and then monoxide, whilst the volatilisation of potash is markedly increased by the addition of chloride, the latter being more effective than rock salt, and is not a high-temperature reaction. It is suggested that by adding limestone and using coal as fuel a commercial blast furnace should produce potash and phosphorus pentoxide at a plant cost of \$25 per ton. The experimental results obtained are not the actual volatilised

products recovered, but are calculated from the composition of the furnace slags. E. A. RYDER.

**Production of superphosphate from Khibinsk apatite.** S. I. VOLFKOVICH and L. E. BERLIN (Udobrenie Urozhai, 1930, 2, 11–24).—Samples of apatite (0.15 mm., 35–51%; 0.15–0.3 mm., 44–49%) gave a product with poor physical properties; others (0.3 mm., 21–58%) gave an improved product. Preheating of the sulphuric acid and addition of calcium carbonate to the apatite had no influence on the course of the reaction. Mixtures of sulphuric and hydrochloric acids gave a product with good, and those of sulphuric acid and sodium chloride one with fair, physical properties. Addition of ammonium sulphate improved the product.

CHEMICAL ABSTRACTS.

**Aluminium chloride and the Friedel-Crafts reaction.** P. H. GROGGINS (Ind. Eng. Chem., 1931, 23, 152–160).—A review of the application of aluminium chloride in the American vat-dye and petroleum industries, with particular reference to the chemical engineering problems involved in the industrial applications of the Friedel-Crafts synthesis. An analysis of a typical sample of technical anhydrous aluminium chloride is given, together with data relating to the comparative rates of flow on an inclined plane, hydration, and hydrolysis of this and the chemically pure product. It appears that the granular nature of the technical product renders it more easily handled and less likely to deteriorate in handling and storage, whilst its somewhat lower reactivity is a definite asset in the Friedel-Crafts reaction under industrial conditions.

H. A. PIGGOTT.

**Thiocyanates.**—See II. Sodium sulphate.—See V. Pigments.—See XIII. Beet-sugar ammonia.—See XVII. Hydrogen sulphide toxicity.—See XXIII.

## PATENTS.

**Preparation of concentrated nitric acid.** N. CARO and A. R. FRANK (B.P. 342,068, 6.12.29. Ger., 6.12.28).—In the synthesis of nitric acid from oxygen and nitrogen oxides or gas mixtures containing these oxides, or liquid nitrogen tetroxide, the initial materials are heated at 70–120° under 50–200 atm. pressure, cooling of the highly concentrated nitric acid thus produced being effected also under full pressure. If liquid nitrogen tetroxide is employed, it may be treated with water or aqueous nitric acid before the working pressure is applied. W. J. WRIGHT.

**Production of ammonium sulphate.** PATENT-VERWERTUNGS A.-G. "ALPINA" (SOC. ANON. L'EXPLOIT. DE BREVETS "ALPINA"; or PATENTS EXPLOITATION CO. "ALPINA," LTD.) (B.P. 342,160, 11.2.30. Ger., 11.2.29).—In a two-stage process, ammonia and sulphuric acid are caused to react to such an extent that no crystals will separate from the solution on cooling to 20°. The solution after passing through a cooler is then treated in a saturator with more ammonia, ammonium sulphate being finally precipitated, and separated in a centrifuge. Before the mother-liquor is returned to the first vessel or to the saturator a portion of the sulphuric acid is added to it to heat it slightly and prevent deposition of salt in the return pipes. W. J. WRIGHT.

**Catalytic oxidation of ammonia.** SELDEN Co., Assees. of A. O. JAEGER (B.P. 342,202, 22.3.30. U.S., 22.3.29).—The contact mass contains a base-exchange material of the two-component zeolite type or consisting of a multi-component zeolite, formed by the reaction of a silicate and one or more metal salts or metallates, or a silicate, a metallate, and a salt of which the basic radical can enter the non-exchangeable nucleus of a zeolite. The "permutogenetic body" thus produced is leached with acid before it is incorporated with the catalytic agent. A mixture of two contact masses may also be used, one of these favouring the selective oxidation of the impurities in the ammonia, and the other the oxidation of the ammonia, one of the contact masses being a leached permutogenetic body, as described. W. J. WRIGHT.

**Production of ammonium sulphate.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 342,524, 31.1.30).—The reaction product from ammonia, sulphur dioxide, and water vapour, consisting of ammonium sulphate and sulphite, is subjected to a countercurrent of free oxygen and nitric oxide, and finally treated with ammonia to neutralise traces of acidity. W. J. WRIGHT.

**Manufacture of urea [carbamide].** A. B. LAMB (B.P. 314,443, 27.6.29. U.S., 27.6.28).—Ammonium carbamate (or other compound of ammonia and carbon dioxide) is heated at 150–200° in an autoclave with excess of ammonia at 250 atm. With 100% excess of ammonia the conversion of carbamate into urea is 60–70%. C. HOLLINS.

**Production of alkali phosphates.** METALLGES. A.-G. (B.P. 342,358, 2.11.29. Ger., 2.11.28).—Metal-phosphorus alloys, *e.g.*, ferrophosphorus, are strongly heated with alkali salts in a rotary furnace to initiate reaction, the subsequent heating being regulated to avoid fusion of the mixture. W. J. WRIGHT.

**Extraction of potash and alumina from leucite.** F. JOURDAN (B.P. 342,617, 5.5.30. It., 7.2.30).—The product obtained by the interaction of leucite and limestone is lixiviated with a solution of potassium carbonate to precipitate any unconverted lime and prevent formation of calcium aluminate. Alternatively, the product may be washed with water and the insoluble residue treated with a solution of potassium carbonate to decompose the calcium aluminate. W. J. WRIGHT.

**Manufacture of anhydrous aluminium chloride** (A) free from iron, (B) pure. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. [A] 342,174, 27.2.30, and [B] 342,208, 26.3.30).—(A) The fused chloride is treated with metallic aluminium to reduce iron compounds to iron, this being removed by means of an electromagnet. (B) Crude aluminium chloride is brought into contact with a fused mixture of aluminium chloride and an alkali chloride, which may contain chips of aluminium or iron, or into which reducing gases may be passed, the purified aluminium chloride being sublimed. W. J. WRIGHT.

**Production of iron compounds and chlorine.** S. I. LEVY and G. W. GRAY (B.P. 342,140, 31.1.30).—Ferrous sulphate or phosphate is oxidised with chlorine,

or ferrous chloride with sulphur dioxide and oxygen, at 350–400°, so as to yield a volatile and a non-volatile ferric salt, the latter being heated at 400–600° with sodium chloride to produce chlorine. W. J. WRIGHT.

**Production of zeolites.** W. W. TRIGGS. From GEN. ZEOLITE Co. (B.P. 342,247, 27.7.29).—A solution of an amphoteric metal hydroxide is caused to react with a soluble silicate in such proportions as to produce a gel comprising the whole of the reaction mixture, the latter being maintained at  $p_H$  7–8.4. An acid, such as sulphuric acid, may be added to regulate the  $p_H$  value or to produce additional quantities of soluble salts. [Stat. ref.] W. J. WRIGHT.

**Preparations which yield oxygen on heating, particularly for respiratory purposes.** W. W. TRIGGS. From INHABAD-GES. M.B.H. (B.P. 341,975, 26.10.29).—The oxidisable constituents of the compositions include a compound of a metal and silicon, *e.g.*, ferrosilicon, which does not accelerate the evolution of oxygen, and an alkali to neutralise traces of acid, or the oxidisable substance may be treated with an alkaline solution before admixture with the oxygen carrier. Fibrous asbestos, on which is deposited a catalyst such as cobalt or a metallic oxide, may be added. The composition is surrounded by an insulating material, which is also treated with alkali. W. J. WRIGHT.

**Manufacture of phosphorus or phosphorus pentoxide.** VICTOR CHEM. WORKS, Assees. of R. WEIGEL and H. W. EASTERWOOD (B.P. 342,559, 28.7.30. U.S., 18.3.29).—Mineral phosphates are formed into briquettes with carbonaceous material and insufficient silicious matter to liberate phosphorus or produce a suitable slag when the briquettes are heated. Additional silica is then introduced into the furnace. W. J. WRIGHT.

**Granular substances. Waste liquors.**—See I. **Hydrogen. Ammoniacal liquors.** Sulphur from gas. **Aluminium chloride sludge.**—See II. **Plaster.**—See IX. **Mixed pigments.**—See XIII.

## VIII.—GLASS; CERAMICS.

**Cascade method of melting glass.** T. C. MOORSHEAD and E. A. COAD-PRYOR (J. Soc. Glass Tech., 1930, 14, 249–260 r).—The experience with a re-designed Ferguson cylindrical-shaft furnace is described. It is concluded that successful operation of the process would probably result if a practical method of introducing finely-powdered hot batch to the flame, and a non-porous refractory lining with the minimum number of joints could be devised. A proposed scheme for further experiment is outlined. M. PARKIN.

**Effect of load on fuel consumption of a glass-tank furnace.** E. A. COAD-PRYOR (J. Soc. Glass Tech., 1930, 14, 88–90 r).—Data taken over a period of 6 years from four oil-fired furnaces with melting areas 325–470 ft.<sup>2</sup>, melting 1 ton of glass per 24 hrs. for each 8.5–14 ft.<sup>2</sup> of melting area, showed that with colourless soda-lime-silica glass the fuel consumption falls into the following well-marked periods: (1) very low during first few weeks of furnace life, rising rapidly at the end; (ii) long period with steady

increase of 3% per month; (iii) final period with rapidly falling efficiency. Stage (ii) corresponded with the thinning of the tank blocks to 4–5 in., and stage (iii) with the period when intensive cooling was necessary to prevent collapse of the tank. For a 10% variation in load fuel consumption varied between 1.28 and 2.45% (av. 1.9%). M. PARKIN.

**Efficiency of tank furnaces for glass melting. Proposals for the standardisation of its nomenclature and methods for its measurement.** W. FRIEDMANN (J. Soc. Glass Tech., 1930, 14, 91–102 p.).

The flow of heat from one reversal to the next should be considered with regard to the following sections: (1) gas and (2) air-chamber up to the port, (3) hearth from burner to waste-gas ports, (4) "exhaust" gas chamber, (5) "exhaust" air chamber. Equations for the heat balances in these sections are given and discussed. It is desirable to measure the air used (e.g., by fan and Venturimeter), and gas, air, and waste-gas temperatures should be taken with suction pyrometers. Accurate determination of wall losses from the furnace was not possible. M. PARKIN.

**Effect of factory organisation and size of pots on production from pot furnaces [for glass].** W. W. WARREN (J. Soc. Glass Tech., 1930, 14, 103–106 p.). The ideal production of a pot furnace should approach the continuity of melting conditions given by a tank furnace. This necessitates large furnaces with large pots and an organisation planned to interfere as little as possible with the melting operation. M. PARKIN.

**Manufacture and working life of glasshouse pots.** O. BARYSCH (J. Soc. Glass Tech., 1930, 14, 107–118 p.). Plastic pot-clay mixture (German) consisted of 4–6 pts. of raw clay to 6 pts. of burnt clay, 40–60% of the latter being, in general, < 0.25 mm. in diam., though coarser for sheet-glass casting pots and finer for lead-glass pots. Pots were made from non-plastic materials by slip-casting or pressing, the "grog" in the latter case being as high as 90% of the mixture. In drying, the temperature was kept at 18–22° with 80% humidity for about 1 week, and at the end of the drying and contracting period (3–4 months for pots 80–100 cm. in diam.) the final drying temperature was 30–40°. Cast pots were more difficult to handle than hand-made pots. Whilst highly albuminous pots resisted corrosion better, in general, than highly siliceous ones, they often gave more cordy glass, and thus the siliceous pots gave better glass on the whole. Resistance to corrosion and to thermal shock called for opposite characteristics, so that a compromise was inevitable. Difficulties increased with the size of pot. M. PARKIN.

**Manufacture of glasshouse pots in England.** G. V. EVERS (J. Soc. Glass Tech., 1930, 14, 119–123 p.). Both the Stourbridge Old Mine clay and the Yorkshire clay used for pot-making were less plastic than the German clays employed for similar purposes, so less grog could be used. Latterly the amount of grog had been increased from 25% to as much as 40%, the grading being through  $\frac{1}{8}$ -in. holes set at 45°. Sizes had increased considerably up to 48 in. high  $\times$  42 in. wide  $\times$  54 in. from back to front. Seasoning for 12 months

after becoming air-dry is recommended. Two pots which had lives of 21 and 24 months, respectively, had 72% SiO<sub>2</sub> in the clay, the grog being probably of the same material and present to the extent of 25% of the total. Pot failures were generally on the bottoms or low down in the side. Best pot performance was found where the pots were made in the glass factory. Agreement is necessary as to the chemical composition of raw clay and grog to be used, the proportion and grain size of the grog, and the temperature and time of arching. M. PARKIN.

**Specifications for tank blocks.** W. J. REES (J. Soc. Glass Tech., 1930, 14, 124–130 p.). The provisional specification for refractories issued by the Refractories Committee of the Society of Glass Technology in 1919 was examined in the light of subsequent experience. Aluminous clays are best for tank blocks, with the possible exception of those intended for tanks melting glasses high in B<sub>2</sub>O<sub>3</sub>. Small-size blocks can be better burned than large, and side blocks 9 in. thick were found to have almost the same life as those 12 in. thick. Comparative pore size is of more importance than total porosity. The 2% expansion or contraction allowed in specifying permanent volume change when test-pieces cut from blocks are reheated is considered too liberal. Firing to 1500°, in addition (or instead of) to 1410° appears to be advantageous. The necessity for considering "refractoriness under load" of the blocks, and for including a standard corrosion test in the specification is suggested. M. PARKIN.

**Dependence of the properties of glass on its thermal history.** E. BERGER (J. Soc. Glass Tech., 1930, 14, 280–294 p.). The relationship, found by Tammann and Crone (A., 1930, 530), between change in grain size,  $a$ , with time,  $z$ , when dealing with recrystallisation in metals, is shown to be similar to the equation  $dn/dz = C/z$  deduced by the author from the study of the minimum annealing time of glass at various temperatures,  $C$  being a constant and  $z$  the time taken by the glass to reach a state of equilibrium for the particular temperature considered. The values of these equilibrium points lie on two straight lines intersecting at the transformation point, viz., that at which viscous glass changes to the brittle state. All properties show a sudden break at this point. The shrinkage of Schönborn's glass rods containing boric oxide is found to be the result of aggregation and not wholly of viscous bending due to the mechanical demands of the apparatus. The annealing process does not, as Adams and Williamson (B., 1921, 81 A) assumed, represent a mechanical levelling out of stresses by viscous flow, but is conditioned by the physico-chemical transformation of the state of aggregation. The double refraction, the refraction, and the density were conditioned by the thermal history of the glass. M. PARKIN.

**Theory of the composition of glass.** E. ZSCHIMMER (J. Soc. Glass Tech., 1930, 14, 229–249 p.). The mixing of glass batch to a constant batch formula is productive of errors owing to variation in purity and moisture content of the materials. The correct way is to adjust continually to give the same glass composition. The effect of a 3° change in temperature on the viscosity of



a soda-lime-silica glass is shown to be as great as that of a 0.2% change of  $\text{Na}_2\text{O}$  content. The best method of calculating glass formulæ is on the basis of wt.-% of oxides present. The results of determination of specific properties of series of glasses can be plotted on spatial diagrams and surfaces obtained by joining up related points of parallel series. The method is illustrated with respect to the thermal expansion ( $\alpha$ ) of the soda-borosilicate glasses (cf. Gehlhoff and Thomas, *Z. tech. Physik*, 1925, 6, 547; 1926, 7, 105, 260), the equation to the surface being:  $\alpha \times 10^7 = 0.0588(\text{B}_2\text{O}_3 - 15)^2 + 3.8(\text{Na}_2\text{O} - 5) + 33$ . The construction of spatial models to represent data from four-component glasses is described.

M. PARKIN.

**Mechanical handling of raw and manufactured material in glass works.** T. E. DIMBLEBY (*J. Soc. Glass Tech.*, 1930, 14, 263—275 T).—A description is given (with illustrations) of the application of Morris runways, hoists, and trucks to the transport needs of the glass industry.

M. PARKIN.

**Determination of alumina in refractory clays.** R. BIAZZO (*Annali Chim. Appl.*, 1931, 21, 3—12).—When the only determination required is that of the alumina, the calcined residue of the clay, which contains most of the aluminium and iron as non-colloidal anhydrous oxides, is treated with hot dilute nitric acid, this dissolving the basic aluminium and iron salts without attacking the oxides. After addition of ammonium nitrate and ammonia, the undissolved oxides and the precipitated hydroxides are collected, rapidly washed, and calcined. The ferric oxide in the weighed mixed oxides is then determined separately as usual.

T. H. POPE.

**Slag corrosion of grog blocks by the action of fused peat ashes.** D. S. BELIANKIN and Y. V. KLYTCHAROV (*Keram. i Steklo*, 1930, 6, 383—387).—The action is noticeable at 1100—1200° and is marked at 1300°. The corrosion products are described.

CHEMICAL ABSTRACTS.

## PATENTS.

**Glassware-annealing Lehr.** A. N. CRAMER, ASSR. to OWENS-ILLINOIS GLASS CO. (U.S.P. 1,771,876, 29.7.30. Appl., 11.5.28).—In an annealing chamber in which articles travel on a conveyor, a false roof, extending over a part of the chamber, is carried on rocker arms and thus made adjustable to the height of articles in the chamber. More regular heating and economy of fuel are claimed.

C. A. KING.

**Manufacture of reinforced glass.** BRIT. CELANESE, LTD. (B.P. 341,890—1, 21.10.29. U.S., 20.10.28).—The reinforcing layers comprise (A) a cellulose derivative, e.g., cellulose acetate, which has been subjected to a physical and/or chemical bleaching treatment, (B) a cellulose derivative (if desired, treated as in [A] or otherwise purified) and triacetin. The laminae of glass and plastic material are united by adhesives comprising (A) phenol-aldehyde, phenol-ketone, sulphonamide-aldehyde, etc. condensation products, and (B) wholly or partly polymerised vinyl compounds, e.g., vinyl acetate.

S. S. WOOLF.

**Manufacture of laminated glass.** BRIT. CELANESE,

LTD. (B.P. 342,337 and 342,458, [A] 28.10.29, [B] 11.12.29. U.S., [A] 2.11.28, [B] 11.12.28).—(A) An adhesive layer containing a synthetic urea-phenol-aldehyde resin, and a plastic layer containing a cellulose derivative (e.g., the acetate or nitrate) and a similar resin, together with plasticisers, solvents, etc., are built up successively upon the sheets of glass to be laminated. (B) The glass sheets are carried by rollers etc. through apparatus in which they are successively scoured with soap or chemicals, washed by steam and/or water, dried, covered with a solution containing a synthetic resin as adhesive and, if desired, a cellulose derivative, and treated with hot air to remove the solvent, which is subsequently recovered for re-use. The sheets are then preferably coated with a solution containing a cellulose derivative and, after removal of the solvent, two sheets are united by heat and compression.

L. A. COLES.

**Splinterless glass manufacture [to prevent its discoloration by light].** BRIT. CELANESE, LTD. (B.P. 335,878, 13.3.30. U.S., 13.3.29).—The incorporation of at least 40% by wt. of an organic, e.g., dibutyl or diethyl, tartrate with the cellulose derivative used as adhesive between sheets of glass is claimed. Also up to 20% of a synthetic resin compatible with the cellulose derivative used may be incorporated, together with a dye, e.g., Ponsol Blue, to neutralise the yellow colour of the sheet.

M. PARKIN.

**Manufacture of material resembling reinforced glass.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,075, 20.9.29).—An open-mesh fabric of wire, jute, etc. is coated with an elastic, waterproof film of hardened synthetic polymerisation products of a butadiene, especially those prepared from butadiene with the aid of alkali metals in the presence of solvents. A sulphurising agent, e.g., sulphur chloride, may be used, and condensation products of aromatic hydrocarbons, e.g., xylene, with diolefines, e.g., butadiene, in the presence of condensing agents, e.g., aluminium chloride, may be mixed with such products. The hardening of the film may be accelerated by the incorporation of siccatives, e.g., linoleates or salts of naphthenic acids.

M. PARKIN.

**Manufacture of safety glass.** S. BARRATT (B.P. 335,890, 29.6.29).—Manipulative details are given for removing the knife lines present on the surface of celluloid (etc.) layers to which sheets of glass are made to adhere by means of an interposed film of gelatin. The process is carried out in apparatus of known type, and depends chiefly on adjusting the moisture content of the celluloid.

M. PARKIN.

**Fortifying frosted-glass articles.** R. D. SMITH, ASSR. to CORNING GLASS WORKS (U.S.P. 1,772,965, 12.8.30. Appl., 30.4.29).—The addition of a small quantity of boric acid to the usual fortifying solution with which frosted glass is treated prevents any precipitation of reaction products on to the glass. Such a solution may contain at least 5% of hydrofluoric acid and not more than 25% of ammonium hydrogen fluoride or 10% of boric acid.

C. A. KING.

**[Tunnel] kilns or ovens for baking pottery etc.** R. M. D'ARLEUX and E. VIOLETTE (B.P. 335,154, 15.4.29. Addn. to B.P. 254,023; B., 1926, 918).—The chassis of

a truck in a tunnel kiln is protected from undue heating by means of a second sand joint formed by a ridge of sand along the upper edge of the truck. This sand is swept off gradually by a row of bricks forming part of the kiln wall, the level of these bricks being lower as the trucks advance. The sand falls and feeds the lower seal. Both gas and air are fed to the combustion chamber under pressure and are preheated in a recuperative chamber above the kiln arch, part of the gases being withdrawn from the cooling zone for this purpose. The track inclines upwardly to the zone of dull redness and then downwardly with a steeper inclination in the cooling zone.

C. A. KING.

**Handling and emptying glass-melting pots.** N.V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEN (B.P. 344,308 and 344,322, [A] 30.5.30, [B] 30.6.30. Ger., [A] 3.6.29, [B] 3.7.29. Addns. to B.P. 212,250).

**[Preheating and] tempering of [sheet] glass.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 344,027, 28.11.29. Fr., 5.7.29).

**Patterning of ceramic or enamelled metal goods [by spraying].** "SFINX" SPOJENÉ SMALTOVNY A TOVÁRNÝ NA KOVOVÉ ZBOŽÍ, AKC. SPOL. (B.P. 343,640, 10.12.29. Czechoslov., 15.12.28).

**Cellulose compositions.**—See XIII. Light filter. —See XXI.

## IX.—BUILDING MATERIALS.

**Reinforced concrete. I. Bond resistance. II. Shrinkage stresses. III. Creep or flow under load.** W. H. GLANVILLE (Dept. Sci. Ind. Res., Building Res., Tech. Papers, 1930, Nos. 10—12, [I] 37, [II] 49, [III] 39 pp.).—I. The surface bond between steel and concrete may be due to a chemical or physical process, producing actual adhesion, or to frictional resistance produced by the pressure between them. The influence of pressure is much greater with aluminous than with Portland cement concretes. Overall bond stress depends on the stress in the bar and on the length of embedment; the results of tests on the latter are tabulated. A theory is developed to connect bond stress, steel stress, and length of embedment, and confirms Abrams' results; further verification has been obtained by tests with a multiple-tube extensometer designed for the purpose.

II. An apparatus for determining the distribution of shrinkage stress along a reinforcing bar is described, and approximate methods for allowing for the creep occurring during shrinkage in making calculations for shrinkage stresses are outlined. Shrinkages in aluminous, rapid-hardening, and normal Portland cement concretes are compared.

III. The creep of plain concrete over long periods is proportional to the applied stress; values are given of the creep for aluminous, rapid-hardening, and normal Portland cement concretes.

W. J. WRIGHT.

**Quantity of lime in a raw mixture for silicate bricks.** A. TAL (Building Mat. [Russia], 1930, No. 2, 63—66).—The quantity of lime depends on (a) its porosity, fineness, and firing temperature; (b) the composition of the sand, the shape of the grains, and the state of their surface; (c) the homogeneity of the

raw mixture; (d) the method of slaking the lime. The solidity due to uncombined lime increases in proportion to its uniform distribution in the brick.

CHEMICAL ABSTRACTS.

**Determination of magnesium in Portland cement and similar materials by the use of 8-hydroxyquinoline.** J. C. REDMOND and H. A. BRIGHT (Bur. Stand. J. Res., 1931, 6, 113—120).—A procedure for the accurate determination of magnesium in cement by precipitating with 8-hydroxyquinoline and titrating the acid solution of the precipitate with bromide-bromate solution (cf. Berg, A., 1927, 639) is described.

R. CUTHILL.

**Determination of wool in boards [roofing felts] etc.** R. KORN and B. SCHULZE (Papier-Fabr., 1931, 29, 68—69).—In the determination of wool in roofing felts etc. by means of hydrolysis of the cellulose with 80% sulphuric acid, lignin from mechanical wood resists the hydrolysis and affects the results. The insoluble portion of mechanical wood is of the order of 12% when the usual method of analysis is employed. By continual shaking during the hydrolysis the insoluble residue is reduced to about 6%. The equivalent of 10 g. of ashless bone-dry material is disintegrated and washed with 96% alcohol. Hydrolysis is effected by shaking for 3 hrs. with 300 c.c. of 80% sulphuric acid in a 2-litre flask. After dilution to 1 litre, filtering, and washing, the residue is dried, weighed, ignited, and reweighed. For each 10% of mechanical wood present, 0.6% is deducted from the ashless residue to give the wool content of the material.

T. T. PORTS.

**Asphalt shingles.** J. M. WEISS (Ind. Eng. Chem., 1931, 23, 168—170).—The results of the examination of 16 brands of asphalt roofing shingles are presented together with comments on the different compositions which are encountered. Certain new specifications are suggested.

H. S. GARLICK.

**Tests on Dinas and grog blocks.** O. LEKHER (Building Mat. [Russia], 1930, 4, 41—46).

**Painting on wood.**—See XIII.

## PATENTS.

**Rotary kilns [for cement, lime, etc.].** J. S. FASTING (B.P. 342,417, 19.11.29).—In order to reduce the loss of dust from the cooler end of such kilns, the material to be burned is fed into and passes through a number of tubes disposed around and connected with the central kiln tube. Combustion gases from the kiln are conducted through different openings into the subsidiary tubes in countercurrent to the material. It is preferred that the openings discharging the material into the central tube should be designed tangentially.

C. A. KING.

**Treatment of raw [cement] slurry before burning in a rotary kiln.** N. NELSON (B.P. 341,875, 21.10.29).—Wet cement slurry is drained, granulated, and heated by the passage of the hot gases from a cement kiln through the mass. This preheating may be effected in a shaft furnace with a downward draught to protect the bottom grate on which the material rests, or in other forms of furnace in which the gases are directed firstly inwardly into the bed and then upwardly through the granular material.

C. A. KING.

**Manufacture of cement.** G. A. WALTER (U.S.P. 1,770,958, 22.7.30. Appl., 30.6.25).—In order to prevent the adherence of cement clinker, when in plastic condition, to the wall of a rotary kiln, sodium chloride (2% on the weight of cement materials) is added to the powdered fuel and injected into the kiln. C. A. KING.

**Manufacture of fused cement.** S. KOHUT (B.P. 342,414, 19.11.29. Ger., 22.11.28).—A reverberatory melting furnace is in connexion with a preheating furnace only so far as the passage of the hot gases is concerned. Calcined material is withdrawn, *e.g.*, from the bottom of the furnace, into an externally operated scoop which then deposits the charge in different positions of the melting-furnace hearth as desired. To prevent overheating in the calcining furnace the waste gases are partly cooled by the insertion of a recuperator or by the admission of steam or air. C. A. KING.

**Oxychloride cement compositions.** E. WOOD (B.P. 335,428, 5.11.29).—Compositions consisting of a mixture of sand, calcined magnesite, and magnesium chloride solution are claimed. Colouring substances may be added as desired, and the sand is preferably washed in dilute (5%) caustic soda solution. The magnesium chloride solution is divided into two portions, one of which is heated to the b.p., allowed to cool, and added to the other portion, together with water to bring the solution to *d* 1.12—1.20. In an example, sand (75 pts.) and calcined magnesite (25 pts.) are mixed with sufficient of a 1:3 mixture of "treated" and untreated magnesium chloride solution to form a paste. C. A. KING.

**Acceleration and improvement of hydraulic cement.** F. W. HUBER (U.S.P. 1,773,001, 12.8.20. Appl., 22.11.28).—Portland cement is mixed with about an equal volume of a 50% solution of calcium chloride and the mixture is stirred until it gels. C. A. KING.

**Cellular gypsum product.** G. A. NEW, Assr. to AMER. GYPSUM Co. (U.S.P. 1,770,797, 15.7.30. Appl., 27.12.27).—A dry mixture of calcined gypsum and 0.5% of blood flour (dried residue of animal blood) is made plastic by adding water containing less than 1% of hydrogen peroxide. The blood flour acts as a catalyst for the decomposition of hydrogen peroxide and accelerates setting of the plaster with the enclosed gas bubbles. C. A. KING.

**Production of plaster from by-product calcium sulphate.** R. S. EDWARDS, Assr. to RUMFORD CHEM. WORKS (U.S.P. 1,770,367, 8.7.30. Appl., 19.1.29).—Calcium sulphate left as a sludge from an acid process, *e.g.*, manufacture of phosphoric acid, is washed with dilute sodium chloride, *e.g.*, sea-water, which removes the greater part of the acid in the form of the sodium salt, *e.g.*, sodium phosphate. Final neutralisation with lime renders the sludge in suitable condition for calcining into plaster. C. A. KING.

**Making of roads.** F. W. HUBER (U.S.P. 1,773,000, 12.8.30. Appl., 24.8.28).—Aggregate is mixed with hydraulic cement, water containing an electrolyte as the disperse phase, *e.g.*, calcium chloride, and a small quantity of petroleum oil emulsion, which waterproofs the road bed. C. A. KING.

**Manufacture of bituminous binders for road stone etc.** SOUTH METROPOLITAN GAS Co., and H. PICKARD (B.P. 341,901, 23.7.29).—Pulverised coal (10—18%) is mixed with tar at 250—300° and pitch is added up to 66% of the weight of the dispersed mixture. The strength of the mixture is then increased by adding finely-ground mineral matter (20—60 wt.-% of the final product). C. A. KING.

**Wood preservation.** V. R. ABRAMS, Assr. to A. N. SHELANDER (U.S.P. 1,769,983, 8.7.30. Appl., 19.11.27).—Wood is impregnated in sequence with a solution of a copper or other toxic metal salt and a resin soap solution to promote the formation of an insoluble toxic metal resinate. C. A. KING.

**Manufacture of hard-lumber substitute.** O. R. SWEENEY, Assr. to IOWA STATE COLLEGE OF AGRICULTURE & MECHANIC ARTS (U.S.P. 1,772,502, 12.8.30. Appl., 2.10.28).—Pulped cornstalks (or other agricultural waste materials) are sized and formed into a mat, which is pressed between heated platens to liberate pentosic compounds and cause them to bind the cellulosic parts and fibres of the material; channels are provided on one side of the pulp, while it is being pressed, in order to facilitate the escape of steam. F. R. ENNOS.

**Treatment of bituminous street coverings.** CONTINENTALE TEERSTRASSEN-BAU-GES.M.B.H., and H. KÖHLER (B.P. 343,692, 15.1.30. Ger., 15.1.29).

**Dryers.**—See I. Hydrocarbons from coal. Bitumen dispersions.—See II. Grain of boards.—See XXI.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Reduction of iron oxide ores in the molten state by means of hydrogen and other gases.** S. L. MADORSKY (Ind. Eng. Chem., 1931, 23, 99—103).—Satisfactory experimental furnace conditions are obtained by heating the ore by means of an electric arc over a chromite hearth, the reducing gas being fed to a point below the surface of the molten ore through a graphite tube. The reduction caused by the arc and graphite tube is measured and allowed for. The metal produced is much purer than that from the hearth process, no coke being introduced, the amount of slag being greater with attendant smaller phosphorus concentration, whilst titanium is not reduced by the gases. Other impurities are also low. In the experiments described, hydrogen, hydrogen-carbon monoxide, and carburised water-gas were the gases used, these being dry in all cases. With these gases carbon is introduced into the iron, depending on the solubility of carbon monoxide in the molten metal, but this carbon is less than that obtained with the open-hearth process. Excluding graphite reduction, the hydrogen used is 1.19 cub. m. per kg. of iron (*i.e.*, 1.45 times the theoretical amount), but this quantity should be reduced in a commercial unit. With hydrogen available at 1 cent per cub. m. the cost of the process is estimated at \$24.80 per metric ton, this figure lying between those for pig iron made by the blast furnace and for steel by the open-hearth or Bessemer process. The quality of the product is an important factor in considering the cost of the process. E. A. RYDER.

**Effect of various annealing temperatures on cold-worked low-carbon steel.** H. E. PUBLOW (Met. and Alloys, 1931, 2, 18—20).—The hardness of the bearing surface of mild steel (0.2% C) brake drums is usually about 30 units (Rockwell B scale) greater than that of the metal before pressing into shape, although microscopical examination fails to reveal deformation of the ferrite or pearlite grains. On annealing for 30 min. at various temperatures, the hardness decreases sharply above 550°, reaches a minimum at 700°, and then increases again with rise of annealing temperatures up to 870°; higher annealing temperatures have little effect on the hardness, but increase the grain size remarkably. Maximum ductility is obtained by annealing at 700° and minimum ductility by annealing at 1035°.

A. R. POWELL.

**Corrosion of copper and high-copper alloys by salt solutions.** A. RAUCH and H. KOLB (Korrosion u. Metallschutz, 1930, 6, 127—132, 151—156, 174—178, 193—200; Chem. Zentr., 1930, ii, 3458).—Corrosion is maximal in 15—25% potassium chloride solutions; it is increased by rise of temperature and velocity of flow. Sulphate solutions are less corrosive than chloride; equivalent chloride solutions behave similarly, whilst air-free solutions are practically inactive. The solubility of oxygen in potassium chloride solutions diminishes with increase of concentration and rise of temperature. Tin bronze and aluminium bronze (very resistant) were examined. Cold-worked, "hard" specimens are more readily attacked than "soft" specimens.

A. A. ELDRIDGE.

**Macro-etching of tin-base bearing metals.** E. B. DRAKE (Met. and Alloys, 1931, 2, 20—24).—Immersion of bearing metals with a high tin content in ammonium polysulphide solution for 20—30 min. develops the structure of all the usual constituents and indicates the size and distribution of the micro-constituents. In centrifugally cast bearings of an alloy of 85% Sn, 7.5% Cu, and 7.5% Sb the structure consists of SnSb cubes and CuSn needles in a ground-mass of tin-rich eutectic, whereas die-cast bearings of this alloy have a structure consisting of CuSn needles in a ground-mass of eutectic and  $\delta$ -solid solution.

A. R. POWELL.

**Mutual action between metallic utensils and dietetic material. I. Action of potable liquids on metals; corrosion.** B. BLEYER and J. SCHWAIBOLD (Biochem. Z., 1931, 230, 136—145).—The action of water, tea, and coffee on the more common metals was followed by determinations of the amount of metal dissolved and by the change in reaction of the liquid under investigation.

F. O. HOWITT.

Paints.—See XIII.

PATENTS.

**Regenerative smelting furnace having gas and air regenerators.** H. MOLL (B.P. 342,452, 5.12.29).—In a furnace with large air regenerators below the furnace which connect through a horizontal passage with a smaller regenerator above the hearth level, a removable slag collector is placed under the upper regenerator. The outlet to the gas regenerators leads downwardly to another slag receptacle before the gases enter the regenerator system.

C. A. KING.

**Regenerators of Siemens-Martin furnaces.** F. FIORELLI (B.P. 341,721, 22.1.30. It., 25.1.29).—Each regenerator is subdivided, the whole being used during the period of intense heat and part cut off later when the flow of gases becomes less.

B. M. VENABLES.

**Refining of iron or steel.** F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 341,915, 21.10.29. Ger., 18.6.29. Addn. to B.P. 317,373; B., 1931, 119).—In refining molten iron by means of water, the latter may be introduced into the bath through ordinary iron pipes coated previously with a liquid slag. The end of the pipe may be splayed to form a finer jet of liquid.

C. A. KING.

**Manufacture of [steel] articles hardened in their marginal layers by nitrogenisation.** R. SERGESON (B.P. 341,113, 15.10.29).—The articles are made from steel containing less than 0.5% (0.2—0.4%) Al, and are case-hardened in ammonia at 650° for 48 hrs. [Stat. ref.]

A. R. POWELL.

**Grinding of slag or similar iron-containing material.** N. AHLMANN (B.P. 341,951, 24.10.29).—Slag is crushed in a preliminary grinding machine, screened, and the finer-ground portion passed to a tube mill. Nodules of iron are separated by magnetic means from the coarser portion, and the latter is returned to the preliminary grinder. The screen may be attached to the tube mill and so made to rotate.

C. A. KING.

**Melting-baths. Disintegrators.**—See I. **Rubber-metal bond.**—See XIV.

## XI.—ELECTROTECHNICS.

**Sulphuric acid [in accumulators].**—See VII. **Iron ores.**—See X. **White lead. Luminescence analysis.**—See XIII.

PATENTS.

**Hydrocarbons from coal.**—See II. **Resistances. Synthetic resin.**—See XIII.

## XII.—FATS; OILS; WAXES.

**Oleic acid rancidity of fats. II. Measurement of rancidity.** K. TÄUFEL and J. MÜLLER (Z. Unters. Lebensm., 1930, 60, 473—484; cf. A., 1930, 891).—A critical discussion of the qualitative tests for rancidity and of likely quantitative modifications. The Kreis test gives reproducible results and, by construction of a colour scale, has been used to follow the evolution of rancidity of ethyl oleate. The results are affected by the presence of hydrogen peroxide or aldehydes of the fatty acids. The rate of production of rancidity is much greater in the light than in the dark and, in the former case only, attains a constant maximum (after 14 days). No relation appears to exist with the rate of increase of acid value, which is considerably slower and almost linear. The daily increase in weight of 2 g. of oil in air is preferable to the volumetric oxygen-absorption method as an indication of the increase in rancidity and shows a somewhat slower rate of increase than the Kreis test, but allows a wider range of time to be studied. No simple relation between the two methods could be traced, though both tend approximately to the same value after about 20 days, and it is probable that they represent changes differing in nature.

J. GRANT.

**Expansion on melting of fats.** W. NORMANN (Chem. Umschau, 1931, 38, 17—22).—A modification of the dilatometer used in the margarine industry was employed, the change of volume being measured by the rise of a column of water in a capillary sealed into the side of the pyknometer bulb near the bottom. Temperature-expansion curves are plotted. The expansions of the liquid arachis, linseed, and castor oils, and of oleic acid (liquid), are practically alike and of the same order as those of liquefied fats. A mixture of dilauromyristin and dimyristolaurin (approx. 1 : 1, from palm-kernel oil) shows, as anticipated, gradual expansion of the solid fat, followed by a sudden large dilatation (8500 mm.<sup>3</sup>/100 g.) at the m.p., and then the straight-line moderate expansion of the liquid. A hardened linseed oil (iodine value 1) behaved abnormally: from 20° the fat expanded, but at 39° began to contract, reaching a minimum volume at 47°, actually smaller than the original volume at 20°. (This contraction is very slow; a few hours are necessary for a rise of 3—4°.) The fat then expanded normally, and from 64° to 69° showed the sudden volume increase (16,900 mm.<sup>3</sup>/100 g.) characteristic of melting. The behaviour of solid oleic acid seemed to depend on the pretreatment. In some cases, working from 0° to —20°, expansion occurred smoothly (*i.e.*, the values were on a straight-line graph) up to 60° or 80°, with only a very slight irregularity at 11—13°, but no sign of m.p. expansion. With other specimens of the acid, frozen under different conditions, a graph was obtained rising sharply from 0° or 20° at a slope comparable with that observed normally only during melting. Elaidic and stearic acids behaved normally, showing on melting sharp expansions of 15,300 and 16,300 mm.<sup>3</sup>/100 g., respectively. Curves for some commercial fats are given. E. LEWKOWITSCH.

**Determination of sp. gr. of solid fats and other solid materials.** W. NORMANN (Chem. Umschau, 1931, 38, 22—23).—The author's modified dilatometer (*cf.* preceding abstract) is suitable for the determination of the sp. gr. of oils and solid fats etc., the manipulation for which is detailed. It is essential in determinations of sp. gr. to free the fat from dissolved air by warming *in vacuo*. E. LEWKOWITSCH.

**Saponification values of edible fats as a function of the content of palmitic acid and other fatty acids.** J. GROSSFELD (Z. Unters. Lebensm., 1930, 60, 64—93).—A general formula  $P = 4.837S - 0.206A + 9.142U - 914.2 + q_1F_1 + q_2F_2 + \dots$  has been derived for the calculation of the palmitic acid content  $P$  of a fat or oil in terms of the saponification value  $S$ , the acid value  $A$ , the content of unsaponifiables  $U$ , and the fatty acids  $F_1, F_2, \dots$  present in addition to stearic and palmitic acids. The values of the factors  $q_1, q_2, \dots$  for the more important fatty acids are given. A simplified expression  $P = 4.837S - 914.2$  is used in the case of a pure glyceride or a pure cacao fat, a saponification value for the latter being evaluated as  $194.3 \pm 0.6$ , unsaponifiables as 0.324% (aver. 8 samples), and stearic acid as 33.3% (aver. 7 samples). The apparent palmitic acid content of cacao fat serves as a means of detecting adulteration by extracted butter, hard ground-nut fat, or coconut or palm-kernel oils. A further general

expression for other edible fats is given with values of the constant for lard, beef and pressed tallow, mutton fat, oleomargarine, butyrin, and almond, apricot-kernel, olive, coconut, palm-kernel, peanut, linseed, and rape oils. Other formulæ are presented for calculating the erucic acid content of rape oil and the lauric acid content of coconut and palm-kernel oils. H. J. DOWDEN.

**Twitchell reagent. IV. Effect of addition of electrolytes on the colloidal property of the reagent. V. Change of colloidal properties of Divulson D by addition of various substances. VI. Fat-splitting power of Divulson D and the darkening of the fatty acid.** K. NISHIZAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 349—352 B, 412—414 B, 414—417 B; *cf.* B., 1930, 870).—IV. [With R. ASADA and B. SAKUMA.] The effect of added electrolytes on the viscosity and surface tensions (Traube stalagmometer) of 1% aqueous solutions of "Idrapid," "Pfeilring," and "Kontakt" reagents was studied. The strong acids (and their salts) effected a marked increase in the relative drop number and viscosity of the solutions: the influence of weak acids was much less, the results generally being similar to those obtained from the study of emulsifying power (*cf.* B., 1929, 946). Correspondingly, more complicated curves were obtained from those reagents which are easily salted out.

V. [With B. SAKUMA.] The effects of concentration and the addition of acids and salts to 1% solutions of Divulson D are similar in type to those obtained with other Twitchell reagents (*cf.* B., 1929, 946); Divulson D markedly resembles Idrapid, except that the latter is the more easily salted out by strong electrolytes.

VI. [With K. FUJIMOTO.] The effect of adding acids and salts on the fat-splitting power was similar to that observed previously with "Idrapid" and similar reagents (*cf.* B., 1930, 870), and hence the conclusions drawn then are applicable in the case of Divulson D. The addition of glycerol or free fatty acids decreased the hydrolytic power of the reagent, which is also much influenced by variation in temperature or speed of stirring. In fat-splitting power Divulson D is superior to Pfeilring or Kontakt, and also to Idrapid in the presence of small amounts of sulphuric acid, but is inferior to the latter as the concentration of acid is increased. With regard to the colour of the fatty acids, the new reagent is superior to Pfeilring and Kontakt, but inferior to Idrapid. E. LEWKOWITSCH.

**Determination of the Reichert-Meissl value of small quantities of mixed fats.** A. MILLIG (Z. Unters. Lebensm., 1930, 60, 318—320).—To a weighed sample of the fat (2 g., 1 g., or 0.5 g.), vegetable oil is added to make the total quantity of fat up to 5 g. The Reichert-Meissl value of the mixture is determined in the usual way. If the resulting value of the mixture be  $R_1$ , of the vegetable oil be  $R_2$ , and of the sample be  $R_3$ , and if  $w$  be the weight of sample used, then  $R_3$  may be found from the equation:  $R_1 = R_2 \times [(5 - w)/5] + R_3(w/5)$ . The values obtained agree closely with those obtained when a 5-g. sample is used.

W. J. BOYD.

**o-Dichlorobenzene an ideal fat solvent.** J. EHRLICH (Oil & Fat Ind., 1931, 8, 19—20).—Commercial

*o*-dichlorobenzene (about 75% *o*- and 25% *p*-isomerides), b.p. 160–200°, readily extracts oil from moist materials, is unattacked by steam, and hence does not corrode steel plant. The solvent is readily removed from the oil solution by steam-distillation under ordinary pressure (freshly purchased crude solvent may need a preliminary distillation), and separates easily from the water on condensation. It is almost non-inflammable and the vapour of the *p*-isomeride at least is non-poisonous.

E. LEWKOWITSCH.

**Course of the hardening of unsaturated fatty acids with a triple linking.** A. STEGER and H. VAN LAARHOVEN (Chem. Umschau, 1931, 38, 23–24).—The thiocyanogen value of stearolic acid is the resultant of an equilibrium reaction, and varies with the temperature and time of reaction, and especially the concentration of the reagent; values as high as 69 (at 18°) have been found. Hydrogenation of ethyl stearolate (with a nickel-kieselguhr catalyst at 180°) proceeds to the stage of the oleic-elaidic ester (checked by determinations of  $n_D$ , iodine and thiocyanogen values); addition of fresh catalyst, as in the case of elæostearic acid (van Loon) and linseed oil fatty acids (van der Veen), is necessary before saturation can be completed. Ozonolysis indicates that no shifting of the triple linking occurs.

E. LEWKOWITSCH.

**Relations between the properties of soaps and the degree of unsaturation of fatty acids.** (I) Surface tension and detergent power, (II) drop number and relative viscosity, (III) lathering power, of the soap solutions of  $C_{18}$  acids. M. HIROSE (J. Soc. Chem. Ind., Japan, 1930, 33, 337–338 B, 338–340 B, 340–341 B).—I. [With T. SHIMOMURA.] The surface tension, at 10–80°, of solutions of the sodium soaps (0.025–2.0%) of oleic, linoleic, and linolenic acids increases with increasing unsaturation of the acids; the value for sodium stearate solutions is lower at high temperatures and higher at low temperatures than those of the unsaturated soaps. The surface tension (except in the case of the stearate at high temperatures) passes through a minimum at a certain concentration. The detergent power of the unsaturated soaps falls with rising unsaturation; the stearate has still lower values at low temperatures, but the greatest detergency at high ones. With solutions more dilute than 1%, the detergent power increases with the concentration (except in the case of stearate solution, which solidifies at room temperature), whilst minimal surface tensions always occur at low concentrations; for solutions of various soaps the two properties are not mutually related except at the same temperature and concentration, and the surface tension cannot be utilised to determine optimum concentration.

II. [With T. SHIMOMURA.] The drop numbers of solutions of the soaps (0.025–2.0%), measured at 10°, 40°, and 80° in a Hillyer stalagmometer, are given. In general, the drop number increases with lowered temperature and increasing concentration (stearate excepted). The linoleate has the greatest drop number at 0.5%, and the oleate at 0.025% concentration. No definite relation between drop number and detergent power could be found. The relative viscosities of stearate

solutions are higher than those of the unsaturated soaps, which do not differ much among themselves.

III. [With S. KITAJIMA.] At low concentrations sodium stearate scarcely lathers. The lathering volume (vol. of lather/vol. of sample) rises with increasing unsaturation, concentration, and temperature; the stability of the lather, however, tends to the reverse proportion. The lathering number (vol. of soap solution passed into lather/vol. of sample) falls with rising temperature and increased unsaturation: with the linoleate it falls steadily with dilution, and with the oleate passes through a maximum at 1%. The sp. vol. of lather (lathering vol./lathering no.) shortly after shaking increases with unsaturation, but after 10 min. the reverse relation holds.

E. LEWKOWITSCH.

**Determination of total fatty acids [in soaps].** A. K. CHURCH (Oil & Fat Ind., 1931, 8, 23–25).—The use of separatory funnels for the extractions is avoided by use of a modified Stokes' flask. The volumes of the solutions are adjusted so that the junction of the ether and aqueous layers occurs just above the constriction between the two bulbs of the flask, and the ether extracts are removed by means of a siphon; three extractions are sufficient. Suitable modifications of the standard determinations of total fatty matter, unsaponifiable matter (A.C.S. method), and rosin (A.C.S.—Wolff's method) are detailed, which give results in good agreement with the standard procedures. For coconut oil soaps it is recommended that the fatty matter be weighed as soap.

E. LEWKOWITSCH.

**Solubilities of soaps in water.** J. MIKUMO (J. Soc. Chem. Ind., Japan, 1930, 33, 367–368 B).—Various binary mixtures were studied in order to determine the influence of the very soluble soaps of (I) the lower saturated fatty acids and of (II) the unsaturated oleic and ricinoleic acids on the solubility of the soaps (III) of the higher saturated fatty acids. The temperature, m.p. of the fatty acids of soaps II and III, and the concentration of their solutions exerts great influence on the solubilities of soaps I. Soaps II diminish the solubility of the less soluble soaps I at low temperatures (by salting them out), but at 50° they increase the solubility of soaps I by peptisation. At moderate concentrations soaps III increase the solubility of soaps I.

E. LEWKOWITSCH.

**Analysis of machine grease.** C. BUNGE (Chem.-Ztg., 1930, 54, 299).—The sample (100 g.) is melted in 200 c.c. of water and 40 c.c. of hydrochloric acid are added. The mixture is boiled for 5 min. with stirring and then kept warm until the fat separates. While still warm it is filtered through a weighed Neubauer crucible and the residue washed first with a fat solvent, then with water, and the crucible is dried, weighed, ignited at a red heat, cooled, washed again with hot hydrochloric acid and with water, and again dried and weighed. From the weight before ignition the total amount of impurity retained mechanically is found, and from the weight after ignition the sand content is obtained. The sand grains may be counted microscopically and photographed if necessary.

W. J. BOYD.

**Oleaginous seeds from the forests of Angola.**



C. DE MELLO GERALDES [with A. D'ALMEIDA and C. DUARTE] (Bull. Mat. Grasses, 1930 14, 332—342).—The ether-extracted oils from the seeds of the following species have been examined. *Allanblackia floribunda*, Oliv., "Lalanjo": dark reddish-brown ovoid three-sided seeds, av. wt. 2.39 g., yielding a whitish, odourless fat, m.p. 40.7°, f.p. 39.5—40.5°, titer 58—60°. *A. saculeuxii*, Nua., "M'bunze": bright red-brown seeds irregularly three-sided, av. wt. 8.6 g., yielding a yellowish-white fat, m.p. 41.8—42.8°, f.p. 41—42°, titer 59—61° (cf. Jumelle "Les Huiles végétales," 1921, p. 227). *Irvingia robur* Mildbr., "Cuma": the fibrous drupe (av. wt. 84 g.) contains only 6.75% of kernel surrounded by a very stony endocarp, and is valueless as a commercial source of oil. *Balanites mayumbensis*, Exell., "Buza pundi": the fleshy drupe yields ovoid stones, av. wt. 35.9 g., containing 48.16% of dark-fleshed kernel. The brownish acid oil obtained had an unpleasant odour and taste. Comparative figures for other *Balanites* species are quoted. (The name of a species examined previously [J. d'Agric. tropicale, 1912, No. 134, 233] and erroneously recorded as *Trachylobium moçambicensis* is corrected to *B. maughamii*.) *Strombosia scheffleri*, Engl., M'senha: the ovoid stones (wt. 4—12 g.) from the drupes (av. wt. 10.3 g.) yield 62.5% of kernel, from which is obtained a yellow-brown clear oil of unpleasant odour and taste. *Mimusops ebolowensis*, Engl. and Krause, "Muabi": the piriform seeds (av. wt. 19 g.) contain 15.17% of kernel containing 4.75% of fat and 14.32% of moisture. The seeds (s) or kernels (k) of these six species contained, respectively (%): moisture 3.44 (s), 2.51 (s), 7.2 (k), 9.15 (k), 13.74 (k), —; oil (on dry basis) 59.96 (s), 62.64 (s), 64.85 (k), 39.37 (k), 18.06 (k), —. The oils had, respectively:  $d_{25}^{20}$  0.89484, 0.91869, 0.9194, —, 0.91715, 0.9353;  $n_D^{20}$  1.4529/50°, 1.4551/50°, —, 1.4602/50°, 1.4716—1.4742/25°, —; saponif. value 184—195, 207.85, —, 204.61, 216.2, —; iodine value 33—36, 29.5, —, 101, 83.6, —; acid value 3.6—7.5, 35.3, —, 71.12, 39.1—98.6, —. The kernels of *Strombosia gossweileri*, S. Moore, and of *Strombosiaopsis congolensis*, De Wild and Durand, contained, respectively, moisture 11.7, 11.7%, oil 3.04, 2.2%. The kernels of an unknown species of N. O. *Sapotaceae* contained 70% of fat.

E. LEWKOWITSCH.

**Solvent extraction [of vegetable oils].** C. F. EDDY (Oil & Fat Ind., 1931, 8, 11—13).—The economics of solvent extraction are discussed in the light of factory experience; for the greatest efficiency plant should be designed and used for one class of raw material only.

E. LEWKOWITSCH.

**Yield differences in oil-mill practice.** E. I. BETTER (Chem. Umschau, 1931, 38, 33—34).—In practice, the actual yield of oil was always found to be a few % below the theoretical yield calculated from analyses of the seed and the cake formed. It is shown that a possible explanation might be advanced on the assumption that in the analysis certain substances (e.g., of lecithin type) are extractable from fresh seed, which become insoluble after steaming and are unavailable in the cake; if so, the changes suffered by these little-understood compounds must be of a complicated nature.

E. LEWKOWITSCH.

**Influence of anti-oxidants on the rate of oxidation of linseed oil. I. Quinol.** A. M. WAGNER and J. C. BRIER (Ind. Eng. Chem., 1931, 23, 40—49).—The effect of the addition of quinol before and during the oxidation of linseed oil has been studied by blowing samples with conditioned air at 30° and 100°, and following the oxidation by iodine value and refractive index determinations. Blowing of pure linseed oil at 30° showed a prolonged induction period (some 300—400 hrs. in the apparatus used) during which oxidation was too slow to be detected, and after which the rate of oxidation increased gradually to a maximum and then decreased as gelation was approached. At 100° the induction period, if any, was too short to be measured and oxidation proceeded much faster than at 30°. The addition of quinol (in amounts greater than 1:10,000, most conveniently between 1:1000 and 1:100) to the oil prolonged the induction period, the effect being proportional to the concentration of the anti-oxidant, and the greater at the lower temperature. During the induction period the oxidation was very slow, being undetectable at 30°, but measurable at 100°. At the end of the induction period oxidation proceeded at a rate identical with that of linseed oil alone at the same temperature and state of oxidation. At 100° secondary additions of quinol after the termination of the induction period had no retarding effect on the oxidation; at 30°, however, quinol was effective as an anti-oxidant, regardless of the state of oxidation of the system, but its influence was inversely proportional to the rate of oxidation. Secondary additions of quinol before the end of the induction period (i.e., before the previous addition had ceased to be effective) did not further delay the end of the inductive period. The addition of partly oxidised linseed oil reduced the induction period. Hence it is concluded that the effectiveness of quinol as an anti-oxidant is determined by the velocity of the oxidation, and that after a certain critical rate is exceeded quinol is unable to inhibit oxidation.

E. LEWKOWITSCH.

**Drying oils. XIV. Rate of oxidation of linseed oil at 160°.** J. S. LONG and H. D. CHATAWAY (Ind. Eng. Chem., 1931, 23, 53—57; cf. B., 1930, 825).—Linseed oil, triolein, and trilinolenin (cf. B., 1930, 154) were oxidised by a stream of oxygen in a closed circulating system at 160°, the gas leaving the oil being passed through sulphuric acid and soda-lime purifiers and over a heated platinum spiral to destroy hydrocarbons, of which a considerable volume was produced during the experiments. Oxidation was followed by measuring the oxygen absorption of the oil. The rate of oxidation decreased almost in direct proportion to the amount of oxygen consumed, until gelation occurred. The addition of driers had relatively little effect on the rate of reaction and the total oxygen absorption was unchanged. Triolein was oxidised at a rate not much below that of linseed oil, but required about 50% more oxygen to produce gelation. The total oxygen absorbed before gelation was about 18.5 wt.-% for triolein, 12—13% for linseed oil, and 8.8% for the linolenic ester of pentaerythritol. Linseed oil fatty acids absorbed oxygen faster than did the esters, but the rate decreased more rapidly, and 15% of oxygen was absorbed before gelation. Mixtures of oil and fatty acids, however, gelled more



rapidly and with less consumption of oxygen, than the pure oil. The quantity of oxygen remaining in the gels varied from 44% (for triolein) to 88% (trilinolenin) of the total oxygen consumption. Loss of carbon and hydrogen from the oils amounted only to 2–3 wt.-% up to the gelling point. It is concluded that gelation occurs when the molecule attains a certain size or degree of complexity or polarity, and is only indirectly a function of the degree of oxidation. The gelled oils showed a peroxide reaction, and ultimate analysis indicated that in the case of linseed oil and trilinolenin gelation occurred when sufficient oxygen had been absorbed to form one peroxide group in each molecule of unsaturated acid (with the pentaerythritol ester somewhat sooner).

E. LEWKOWITSCH.

**Manipulation of the Mackey test [for oleines].** H. WOLF and R. HEILINGÖTTER (Chem. Umschau, 1931, 38, 24–25; cf. B., 1931, 124).—The heating effect due to the presence of metallic soaps is distinguished from that due to unsaturated acids. The time (1½ hrs.) allowed for the test should be counted from the moment when the oiled wool attains 70°; a forced air current is unnecessary.

E. LEWKOWITSCH.

**Colour reactions with certain oils and fats.** A. F. MCCARLEY (Analyst, 1931, 56, 104–105).—Characteristic colours are produced by the addition of 3–4 drops of certain vegetable and animal oils or melted fats to a saturated solution of resorcinol in chloroform followed by 5 drops of hydrochloric acid saturated with bromine, added with vigorous shaking. Thus cod-liver oil produces a pink colour, slowly becoming deep red on keeping; butter fat, whale oil, seal oil, soya-bean oil, and rape oil produce various shades of pink; linseed oil gives a deep green, and palm oil a light-green coloration; olive, coconut, and almond oils give no colour. The reactive oils thus appear to be those which contain vitamin-A. A. R. POWELL.

**Jack pine.**—See XIII. **Cheese.**—See XIX.

## PATENTS.

**Separating the constituents of mixtures containing soap and neutral oil or fat.** METALLGES. A.-G. (B.P. 341,141, 22.10.29. Ger., 31.10.28).—The neutral fat in the mixture, e.g., soap stock, is extracted with a suitable solvent (two or three extractions), and recovered by evaporating the solution. The soap residue, containing solvent, is acidified with, e.g., 20% excess of aqueous sulphuric acid, the solvent is evaporated, and the fatty acids are allowed to settle out over the aqueous liquor.

E. LEWKOWITSCH.

**Continuous extraction process [for oil-bearing seeds] and apparatus therefor.** I. G. FARBENIND. (B.P. 341,581, 30.10.29. Ger., 27.7.29).—The seeds are pushed down, then up, through a U-shaped passage while the extraction liquid is passed countercurrent to it. The material is caused to progress by occasional long strokes of a piston (at the feed end of the passage) which at other times is kept moving in short strokes to maintain vibrations in the material.

B. M. VENABLES.

**Extraction of oil from vegetable matter.** K. YOSHIDA (B.P. 341,079, 7.10.29. Jap., 29.10.28).—The

comminuted oilseed etc. is soaked in hot oil (fatty or mineral) in order to drive off the moisture and to loosen the tissues, then drained while hot, and pressed in the usual way.

E. LEWKOWITSCH.

**Petroleum emulsions. Hydrocarbon-alcohol mixtures.**—See II. **Esters. Wetting etc. agents.** See III. **Bonding rubber to metal.**—See XIV.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Turpentine and wood-turpentine oils.** V. KRES-TINSKI. I. Composition of wood-turpentine from *Pinus sylvestris*. II. Lævorotatory turpentine oil from *Pinus sylvestris*. III. Composition of alcohol fraction of wood-turpentine (J. pr. Chem., 1931, [ii], 129, 97–103, 104–114, 115–120).—I. [With A. LIVEROVSKI and W. MALMBERG.] A further specimen of old root resin (cf. B., 1930, 621), showing results analogous to those of the “sulphur” turpentine, has been examined. The “alkaline” oil,  $d_{20}^{20}$  0.866  $[\alpha]_D +13.64^\circ$ ,  $n_D^{20}$  1.4716, is obtained by distillation at 13 mm. in 21 fractions. The fractions are shown graphically to be divisible into four zones: (a)  $d$ - $\alpha$ -pinene approx. 67%,  $l$ - $\Delta^3$ -carene (now first observed in  $l$ -form) 20%, terpene alcohols 3%, sesquiterpene and residue 10%. The residue yields after washing with alcohol and recrystallisation a substance  $C_{10}H_{20}$ , m.p. 51°, whilst a further compound is obtained from the alcohol washings. The “steam” oil,  $d_{20}^{20}$  0.860,  $[\alpha]_D +12.60^\circ$ , gave similar results.

II. [With L. BASHENOVA-KOSLOVSKAIA.] A sample of lævorotatory oil,  $[\alpha] -6.01^\circ$ ,  $d_{20}^{20}$  0.873,  $n_D^{20}$  1.4726, is shown similarly to consist (approx.) of:  $\alpha$ -pinene (69%), camphene (5%),  $\Delta^3$ -carene 1.5%, phellandrene 1.5%, together with 10% of alcohols, sesquiterpenes, and residue. Oxidation of the middle fractions, which show abnormal dispersion, gives no nopinic acid (absence of  $\beta$ -pinene), but a little *cis*-caronic acid, derived from  $\Delta^4$ -carene was present. The abnormality is probably due to an unidentified  $d$ -compound.

III. [With F. SOLODKI and G. TOLSKI.] A preliminary investigation of the alcohol fraction (7–14%) of wood-turpentine (2 samples:  $d_{16}^{16}$  0.920, 0.916;  $[\alpha]_D +15.56^\circ$ ,  $+20.16^\circ$ ;  $[\alpha]_{546.1}^{20}/[\alpha]_{578}^{20}$  1.42, 1.54), shows it to consist of: (a)  $\alpha$ -pinene 5–10%, (b)  $\Delta^3$ -carene 10–12%, (c) and (d) terpene alcohols 37–48% and 23–24%, and (e) sesquiterpenes 8–23%. Fractions (c) and (d) probably contain alcohols of the *p*- and *m*-pineol series, respectively, since when mixed and dehydrated with oxalic acid they yield a mixture of terpinene and sylvestrene, and when saturated with hydrogen chloride give terpinene and sylvestrene dihydrochlorides. Careful fractionation of (c) yields camphor (1.5%), after the removal of which a saturated and an unsaturated alcohol are shown by oxidation and bromination experiments to be present; from (e) are obtained two dihydrochlorides, m.p. 114–115° (? cadinene) and 82–83°.

E. HOPKINS.

**Evaluation of turpentine oil.** V. KUBELKA and A. SCHNEIDER (Chem. Umschau, 1931, 38, 29–32).—Turpentine should be evaluated by following the whole course of a distillation performed under standardised conditions. The curve, temperature–volume distilled,

provides an excellent means for comparing samples. True balsam turpentine give almost horizontal distillation curves over the whole range; in aged specimens higher-boiling constituents have been produced and are indicated by a rise of the curve near the end of the test. Highly purified pine oils give smooth curves covering a rather wider temperature range; cruder oils give curves more or less irregular and inclined to the vertical. Full figures are given for a number of samples.

E. LEWKOWITSCH.

**Some properties of oil paints.** W. DROSTE (Farben-Ztg., 1931, 36, 916—918).—A dissertation in general terms dealing with the significance of such factors as proportion of vehicle in individual and composite paint coatings, consistency of ready-mixed and paste paints, and their standardisation, degree of grinding and wetting, and retention of properties on storage. S. S. WOOLF.

**Painting of wood. I. Influence of wood structure on paint behaviour.** J. H. HASLAM and S. WERTHAN (Ind. Eng. Chem., 1931, 23, 226—233).—The structure of soft woods is discussed at length and illustrated by photomicrographs etc., special reference being made to the changes occurring in spring and summer woods under the influence of moisture. The penetration of the wood by the vehicle of applied paint was studied by microscopical examination of thin sections stained selectively with Brilliant Green (staining wood) and Soudan III or IV or Scarlet R (staining oil). The effects of the changes in paint film and wood during ageing and exposure on adhesion, porosity, and durability of the paint are discussed. It is indicated that a slight uniform penetration of paint vehicle into wood is more desirable than deep irregular penetration (achieved, e.g., by undue thinning). S. S. WOOLF.

**New rust-preventive paints as red lead substitutes.** J. F. SACHER (Chem.-Ztg., 1930, 54, 781—782).—"Arcanol" ("grey red lead"), a pigment containing finely-divided lead and lead oxide, is considered to be free from the storage and colour disadvantages of red lead. "Tropisco" or "Tropic" paints are new oil-free products that dry by evaporation of solvent only. Rust prevention, heat resistance, anti-fouling properties, etc. are claimed for them. S. S. WOOLF.

**Manufacture of pigments.** L. V. ITZKOVICH and A. I. GELIKH (J. Chem. Ind., Moscow, 1929, 6, 1678—1685).—Of seven methods of production of lead chrome, those employing insoluble salts as starting materials are preferred; the  $p_H$  of the mixture and the dispersion of the insoluble salts influence the product. The preparation of artificial vermilion is reviewed. Calcination of kaolin (1 hr. at 650—700°) for use in the manufacture of ultramarine increases its reactivity; its dispersion is particularly important. Definite conditions must be observed during ignition. CHEMICAL ABSTRACTS.

**Emerald (chrome)-green.** V. N. SCHULTZ, Y. P. NIKOLSKAYA, and L. F. PENKOVA (J. Chem. Ind., Moscow, 1929, 6, 1412—1418).—The pigment is formed according to the equation  $3K_2Cr_2O_7 + 12H_3BO_3 = 3K_2B_4O_7 + 3Cr_2O_3 \cdot 2H_2O + 4 \cdot 5O_2 + 12H_2O$ . Two or three times the theoretical quantity of boric acid should be used, and the excess removed from the pigment. The water content can be reduced to about 5% without

injury to the pigment. The pigment obtained when arsenious acid is used instead of boric acid is not sufficiently bright, whilst phosphoric acid gives a pigment which is readily soluble in water.

CHEMICAL ABSTRACTS.

**Electrolytic manufacture of white lead.** J. F. SACHER (Chem.-Ztg., 1931, 55, 189—190).—Electrolytic methods which have been suggested in the journal and patent literature for the manufacture of white lead without danger to the health of the operatives caused by dusting are critically reviewed. None of the methods appears to yield such a uniform and satisfactory product as is obtained by the chamber process. A. R. POWELL.

**Luminescence analysis. II. Luminescence of white pigments and application of luminescence analysis to the examination of paintings.** E. BEUTEL and A. KUTZELNIGG (Monatsh., 1931, 57, 9—14; cf. A., 1930, 727).—Zinc white, titanium white, and white lead give well-marked luminescence when irradiated by a quartz mercury lamp. The luminescence of zinc white is so intense that a specimen containing 10% is scarcely distinguishable from the pure substance. White lead is characterised by the non-uniform nature of the luminescence. The character of the luminescence of white lead, but not of the other pigments, undergoes a striking change when examined in a medium of linseed or poppy oil, owing to combination with a constituent of the oil. The effect varies with time and with exposure to daylight, circumstances which make it possible to gain information about the history of oil paintings in which white lead has been used. F. L. USHER.

**Colorimetry of pigments and a suggested scale of fastness.** G. F. NEW, G. S. DISNEY, and D. L. TILLEARD (J. Oil Col. Chem. Assoc., 1931, 14, 3—44).—Any unknown colour can be specified or matched by a mixture of any three other coloured radiations, and it is best to choose these primaries in such a way that the maximum proportion of the colour field can be matched with simple positive additions of the three. In the series of experiments on the fading of pigments described by the authors, the Guild trichromatic colorimeter was used throughout, and the effects of solid diluents, different media, and varying thicknesses of pigment film were observed in detail. It is found that fading during the first 50 hrs.' exposure to the carbon arc is occasionally abnormal, and should not be used for purposes of comparison; a fading period of 250 hrs. is considered ample for determining the relative fastness of all dye pigments. It is suggested that fastness should be specified by a statement of the extent of the fading which will take place over some definite period of time when the pigments are exposed to a definite intensity of radiation under known conditions.

F. C. HARWOOD.

**Nature of the resins in jack pine (*Pinus banksiana*).** H. HIBBERT and J. B. PHILLIPS (Canad. J. Res., 1931, 4, 1—34).—Analyses of resin from seasoned and green jack pine are recorded. The total acid content was the same in both cases; fats, fatty acids, phytosterol, and "resene" were higher in the green wood, whilst unsaponifiable matter, resin acids, and unsaponifiable polymerised terpenic matter were higher

in the seasoned wood. A higher proportion of crystalline acids was found among the resin acids from green wood. The fatty constituents, mainly unsaturated, consisted of free acids, glycerides, and other esters. Less linoleic acid occurred in the glycerides from seasoned than from green wood. The proportion of oleic acid was practically the same in both cases. The total unsaturated fatty acids in the glycerides and free acids was higher in the green wood. Seasoned wood contained a high proportion of oxidised matter in the fatty glycerides, suggesting extensive polymerisation or decomposition during seasoning. Small amounts (1.5 and 4.3%) of essential oils were present. A. G. POLLARD.

**Compatibility of resins with nitrocellulose solutions.** H. E. HOFMANN (Ind. Eng. Chem., 1931, 23, 127—130).—Mixtures of resins and nitrocellulose were treated with various solvents, and the resulting solutions were classified according as they gave cloudy solutions, or clear solutions giving rise to clear or cloudy films. Further additions of different solvents and plasticisers to clarify cloudy solutions and films were made. From the results of these tests, which are tabulated, solvents and resins are grouped into alcohol and hydrocarbon types, and it is shown that compatibility is most readily obtained by the use of resin and solvent in the same group. In correcting incompatibility the effects of alcohol- and hydrocarbon-type ingredients appear to be complementary. "Gum blush" is due to the different rates of evaporation of the solvent constituents: thus gradual increase in relative concentration of alcohol-type solvent will tend to cause film-clouding with hydrocarbon-type resins and *vice versa*. The various factors discussed are illustrated by ternary diagrams. S. S. WOOLF.

**Cobalt driers.** E. GEBAUER-FÜLNEGG and G. KONOPATSCHEK (Ind. Eng. Chem., 1931, 23, 163—165).—The drying action on tung oil of solutions of cobalt compounds of well-defined chemical constitution, *e.g.*, cobalt thiocyanate, chloride, or nitrosoacetophenone (as distinct from chemically non-uniform resinates, oleates, etc.), in various organic solvents readily miscible with the oil, *e.g.*, benzonitrile, benzaldehyde, benzyl alcohol, benzyl cyanide, and nitrobenzene, was observed and found to be generally efficient. Of other solvents tested, benzene, benzyl chloride, furfuraldehyde, aniline, and amyl alcohol proved unsatisfactory for various reasons. It was shown that cobalt compounds having apparently the same solubility in the organic solvent-oil mixture did not exhibit the same drying activity, and also that the character of the dried tung oil film changed with the solvent used, clear films with very slight wrinkling only (of types characteristic of the solvent) being obtained with several solvents. A conception of the action of a siccativ is propounded. S. S. WOOLF.

#### PATENTS.

**Impregnating composition: its preparation and use.** W. C. GEER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,773,101, 19.8.30. Appl., 17.8.22).—An oil such as rape, linseed, or tung oil is dissolved, together with a gum (preferably chlorinated rubber or a varnish resin), in a solvent such as benzene; the solution is then stiffened by oxidation or vulcanisation, *e.g.*, with sul-

phur chloride. After mashing, it may be used as a coating for cardboard food containers, as an acid-resistant varnish for metals, or as a dope for balloon fabric. Suitable proportions are: rubber heptachloride 5 g., benzene 25 g., sulphur chloride 1 g., and oil 5 g., added in the order given. D. F. TWISS.

**Resistance paint and [electrical] resistances.** L. L. JONES, A. SCHMIDT, and J. JONES, Assrs. to TECHNIDYNE CORP. (U.S.P. 1,773,105, 19.8.30. Appl., 11.2.27).—Insulated bases are coated with a very thin electrically conductive film of a resistance paint comprising colloidal graphite in a vehicle consisting of a metallic phosphate, *e.g.*, aluminium phosphate, in a slight excess of phosphoric acid. S. S. WOOLF.

**Manufacture of black colouring matter.** S. KISLITZIN, Assr. to STATE IMPORT & EXPORT TRADING OFFICE "GOSTORG" (U.S.P. 1,774,202, 26.8.30. Appl., 28.11.28. U.S.S.R., 23.6.26).—Raw peat is treated with an aqueous solution of an alkali-metal salt, *e.g.*, common salt, and an aluminium salt, *e.g.*, alum, and after drying and disintegrating is mixed with a bituminous material, *e.g.*, tar. The mixture is then destructively distilled, the solid residue therefrom ground wet and treated with hot hydrochloric acid, and the resulting pigment separated, washed, dried, and ground. S. S. WOOLF.

**Manufacture of mixed crystal pigments.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,881, 16.9.29).—A coloured mineral salt, *e.g.*, barium manganate or strontium ferrate, is combined with at least two further mineral salts of the same chemical type and crystal lattice structure to form mixed crystals with the first-mentioned salt, at least one of the accessory salts having  $n_D$  above 1.63. Such salts are alkaline-earth sulphates, selenates, tungstates, molybdates, etc. S. S. WOOLF.

**Manufacture of colour lakes.** I. G. FARBENIND. A.-G. (B.P. 318,834, 9.9.29. Ger., 8.9.28).—A basic dye is precipitated from an aqueous solution, in the presence or absence of a substratum, with a water-soluble naphthalene- or anthracene-sulphonic acid, or certain nuclear-substituted or hydrogenated products of such acids, which may not themselves be dyes. S. S. WOOLF.

**Reducing the viscosity of nitrocellulose solutions.** E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,770,108, 8.7.30. Appl., 16.10.22. Renewed 26.9.23).—Neutral or slightly acid nitrocellulose solutions are maintained at 35—60° in the presence of an alkaline substance of denitrating power substantially less than that of calcium sulphide, *e.g.*, alkali hydroxides, in quantity insufficient to reduce the nitrogen content of the nitrocellulose below 10.5%, *e.g.*, less than 5% of the weight of nitrocellulose. The alkaline substance may be neutralised by addition of hydrochloric or sulphuric acid when the requisite viscosity reduction has been achieved. S. S. WOOLF.

**Manufacture of lacquer coatings, films, plastic masses, and artificial threads.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,288, 26.9.29).—In preparing such coatings etc. based on cellulose ethers, *e.g.*, butyl- or benzyl-cellulose, a quantity up to 5% of

organic bases, *e.g.*, substituted guanidines or aralkylated aromatic bases, is added to the starting materials. If desired, further additions of inorganic bases, *e.g.*, zinc oxide or white lead, may be made. The bases of each type should be non-volatile or difficultly volatile.

S. S. WOOLF.

**Compositions having a basis of a cellulose derivative.** BRIT. CELANESE, LTD. (B.P. 342,429, 26.11.29. U.S., 26.11.28).—Mixtures of organic derivatives of cellulose, *e.g.*, cellulose acetate, with diphenylolcycloparaffins, *e.g.*, the condensation product of phenol and cyclohexane, and, if desired, natural or synthetic resins, plasticisers, solvents, pigments, and/or dyes, are claimed. Eight examples are given, useful in the manufacturing of reinforced glass.

S. S. WOOLF.

**Composition containing cellulose esters.** J. G. DAVIDSON, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,770,153, 8.7.30. Appl., 31.5.27. Cf. U.S.P. 1,633,927; B., 1927, 859).—Cellulose ester compositions are plasticised with polyolefine glycol ethers, *e.g.*, diethylene glycol monoethyl ether. S. S. WOOLF.

**Varnishes for coating etc.** H. WADE. From BAKELITE CORP. (B.P. 342,286, 24.9.29).—A cellulose ester, *e.g.*, cellulose nitrate (preferably 5–10% by wt. of the total solids), is incorporated with a solution of the resinous reaction product of a phenol, a fatty oil, *e.g.*, tung oil, and a methylene-containing substance, *e.g.*, formaldehyde.

S. S. WOOLF.

**Coating of surfaces or articles.** BRIT. CELANESE, LTD. (B.P. 342,211, 9.4.30. U.S., 22.5.29).—In the coating of surfaces with cellulose lacquers, *e.g.*, those based on cellulose acetate, priming or filling coats containing a non-drying oil, *e.g.*, coconut oil or the fatty acids thereof, preferably at least partly compatible with the cellulose lacquer are used; plasticisers, synthetic resin, fillers, and/or pigments may be incorporated, if desired.

S. S. WOOLF.

**[Pyroxylin] composition.** J. F. WALSH, H. E. SMITH, and A. F. CAPRIO, Assrs. to CELLULOID CORP. (U.S.P. 1,772,529, 12.8.30. Appl., 29.12.26).—Butyl phthalate (10 pts.) is mixed with 100 pts. of moist pyroxylin, the resultant cake is freed from water, and 20 pts. of camphor in solution are incorporated.

S. S. WOOLF.

**[Spirit-oil "combination"] varnish.** M. FECHTER (U.S.P. 1,773,666, 19.8.30. Appl., 6.5.25).—An alcoholic solution of pyroxylin including sassafras oil is incorporated with a mixture of a spirit varnish including oxalic acid, *e.g.*, a sandarac-mastic solution, and an oil varnish, *e.g.*, dammar-linseed oil-turpentine, to give a hard, quick-drying varnish which obviates the need for a priming coat of shellac on wood. S. S. WOOLF.

**Synthetic resins.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 341,012, 1.7.29).—Polyhydric alcohol-polybasic acid synthetic resins are modified by the use of pure  $\beta$ -elaeostearic acid (details of preparation from tung oil given) together with other monobasic acids, *e.g.*, resin acids, if desired, for partial esterification of the alcohol component. S. S. WOOLF.

**Manufacture of substantially anhydrous finely-divided synthetic resins.** E. E. NOVOTNY, Assr. to

J. S. STOKES (U.S.P. 1,771,139 and 1,771,140, 22.7.30. Appl., 12.3.28).—(A) A relatively wet, sludge-like, synthetic resin mass containing occluded water or mother-liquor is cooled to a temperature at which it becomes substantially solid and can be mechanically ground, but below which the ground particles will agglomerate. The finely-ground particles are then dehydrated by means of air currents etc., thus preventing agglomeration and removing volatile substances. (B) The mass, of low volatile content, is atomised and quickly wetted by directing the spray into water. The finely-divided material is then separated from the water and dried.

S. S. WOOLF.

**Synthetic resin manufacture.** E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,773,598, 19.8.30. Appl., 12.8.27).—In the reaction between phenols, *e.g.*, xylenols, and active methylene compounds, *e.g.*, formaldehyde, in the absence of catalysts, water vapour, including that formed by the condensation, is removed from the reaction chamber as fast as it is formed. The product, which may, if desired, be hardened, *e.g.*, by use of furfuraldehyde, and admixed with pigments etc., has high dielectric values under adverse conditions of humidity and heat.

S. S. WOOLF.

**Decolorisation of phenol-formaldehyde artificial resins condensed by means of ammonia.** A. JÄGER, and HEROLD A.-G. (B.P. 341,083, 9.10.29).—Dithiocarbamates of strong organic bases, *e.g.*, piperidine pentamethylenedithiocarbamate, are added to the phenol-formaldehyde-ammonia reaction mixture before or after condensation, and the resin formed by condensation is evaporated until it breaks under water, the product being colourless.

S. S. WOOLF.

**Decolorisation of synthetic resins.** BRIT. CELANESE, LTD. (B.P. 342,614, 2.5.30. U.S., 11.5.29).—Aromatic sulphonamide-aldehyde resins are treated with an absorbent decolorising agent, *e.g.*, bone black, carbon black, activated charcoal, fuller's earth, silica gel.

S. S. WOOLF.

**Manufacture of artificial masses.** SOC. CHEM. IND. IN BASLE (B.P. 342,325–6, 23.10.29. Switz., 23.10.28. Addns. [A, B] to B.P. 284,589; B., 1928, 826).—(A) An aromatic amine (1 mol.) is condensed, in the presence of an acid, with formaldehyde (not more than 1 mol.) or the equivalent amount of a substance yielding it, and the fusible resin obtained after elimination of the acid is dried. A further quantity of the aldehyde component is then added and the mixture is comminuted and compressed, alone or admixed with fillers, colouring matter, or plasticisers. (B) Filling material is saturated with condensation product before the acid is eliminated; thus, when the acid binding agent is added the condensation product is precipitated in and on the filler, thoroughly impregnating it.

S. S. WOOLF.

**Binding material for use in the manufacture of compressed fibrous material.** HALIZITE CORP., Assees. of A. HAWERLANDER (B.P. 342,278, 30.9.29. U.S., 13.3.29. Cf. B.P. 336,754; B., 1931, 15).—A mixture of 1 kg. of resorcinol and 1 litre of formalin is heated to 30°, 3 g. of sodium sulphite are added, and the mixture is heated to 75° and quickly cooled to 25°.

[Stat. ref.] S. S. WOOLF.

[Composite] film. C. ELLIS (U.S.P. 1,773,974, 26.8.30. Appl., 23.9.26).—Duplex films are made by covering a support with one or more coats of a pigmented nitrocellulose (etc.) substratum, applying thereto, when dry, a substantially transparent stratum comprising low-viscosity nitrocellulose and a synthetic resin compatible therewith, *e.g.*, of the "oily fatty acid-phthalic-glyceride" type, and stripping the dry product from the support. S. S. WOOLF.

Hydrocarbon-alcohol mixtures.—See II. Cellulose ethers etc. Paper products.—See V. Laminated glass.—See VIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Determination of  $p_H$  of ammonia[cal] latex. J. MCGAVACK and J. S. RUMBOLD (Ind. Eng. Chem. [Anal.], 1931, 3, 94—97).—Most of the indicators available for  $p_H$  determinations in alkaline liquids are unsatisfactory on account of their small colour change; the presence of proteins also frequently causes large errors. The glass electrode is very satisfactory with latex; up to  $p_H$  9.5 the variation is negligible, and a reasonable degree of accuracy is obtained even at  $p_H$  11.0; above this value, however, it is necessary to calibrate each electrode, using, *e.g.*, Sørensen's glycine-sodium chloride-sodium hydroxide standards. (Cf. Hughes, A., 1928, 370.) D. F. TWISS.

Determination of alkalinity of reclaimed rubber. H. F. PALMER and G. W. MILLER (Ind. Eng. Chem. [Anal.], 1931, 3, 45—48; cf. Shepard, Palmer, and Miller, B., 1928, 238).—The reclaim, sheeted to a thickness of approx. 0.001 in. and torn into pieces about 1 in. in diam., is heated under reflux on a hot-plate at 110° with a neutral mixture of benzene (60 c.c.), alcohol (40 c.c.), and water (100 c.c.) for 2 hrs. More water (500 c.c.) is then added and the benzene removed by evaporation. The solution is decanted and the solid then washed thrice with 75 c.c. of boiling distilled water. Hydrochloric acid (0.1N) is added to render the combined washings and original extract distinctly acid, followed by approx. 5 c.c. each of 3% potassium iodate solution and of *N*-potassium iodide; after 3 min. the same volume of 0.1N-thiosulphate is then added as of *N*-hydrochloric acid earlier, and after 15 min. the mixture is titrated with 0.1N-iodine and starch. This indirect method of titration avoids the difficulty offered by the colour of the extract. The procedure gives much more accurate results than those obtainable previously (*loc. cit.*). D. F. TWISS.

Manganese and iron contents of various types of rubber and fillers and their importance for the ageing behaviour of rubber. F. KIRCHHOF (Kautschuk, 1931, 7, 26—33).—Washed plantation *Hevea* rubber normally contains manganese up to about 0.0001% Mn, but wild rubbers may contain up to 0.002%; raw *Tijpetir gutta* also shows a distinct manganese content. Sticky low-grade rubbers always have a relatively high content of manganese and of iron. The contents of these metals are not related in any very definite way to the percentage of ash, and it is possible that the manganese is, in part at least, in organic combination. This possibility is supported by the fact that

the viscosity of sticky low-grade rubbers varies inversely as the proportion of manganese in the soluble portion. In a sample of Peruvian ball rubber containing 0.007% Mn and also iron, the condition was one of fluidity rather than stickiness. The degradation is attributable to an oxidation-depolymerisation process in which the manganese acts as catalyst; the change can eventually proceed further to the formation of a hard resinous surface layer. Some mineral fillers such as ochres, umbers, whiting, and mineral blacks also contain distinct proportions of manganese and impair the ageing qualities of vulcanised mixtures, which consequently show a rapid increase in the acetone-soluble fraction. The presence of 0.05% Mn in a vulcanisate is regarded as dangerous, particularly if the mixing contains much iron oxide. D. F. TWISS.

Organic colours in the rubber industry. F. JACOBS (Kautschuk, 1931, 7, 22—26).—A large number of organic rubber colours of British, American, German, and French manufacture are brought under review as to their behaviour during vulcanisation (in open steam) in rubber mixings containing various accelerators and with and without the additional presence of tube reclaim. The presence of reclaim is generally detrimental to the colour and necessitates the use of an increased proportion of white filler and of pigment. The identity of certain colours sold commercially under different names is indicated. D. F. TWISS.

#### PATENTS.

Coagulation of [rubber] latex. GOODYEAR TIRE & RUBBER Co., Assees. of A. J. GRACIA (B.P. 342,485, 2.1.30. U.S., 8.4.29).—Latex is coagulated by the addition of an amine of the formula  $CH_2R \cdot NR'R''$ , where R is an aromatic or aliphatic hydrocarbon group and R' and R'' may be hydrocarbon groups or hydrogen. Examples are ethylamine, diethylamine, and their homologues; a satisfactory proportion is 5 c.c. per 50 c.c. of latex. D. F. TWISS.

Production of aqueous dispersions of organic substances [for addition to rubber latex]. DUNLOP RUBBER Co., LTD., and ANODE RUBBER Co., LTD., Assees. of A. SZEGVARI (B.P. 342,194, 18.3.30. U.S., 20.4.29).—"Rubber oil," prepared by the destructive distillation of raw or vulcanised rubber, is used to dissolve organic materials such as accelerators or anti-oxidants, *e.g.*, of the aldehyde-amine type, asphaltum, or vaseline. The solutions are then dispersed in water by shaking, preferably with the addition of emulsifying agents. D. F. TWISS.

Thickening and stabilising of [rubber] latex. M. C. TEAGUE, ASSR. to AMER. RUBBER Co. (U.S.P. 1,772,647, 12.8.30. Appl., 7.12.27).—Latex of not less than 60% concentration, compounded if desired, is thickened and stabilised by the addition of at least 0.5% of saponin (rubber 100). The product is suitable for such purposes as dipping, spreading, extruding. D. F. TWISS.

(A) Treatment, (B) preservation, of [rubber] latex. J. MCGAVACK, ASSR. to NAUGATUCK CHEM. Co. (U.S.P. 1,772,752—3, 12.8.30. Appl., [A] 27.3.29, [B] 16.4.29).—(A) In the concentrating of latex by creaming, economy may be effected in the amount of creaming agent (*e.g.*,

ammonium alginate) consumed, and an increased proportion of serum solids may be ensured in the cream, by introducing into the uncreamed latex a quantity of the serum obtained in a previous creaming operation. (b) The soluble salts of selenious and tellurous acids are effective anti-coagulants for rubber latex: 0.1% (on the latex) will preserve latex for several months.

D. F. TWISS.

**Vulcanisation of rubber.** B. S. GARVEY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,774,322, 26.8.30. Appl., 19.4.29).—Rubber containing sufficient rubber for complete vulcanisation is mixed with a metallic trithiocarbonate, *e.g.*, barium trithiocarbonate, and a substance such as a secondary aliphatic amine or a reactive non-volatile derivative of such a base, *e.g.*, piperidine-formaldehyde. Such mixtures show substantially no tendency to vulcanisation below 100°, but at higher temperatures decomposition of the trithiocarbonate occurs with liberation of carbon disulphide; this then combines with the secondary amine with formation of a very active accelerator of vulcanisation.

D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,773,379, 19.8.30. Appl., 10.5.29).—The reaction product obtained from molecular proportions of an aliphatic aldehyde, such as acetaldehyde or butaldehyde, and a hydroxyaryl compound, such as phenol or  $\beta$ -naphthol, in the presence of a small proportion of condensing agent, *e.g.*, hydrochloric acid, is further heated at 170–200° with an atomic proportion of sulphur and a catalyst, *e.g.*, 0.2% of iodine. The final product is of value as an anti-oxidant in vulcanised rubber.

D. F. TWISS.

**Treatment of rubber.** NAUGATUCK CHEM. Co., Assees. of L. H. HOWLAND (B.P. 342,634, 6.6.30. U.S., 22.6.29).—The products obtained by the interaction of a carboxylic acid, *e.g.*, acetic, stearic, or salicylic acid, with a diarylamine, *e.g.*, diphenylamine or phenylnaphthylamine, in the presence of a dehydrating agent are of value for retarding the deterioration of rubber. They are *meso*-substituted acridines of the general formula  $RC \begin{smallmatrix} \diagup R' \\ \diagdown R'' \end{smallmatrix} N$ , where R' and R'' are *o*-arylene radicals and R may be any substituted organic radical including, if desired, an additional *meso*-substituted acridine.

D. F. TWISS.

**Treatment of rubber.** NAUGATUCK CHEM. Co., Assees. of S. M. CADWELL and S. I. STRICKHOUSER (B.P. 342,502, 18.1.30. U.S., 19.1.29).—Deterioration of rubber during vulcanisation and subsequently is retarded, without leading to discoloration, by incorporating a mixture of an organic base and a naphthol, particularly a monohydric naphthol. The naphthol neutralises the accelerating effect of the organic base and at the same time itself acquires anti-oxidant properties, thereby augmenting the anti-oxidant effect of the base. Suitable bases are, *e.g.*, open-chain or other polyalkylenepolyamines, *pp'*-diaminodiphenylmethane, and their aldehyde or nitroso-derivatives.

D. F. TWISS.

**Dispersed plastic masses.** K.D.P., LTD. (B.P. 342,469, 20.12.29. Ger., 22.12.28).—By the addition of

thixotropic colloids such as bentonite, or of lithium sulphate or other suitable electrolytes, the natural or added colloids in colloidal dispersions of plastic materials such as rubber are rendered thixotropic. Liquids such as oil may also be added to increase the elasticity of the thixotropic mixture. The ability of such mixtures to undergo reversible conversion into a fluid condition by agitation renders them advantageous for manufacturing operations such as dipping, spreading, etc.

D. F. TWISS.

**Manufacture of rubber-like masses and articles therefrom.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,314, 30.8.29. Addn. to B.P. 339,255; B., 1931, 264).—In the process of the prior patent, the styrene can be replaced by other olefines such as homologues of styrene, benzene hydrocarbons containing two or more substituent vinyl groups, or homologues of these hydrocarbons.

D. F. TWISS.

**Production of variated surface effects on rubber articles.** J. B. CROCKETT, Assr. to CAMBRIDGE RUBBER Co. (U.S.P. 1,773,724, 26.8.30. Appl., 14.5.29).—A former of the desired shape is coated, by spreading, spraying, or similar procedure, with a layer of vulcanisable or vulcanised compounded latex containing a high proportion of mineral filler. On drying, the rubber layer cracks or checks in an irregular manner over its entire surface. After drying, compounded latex of contrasting colour and containing a much lower proportion of filler is applied to the coated former until the article has been built up to the desired thickness. The article is then dried and/or vulcanised preferably before stripping. The character of the irregular pattern may be modified by varying the proportion of filler and the drying temperature of the first coating.

D. F. TWISS.

**Composite product [rubber bonded to metal].** H. GRAY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,774,324, 26.8.30. Appl., 22.4.27).—A vegetable oil (tung, linseed, castor, or rosin oil) is treated with one or more of a class of compounds comprising strong inorganic, non-oxidising acids and compounds capable of undergoing thermal or hydrolytic dissociation to form such acids, *e.g.*, sulphuric acid, aluminium chloride, phosphorus oxychloride, sulphur chloride, benzotrichloride, trichloroacetic acid. The products form strong bonds between solid materials, particularly metals and rubber.

D. F. TWISS.

**Production of combinations of rubber and paper.** R. P. ROSE and H. E. CUDE, Assrs. to GEN. RUBBER Co. (U.S.P. 1,773,201, 19.8.30. Appl., 18.12.26).—During the beating of papermaking fibre, a protective colloid, such as glue or a soluble starch ester, is added. Rubber latex is then introduced, preferably after rendering the fibre mixture alkaline, and the deposition of the rubber on the fibre is then effected, *e.g.*, by the addition of a coagulant. When glue or other protein is used as protective colloid it is advantageous to effect precipitation of the protein as well as of the rubber. This procedure facilitates the manufacturing operations and gives rubber particles of much smaller size than those in ordinary latex paper.

D. F. TWISS.

**Impregnating composition.**—See XIII.



## XV.—LEATHER; GLUE.

**Defects in raw hides and their resulting effects on the finished leather.** F. STATHER (J. Soc. Leather Trades' Chem., 1931, 15, 12—21).—In addition to mechanical damage and damage arising from parasitic attack and pathological causes, many defects arise through the action of micro-organisms, which can cause salt stains, red coloration, violet stains, low grain, decomposed grain, development of arteries, and the marbling of the grain. Stains or grain damage is produced by alum, lime, iron, copper, and chromium salts, also by the crystallisation of salts in the skins. Stains are produced by dung and urine. Damage is caused if the hides are allowed to heat, sweat, or dry too quickly. These various causes are discussed in detail and in some cases the appearance of or effect on the finished leather is described.

D. WOODROFFE.

**Origin of ammonia found in lime liquors [from treatment of hides or skins].** R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1931, 15, 25—30).—By treatment of hair and collagen, respectively, with lime liquor, it was shown that relatively large amounts of ammonia were formed from collagen, and after 8 days the ratio of non-amino- to amino-nitrogen was larger than in any commercial liquor which had been tested. The ratio for the hair was not so large. Analysis of the lime liquors and of the protein matter showed that, with collagen, all the ammonia in the lime liquor was derived from the amino-groupings of the protein. The ammonia derived from the hair was greater than could have been derived from the amino-groupings alone, and it is concluded that the excess must have resulted from the decomposition of cystine groups. The ammonia in lime liquors is derived partly from the decomposition of protein amino-groups and partly from the alkaline hydrolysis of the cystine groups in the hair and of the albumins and globulins in the interfibrillary proteins.

D. WOODROFFE.

**Importance of the  $p_H$  of pelt in vegetable tanning.** G. PEACE (J. Soc. Leather Trades' Chem., 1931, 15, 22—24).—Hide powder plumping curves are not strictly comparable with the plumping of hides in actual tanning. In most tanneries sole leather is tanned in liquors of too low a  $p_H$  value, resulting in dark cracky grain unless extra finishing processes are applied. The addition of acid to tan liquors to reduce the  $p_H$  to below 3 is unnecessary, since excellent substance, weight, and solidity are obtained at  $p_H$  values above 3. The  $p_H$  of the tan liquors regulates itself if the tannage is commenced in weak liquors, and if the  $p_H$  of the hide fibres is at the best value when tanning commences. The hides should be delimited completely and then their  $p_H$  adjusted to the required value, which will depend on the kind of leather to be made and the materials in use. The most useful values are  $p_H$  3.5—5.0.

D. WOODROFFE.

**Quick tanning.** M. BERGMANN, W. MUNZ, and L. SELIGSBERGER (J. Soc. Leather Trades' Chem., 1931, 15, 67—72).—The speed of penetration of solutions of Neradol ND,  $\alpha$ - and  $\beta$ -naphthalenesulphonic acids is 10—20 times as great as that of water. Although their solutions have  $p_H$  1.02—1.88, the swelling pro-

duced by them on hide powder is less than that in water. From figures obtained by the Dresden penetration apparatus, it has been shown that the capillaries of the pelt are enlarged by treatment of the latter with the above solutions. The sum of the water of swelling and of absorption is reduced, so that a reduction of swelling must have taken place. The pelt is rendered thicker, less compressible, and more resistant by the above solutions. By their use the rate of tanning action is increased.

D. WOODROFFE.

**Determination and control of the buffer index of tan liquors.** (MISS) W. B. PLEASS (J. Soc. Leather Trades' Chem., 1931, 15, 73—78).—The "buffer index" of a tan liquor (c.c. of *N*-hydrochloric acid or *N*-sodium hydroxide required to alter the  $p_H$  of 100 c.c. of the liquor at  $d$  1.020 by 1 unit) may be ascertained by diluting the liquor to  $d$  1.020, measuring its  $p_H$  value, and adding to 100 c.c. of the diluted liquor known volumes of *N*-sodium hydroxide (if the  $p_H$  is less than 3) or *N*-hydrochloric acid (if greater than 3) until the  $p_H$  value has been altered by 2 units, the  $p_H$  of the liquor being determined after each addition of acid or alkali. The  $p_H$  values are plotted against the volumes of alkali or acid added and the buffer index is deduced from the slope of the curve. For liquors of  $d < 1.020$ , the titration curve is obtained at the original concentration of the liquor and a correction factor to the buffer index, as computed at that concentration, is applied. This index is a measure of the mellowness of a tan liquor, and varies with the tanning materials used (bottom suspender liquor, average value 4.5; top, 3.0). Liquors of high buffer index usually give poor plumping, but a softer, fuller leather. Unevenness in the weave of the leather fibres is produced if the buffer index and  $p_H$  value vary much from the middle suspender liquor to the lower handlers. The buffer index is increased as the tan liquor passes down the yard or by additions of lactic acid, but is diminished by sulphuric acid. The  $p_H$  is slightly lowered by additions of these acids.

D. WOODROFFE.

**Fat-liquoring of chrome[-tanned] leather. Effect of hydrogen-ion concentrations on oil adsorption.** E. R. THEIS and F. S. HUNT (Ind. Eng. Chem., 1931, 23, 50—53).—Characteristic fat-adsorption curves for various oils were obtained by fat-liquoring chrome leather over the range  $p_H$  1—12. Maximum absorption of oil was shown at  $p_H$  4.0. Greater dispersion of the oil and speedier absorption were obtained by the addition of "triethanolamine" to the fat-liquor; less oil was absorbed, but the leather was more pliable. By varying the  $p_H$  of the fat-liquor, the adsorption curves exhibited maxima in all cases; thus for sulphonated neatsfoot oil this occurred at  $p_H$  10, for sulphonated cod oil over the range 1—5, and for moellon at  $p_H$  6—8. Less oil was absorbed from a sulphonated neatsfoot oil-egg yolk fat-liquor than from a sulphonated neatsfoot oil fat-liquor at  $p_H$  1—4.5 and  $>6.75$ , but more over the range  $p_H$  4.5—6.75 (cf. Merrill, B., 1928, 580). The contraction of the skin by fat-liquoring was rapid during the first hour and attained equilibrium in 90 min.

D. WOODROFFE.

**Acidity of leather.** H. G. BENNETT (J. Soc. Leather Trades' Chem., 1931, 15, 31—38).—A modification



of Thompson and Atkin's method of determining acidity (B., 1929, 865) is proposed. The fat-extracted leather is exposed to the air for 48 hrs. and its moisture content ( $W\%$ ) determined. Two portions of leather ( $101/W$  g. and  $10.1/W$  g.) are left in contact with  $0.1N$ -potassium chloride for 24 hrs., and, after filtration the  $p_H$  values of the filtrates,  $p_{H_2}$  and  $p_{H_3}$ , respectively, are determined by means of the comparator. The  $p_H$  (or "acid figure") of the original leather is then expressed by  $3p_{H_2}-2p_{H_3}$  and the equation of the graph showing the relation between the  $p_H$  of the leather and dilution ( $D$ ) is:  $p_H = (p_{H_3}-p_{H_2})D + (3p_{H_2}-2p_{H_3})$ . The average  $p_H$  for leathers was 3.5. In one case it was below 2.5, the minimum suggested by Atkin and Thompson. High  $p_H$  values were usually associated with a steep slope to the curve, and low values with leathers of low ash value. The  $p_H$  appeared to be buffered by the presence of mineral matter in the leather.

D. WOODROFFE.

**Density data on leather.** I. D. CLARKE (Ind. Eng. Chem., 1931, 23, 62—67).—The actual density was determined as follows: the weighed leather was put into a known volume of kerosene in a graduated tube or pycnometer and suction was applied intermittently for 2—3 hrs. until all the air had been removed. The increase in the volume occupied by the leather and kerosene was the actual volume of the leather. The apparent volume was determined by removing the leather from the tube, wiping off surface kerosene, and noting the increase in volume caused by returning the leather to the tube. The percentage of voids was obtained by expressing the difference between the apparent and actual volumes as a percentage of the apparent volume. Possible sources of error are indicated. Temperature control is necessary. Actual densities of different leathers are nearly constant at 1.327—1.433. Determinations of the apparent densities and percentage voids of leathers yielded the following results: vegetable-tanned sole leather 0.95—1.05, 25—30%; vegetable-tanned upper and dressing leathers 0.80—0.90, 30—40%; unwaxed chrome-tanned leather 0.60—0.70, 50—60%. The percentage of voids was diminished by filling the leather with grease, tannin, etc. or by mechanical means, e.g., rolling. It was affected by the nature of both the skin and the tannage. Fewer voids are found in vegetable than in chrome tannages.

D. WOODROFFE.

Furs.—See VI. Tannery effluents.—See XXIII.

## PATENTS.

Dryer.—See I.

## XVI.—AGRICULTURE.

**Presence of uronic acids in soils.** E. C. SHOREY and J. B. MARTIN (J. Amer. Chem. Soc., 1930, 52, 4907—4915).—Application of the modification of Lefèvre and Tollens' method for determining uronic acids described by Dickson and co-workers (A., 1930, 453) to 11 soils indicates the presence of 0.308—7.938% of uronic acids; the soils are first heated with 1% hydrochloric acid to decompose inorganic carbonates (the possibility of some of the carbon dioxide evolved being formed from uronic acids is noted as being probable). The presence

of uronic acids (or polyuronides) in two of the soils is confirmed by extraction with 2% sodium hydroxide solution; the extracts are acidified with acetic acid, then neutralised with barium carbonate, evaporated to syrups, and these added to 95% alcohol, whereby substances similar to uronic acid complexes are obtained.

H. BURTON.

**Soil and eucalyptus trees in the Forests of La Plata.** C. A. GRAU (Anal. oficin. quim. prov. Buenos Aires, 1928, 2, 23—31).—Soil analyses are recorded and means of improving plantations described.

CHEMICAL ABSTRACTS.

**Calcium relationships of forage crops.** L. I. PUGSLEY and R. R. MCKIBBIN (Canad. J. Res., 1931, 4, 39—51).—Comparison is made of the composition of red clover and timothy hays and the soils in which they were grown. On clay-loam soils the calcium content of the hays varied directly with the total calcium content of the soils and inversely with their lime requirements. There was no proportionate increase in protein or phosphorus content with increasing calcium content of the hays. In the timothy hays examined the ratio  $CaO:P_2O_5$  was abnormally low and protein:CaO abnormally high, the calcium content being low. A. G. POLLARD.

**Sodium in plants.** P. DE SORNAY (Bull. Assoc. Chim. Sucr., 1930, 47, 370—377).—Evidence that sodium salts are of some physiological importance is adduced in respect of various plants grown in Mauritius. In the leaves of the sugar cane, soda amounts to only a few % of the amount of potash, but it increases appreciably, at least for a time, when ripening commences (July, August), at which stage the amount of potash begins to diminish considerably. In the canes themselves these changes are not so marked, but there is often a temporary rise in soda content about July or August. If acid phosphate fertilisers are withheld the assimilation of sodium salts is greatly inhibited, whilst abnormally large amounts of chlorides, iron, and potash accumulate in the leaves and large amounts of chlorides and sulphates in the stems. The coconut palm contains very large proportions of sodium chloride even at long distances from the sea. The relative proportions of soda to potash in the ash of a large number of plants are tabulated; their wide variations indicate highly selective assimilation of these bases by different plants.

J. H. LANE.

**Effect of various potash fertilisers on the firmness and keeping quality of fruits.** J. H. WEINBERGER (Proc. Amer. Soc. Hort. Sci., 1929, 26, 174—179).—In Maryland potash fertilisation does not affect the keeping quality.

CHEMICAL ABSTRACTS.

**Relation of nitrogen to the keeping quality of fruit.** J. H. GOURLLEY and E. F. HOPKINS (Proc. Amer. Soc. Hort. Sci., 1929, 26, 167—171).—Nitrates produce larger and less highly coloured fruit, with greater water, nitrogen, and catalase content. The nitrogen content of the soil does not affect the keeping quality.

CHEMICAL ABSTRACTS.

**Recent experiments on the preparation of organic manure: a review.** G. J. FOWLER (Agric. J. India, 1930, 25, 363—385).—Numerous experiments

have been carried out at various centres in India to determine the suitability of such materials as weeds, banana stems, sunn-hemp, other leguminous plants, and town refuse for the preparation of organic manures, and to determine how the time of fermentation may be reduced. Fermentation is facilitated by passing the materials through a chaff cutter and by the preliminary preparation of a quantity of vigorously fermenting material to act as an "activator" to the main mass. When carried out in pits the process is completely anaerobic and insect larvæ are practically eliminated. The necessary fermentation organisms may be obtained from cow-dung, cattle urine, or night-soil, the last-named producing the greatest effect. Rege's observations that fungi play an important part in the fermentation process are confirmed, and it is shown that when the ratio of pentosans to lignin exceeds 1 the material is easily decomposed, whereas if the ratio is below 0.5 the material is very resistant to microbial attack. Evidence as to the direct fixation of nitrogen during these processes has been obtained, and the resulting manures have given results as good as, or better than, those due to farmyard manure when tested in the field on rice, maize, wheat, onions, and garlic. E. HOLMES.

**Ethylene oxide for the eradication of pests.** R. B. HARVEY (Science, 1931, 73, 100—101).—Ethylene oxide alone or in aqueous solution kills barberry, currant, gooseberry, poison wig, prickly ash, scrub oak, popple, box-elder, etc. when injected into the soil at the roots. Admixture with chlorates or formaldehyde does not destroy the toxicity. The ethylene oxide penetrates quickly through the tissues and discolours the leaves in a few days. L. S. THEOBALD.

**Sugar beet.**—See XVII.

#### PATENTS.

**Manufacture of fertilisers stable for storage and capable of being scattered.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,303, 30.10.29).—An ammonium sulphate-nitrate mixture (1 : 2.2—4, preferably 1 : 3 mols.) prepared by crystallisation, *e.g.*, as described in B.P. 304,872 (B., 1929, 282), is mixed with material capable of preventing the mixture from caking and hardening, *e.g.*, ammonium sulphate, kieselsguhr, calcium sulphate or phosphate, kaolin, potassium salts, etc. L. A. COLES.

**Apparatus for drying crops artificially.** B. J. OWEN, R. B. STEWART, and W. H. CASHMORE (B.P. 344,080, 6.12.29).

**Fertilisers.**—See II. **Lumber substitute.**—See IX.

### XVII.—SUGARS; STARCHES; GUMS.

**Sugar-beet problems.** I. Storage of beet. II. Methods of analysis. III. Rate of production of sugar during the growing period. G. R. CLARKE, L. F. NEWMAN, and A. W. LING (J. Min. Agric., 1930, 36, 950—960, 1061—1068, 1159—1166).—I. Results during the 1927—8 campaign at Bristol, Cambridge, and Oxford indicate that under adverse climatic conditions beets stored either in covered clamps (4-ft. triangular section) or under sheds lose considerable moisture and solid matter. In some cases about 10% of the sugar was lost

in 2—3 months. Crowned beets may undergo serious deterioration as the result of fungoid attack at the cut surface, but it appears probable that greater losses may be produced by secondary growth in imperfectly cut or in uncrowned beets. Results at the same centres during the 1928—9 campaign, under much more normal weather conditions, showed relatively very small losses. In most cases the loss of weight of the beets in 2—3 months did not exceed 5%, and the percentage contents of dry matter and sucrose remained fairly stationary or increased.

II. In the sampling of beets, not less than 50 roots should be taken for preparing a pulp sample. Pulps obtained by means of the circular saw, the Pellet conical rasp, and the Sans Pareille press gave polarisation values in close agreement by the Sachs-Le Docte method. For the determination of sugar in pulp samples, Krüger's method of cold aqueous digestions was found to give less concordant results than those from the Sachs-Le Docte method or Pellet's method of hot aqueous digestion. The last is applicable to coarse as well as to fine pulp, but for routine purposes, given sufficiently fine pulp, the Sachs-Le Docte method is equally accurate and much more rapid, shaking for 30 sec. being sufficient for complete extraction of the sugar from fine pulp obtained by saw or rasp.

III. From periodic analyses of growing beets in several centres in England, during three seasons, it is concluded that, in general, the crop is at its best, as regards weight and sugar content of the roots, at the end of October.

J. II. LANE.

**Oxford process [of beet-sugar manufacture].** O. SPENGLER (Z. Ver. deut. Zucker-Ind., 1930, 80, 362—373).—A report on an investigation into the Oxford desiccation process at Eynsham factory by representatives of the German Sugar Institute and other German sugar technologists. A close control of the drying process over a period of 21 hrs. furnished the following data: 1893 dz. (about 189 tons) of fresh beets having polarisation 16.2 and invert sugar 0.17% (they had been in store from Nov. to Jan.) yielded 478 dz. of dried cossettes of pale ivory colour, having polarisation 62.73%, moisture 7.8%, and invert sugar 0.803%. The apparent loss of sucrose was 0.425% of the beets or 2.61% of the total sugar. The time required for drying was 55—60 min. and practically no invert sugar was formed. The coal consumption was 11.0% of the weight of fresh beets, the calorific value of the coal being 5417 cal. (9750 B.Th.U.). For German conditions the cost of coal and power for the drying process is estimated at about RM. 30 per 100 dz. of fresh beets (roughly 3s. per ton), which is about equal to the corresponding items in the production of white sugar from fresh beets by the ordinary process. The production of sugar from the dried beets at Eynsham was considered very unsatisfactory. The main plant was not in operation, but the process was studied in a small experimental plant at the factory. Juice of 40—50° Brix, having a pale wine-yellow colour, was obtained, but the method of purification employed (liming to pH 7.4 and separating the precipitate in a centrifugal separator) raised the purity only by 0.3% and rendered the juice almost black. Even after filtration with decolorising carbon

the juice was still very dark and yielded an ash-grey sugar which could not be affined to a white sugar of more than medium quality. It is concluded that the drying of the cossettes at Eynsham is successfully accomplished without appreciable inversion or discoloration, but it is too costly to enable this method of sugar manufacture to compete with the diffusion method; moreover, the method employed for producing sugar from the dried cossettes does not yield a first-class product. A conspicuous advantage of the process is the complete absence of pulp from the diffusion battery waste waters.

J. H. LANE.

**Aqueous digestion and diffusion [of sugar beets].** D. SIDERSKY (Bull. Assoc. Chim. Sucr., 1930, 47, 362—366).—The high polarisation values obtained by Bothy (B., 1930, 879) by successive hot aqueous extractions of beet cossettes, compared with those found by the usual methods of analysis, are attributed to the formation of soluble dextrorotatory non-sugars from hemicelluloses, owing to the high temperatures and relatively large volumes of water employed for the extractions.

J. H. LANE.

**Full utilisation of the sulphurous acid in sulphitation of thin [sugar] juice.** III. E. THIELEPAPE and A. FULDE (Z. Ver. deut. Zucker-Ind., 1930, 80, 344—361; cf. B., 1929, 448).—In laboratory experiments, factory juices after the second carbonatation and filtration, having alkalinities of 0.022—0.013% CaO, were sulphited to alkalinities of 0.014—0.008% CaO and boiled for 5 min. None required filtration. After cooling they still contained about 90% of the added sulphur dioxide in active form, to check discoloration of the juice during evaporation. Weisberg's process, in which the juice is sulphited before the second carbonatation, was found to be wasteful, inasmuch as 13—85% of the added sulphurous acid appeared to be lost in the carbonatation filter cakes. The authors' process of sulphitation, as indicated above, was employed in two factories throughout most of the 1929—30 campaign. Although the beets were of much better quality than those of the preceding year, it was found desirable to add about 0.03—0.05% (calc. on beets) of sodium carbonate to the juice to eliminate calcium salts as far as possible and ensure a sufficient alkalinity in the final juice. Sulphitation did not lighten the colour of the thin juice, but it greatly lessened the development of colour during evaporation. The juices were lightly sulphited also at the syrup stage.

J. H. LANE.

**Applications of electrometric methods to the control of the purification of diffusion juice.** G. MICHELASSI (Bull. Assoc. Chim. Sucr., 1930, 47, 322—326).—A review of known methods.

J. H. LANE.

**Possible recovery of ammonia in beet-sugar manufacture.** E. DEPASSE (Bull. Assoc. Chim. Sucr., 1930, 47, 295—300).—Ammonia evolved in the evaporation of beet-sugar juices (cf. Zamaron, B., 1930, 1084) might be recovered by means of a gas-washing apparatus (described) interposed in the vapour outlet from each unit of the multiple effect. The apparatus is patented and is used for the recovery of acetic acid in the evaporation of tannin extracts. It might be adapted also to

the washing of carbonatation gas and to the carbonatation process itself.

J. H. LANE.

**Naudet's "plus-sugar."** M. BECHARD and L. FOUQUEREAUX DE FROBERVILLE (Bull. Assoc. Chim. Sucr., 1930, 47, 327—328).—The juice and bagasse fractions obtained from pulped sugar-cane samples by pressure, when re-united and analysed, indicated 0.22—0.41% more sugar in the cane than when the fractions were analysed separately. The original pulped samples gave intermediate values. The effect of addition of antiseptics to the reconstituted samples was studied. The results seem to point to synthesis of sucrose in the reconstituted samples. No particulars of analytical methods used are given.

J. H. LANE.

**Industrial crystallisation of sugar.** J. DUBOURG (Bull. Assoc. Chim. Sucr., 1930, 47, 306—313).—The main results of investigations by Fouquet (B., 1910, 290) and Kukharenko (A., 1927, 341, 820; B., 1927, 567, 730, 952; 1928, 540) are described, with graphs, and made the basis of a discussion of the various stages in the technical crystallisation of sugar.

J. H. LANE.

**Experiment on the purification [of sugars] in presence of sea-water.** COLIN and MIOSEC (Bull. Assoc. Chim. Sucr., 1930, 47, 303—305).—The purification of sugar juices containing magnesium salts by treatment with baryta or lime followed by carbonatation may leave some soluble barium or calcium salts in solution, e.g., chlorides formed by precipitation of magnesia from magnesium chloride. This was found to be the case in the isolation of a new galactose compound from the juice of the seaweed *Rhodymenia palmata*, and confirmed by experiments on sea-water. Sugar beets contain 0.02—0.04% MgO, which would be largely precipitated from its salts on liming the juice, leaving an equivalent amount of calcium salts in the juice after carbonatation unless the natural alkalinity were sufficiently high or reinforced by addition of sodium carbonate (cf. Spengler and Brendel, B., 1928, 421). The natural alkalinity of beet juice has no meaning apart from the particular defecant employed; it has different values according as lime or baryta is used, owing to the different solubilities of the salts of these bases, especially the sulphates and oxalates.

J. H. LANE.

**Tables showing starch content of starch milk [in relation to density].** SPROCKHOFF (Chem.-Ztg., 1930, 54, 299).—Two tables are given, one referring to the volume, the other to the weight of starch milk. The starch content is expressed in terms of dry starch and of starches containing 20%, and 50% of moisture.

W. J. BOYD.

**Formation of plus-sugar in the Naudet diffusion process and variations of "pre-sugar" or ratio of invert sugar to sucrose during vegetation.** F. GUILBERT (Bull. Assoc. Chim. Sucr., 1930, 47, 301—303).

Molasses.—See XVIII.

## PATENTS.

**Production of (A, B) degradation products [sugars], (c) polyoses or monoses, from cellulose-like substances and cellulose-containing materials.** COMMERCIAL ALCOHOL CO., LTD., J. S. ARTHUR, and R.

GOZARTEN (B.P. 336,934—5 and 337,017, [A—C] 20.4.29).—(A) In the production of sugar from cellulosic materials by means of hydrochloric acid in two stages, viz., decomposition of the cellulose, followed by saccharification (cf. B.P. 292,918—9; B., 1930, 77), improvements claimed consist in starting with material dried to a moisture content of 10% or less, treating this in a rotating autoclave with a mixture of steam and hydrochloric acid gas until it contains about 25—35% of moisture, then saturating at a low temperature with strongly cooled hydrochloric acid introduced in gaseous or liquefied form under atmospheric or higher pressures, to decompose the cellulose, after which saccharification is rapidly effected by heating preferably to 70—75°, either indirectly or by admission of steam or mixtures of steam with hydrochloric acid. The saccharified material is a loose mass from which most of the acid may be removed by vacuum distillation or by a current of inert gas. All operations may be carried out in the same vessel, and a battery of such vessels enables the acid evolved during the saccharification or subsequent de-acidification of one charge to be used, directly or after condensation and cooling, for the treatment of another charge. To convert the sugar formed into fermentable monoses, the saccharified material may be treated with steam or steam and acid, or may be lixiviated and the extract boiled with acid. (B) Cellulosic materials containing not more than 50% of moisture, and preferably 20—40%, or drier materials brought to this moisture content by steaming, are saturated at a low temperature with strongly cooled hydrochloric acid and afterwards saccharified without previous removal of the acid, substantially as described in (A). (C) Materials such as artificial silk waste or sawdust, containing not more than 10% of moisture, are steamed to a moisture content of 20—40% in a jacketed autoclave, with simultaneous cooling and, if necessary, under reduced pressure. Decomposition of the cellulose is effected at a low temperature by admission of hydrochloric acid gas, preferably under pressure, and saccharification follows, without previous removal of acid, by treatment with steam or a mixture of steam and inert gas. The product may be substantially de-acidified and dried under reduced pressure and lixiviated, and the extracted sugar converted into fermentable monoses by boiling or steaming the solution, with additional acid if necessary.

J. H. LANE.

**Obtaining sugar from cellulose or cellulose-containing substances.** COMMERCIAL ALCOHOL CO., LTD. (B.P. 341,501, 18.9.29. Ger., 2.10.28).—In a process of the type described in B.P. 336,934 etc. (cf. preceding abstract) for decomposing the cellulose contained in sawdust etc., the subsequent saccharification is effected rapidly by indirect heating of the charge to temperatures above 50°, preferably about 70°, and the containing vessel is preferably kept closed during the first part of the saccharification period, to prevent evolution of hydrochloric acid and consequent reversion of decomposed, but not yet saccharified, cellulose to an insoluble form.

J. H. LANE.

## XVIII.—FERMENTATION INDUSTRIES.

**Fermentation of molasses produced from refined-sugar factories.** N. TAKETOMI and T. MATSUMOTO

(J. Soc. Chem. Ind., Japan, 1930, 33, 531 B).—Ammonium salts greatly increase the velocity of fermentation: a suitable proportion is 0.1—0.4 g. per 100 c.c. of the diluted molasses. Magnesium and calcium salts and potassium diphosphate have no appreciable influence. Refined sugar molasses contains about 4.5—6% of glucose, which is non-fermentable. W. J. WRIGHT.

**Manufacture of absolute alcohol.** II. GUINOT (Chim. et Ind., 1931, 25, 26—32).—The manufacture of absolute alcohol by azeotropic distillation methods is described and reviewed. Emphasis is laid on its application for motor spirit, including "carburant national" (50% alcohol), and its advantages for this purpose are detailed. Recent methods permit the production of pure absolute alcohol from grape must, using the old type of apparatus employed for rectified spirit, at a cheaper cost than was previously possible for the higher grades of crude spirit. L. J. HOOLEY.

**New Wagner refractive index table for ethyl alcohol-water mixtures at 17.5°.** K. SENNEWALD (Z. Unters. Lebensm., 1930, 60, 409—419).—The author disagrees with Beckel's revision (B., 1930, 78) of Wagner's tables, especially in its application to brandy distillates. Alternative tables and graphs show (1) the relation between the (Zeiss immersion) refractive index and percentage of ethyl alcohol by weight and volume at 17.5° for alcohol containing 0—100% of water; (2) the temperature corrections for the above table at degree intervals between 10° and 30° for mixtures containing 0—55 vol.-% of alcohol. At 17.5°  $n$  increases almost linearly with increase in alcohol content to a maximum (1.035) for 80.28 wt.-%, and then falls. Examples of the calculation are included. J. GRANT.

**Wine distillates and wine brandies.** H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1930, 60, 530—532).—Mainly polemical in reply to Büttner and Miermeister (B., 1930, 527, 963). Examples are given in support of the authors' contention (cf. Zellner, B., 1930, 963) that, in addition to analyses, taste should be considered in the accurate evaluation of wine distillates and brandies. J. GRANT.

**Origin, occurrence, and detection of  $\beta$ -butylene glycol in wine and fruit wine.** J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1930, 60, 484—488).—Wine (25 c.c.) is evaporated to 5 c.c. and 4 g. of sodium carbonate are added, followed by 0.5 c.c. of bromine water (at 0°). The mixture is neutralised with hydrochloric acid and the resulting acetylmethylcarbinol detected by distillation with 50 c.c. of 10% ferric chloride solution in a Reichert-Meissl apparatus, the distillate (20 c.c.) being collected in a mixture of 2 c.c. of 20% hydroxylamine hydrochloride, 3 c.c. of 20% sodium acetate, and 1 c.c. of 10% nickel chloride solutions. A ring of dichroic crystals of nickel dimethylglyoxime is formed at the junction of the two layers, and the weight of the crystals  $\times 0.624$  gives the amount of  $\beta$ -butylene glycol. About 0.6% was found in wine and fruit wine, but none in bottom-fermentation beer. The German official method for the determination of glycerol requires no correction for the presence of  $\beta$ -butylene glycol. The test is a specific distinction between fermented and unfermented liquors, e.g., between true sweet wine and wine prepared from unfermented must. J. GRANT.

**Detection of sorbitol in wine.** W. DIEMAIR and G. LIX (Z. Unters. Lebensm., 1930, 60, 304—310).—Pure monobenzylidenesorbitol has m.p. 169—170° and the pure dibenzylidene compound m.p. 182—184°. The divergent values given in the literature for the m.p. and composition of dibenzylidenesorbitol are due to contamination with the mono-derivative. Identification of sorbitol by means of the m.p. of its mono- and di-benzylidene derivatives is difficult. Such crystalline derivatives can under certain conditions be obtained from cider. W. J. BOYD.

**Detection of isopropyl alcohol in brandies, spirits, tinctures, cosmetics, and embrocations by means of piperonal.** G. REIF (Z. Unters. Lebensm., 1930, 60, 243—254).—The author's colorimetric methods for detection of isopropyl alcohol in brandies, cosmetics, etc. (B., 1928, 686; 1929, 737) have been modified to a single method suitable for all the above liquids, full details for which are given. W. J. BOYD.

**Sulphite-pulp liquors.**—See V. Bread.—See XIX.

### XIX.—FOODS.

**Bread prepared with beer yeast.** M. L. KOSCHKIN (Z. Unters. Lebensm., 1930, 60, 489—495).—Beer yeast, washed with a large excess of water to remove hop resins and other sources of unpleasant taste, may be used in the preparation of bread. The resulting loaves have slightly lower porosities and higher acidities and nitrogen and water contents than the usual loaf. The dough was prepared from a mixture of wheat with 15—20% of barley flour and contained 10—50% of washed pressed yeast (containing 73% of water and 58% of protein). The unpleasant taste of the yeast was perceptible in loaves from dough containing over 40%. Tests on dogs fed on the bread showed that 88% of the nitrogen is digested, this corresponding with a loaf having a total protein content of 46.4 g. more than in the case of the control loaf. J. GRANT.

**Phosphatide contents of [German] bread.** B. REWALD (Z. Unters. Lebensm., 1930, 60, 315—318).—With one exception, the varieties examined contained 0.174—0.302% of phosphatide (calc. on the dry substance). Crust and crumb had almost equal contents. The temperature of baking and the action of the yeast had no destructive effect on the phosphatides. W. J. BOYD.

**Detection of pasteurised milk.** K. EBLE and H. PFEIFFER (Z. Unters. Lebensm., 1930, 60, 311—314).—A modified Wilkinson-Peters benzidine reaction is carried out by treating 5 c.c. of the milk with 0.5 c.c. of a 4% solution of benzidine in 96% alcohol and, after 1 min., with 1 drop of 1% hydrogen peroxide solution. After shaking, a transient blue-green coloration is obtained in the case of pasteurised milk. With unheated milk the colour develops more slowly, but becomes more intense and less evanescent than with pasteurised milk. Milk heated to a higher temperature remains colourless. After 1 min. 10 c.c. of saturated magnesium sulphate solution are added with shaking. Unheated milk shows an immediate grey-green coloration (blue-green after 10 min.); pasteurised milk is coloured grey-green to

grey-blue. After a further 10 min. the curd separates (dark blue, unheated milk; greyish, pasteurised milk). Strongly heated milk gives a white curd. The reactions are intensified if 10 c.c. of saturated magnesium (or sodium) sulphate solution and 1 drop of hydrogen peroxide solution are added to 5 c.c. of milk, followed by 0.5 c.c. of benzidine solution after 1 min. These colour reactions become more distinctive in the case of pasteurised milk 10—20 hrs. after pasteurisation. The benzidine reaction of pasteurised milk becomes more intense with increasing acidity and returns to its normal intensity on neutralisation. Milk heated for a short time at not above 63° gives a reaction equal to that of normal milk. If the benzidine reaction is uncertain, as in the case of mixtures of unheated and strongly heated milk, 10 c.c. of the sample are treated with 10 c.c. of saturated magnesium sulphate solution and 1 c.c. of 20% acetic acid, with vigorous shaking after each addition, and then centrifuged for 6 min. at 1200 r.p.m. The serum of unheated milk, or of mixtures of unheated with 20% or less of strongly heated milk, is opalescent, whereas sera of pasteurised and strongly heated samples are water-clear. W. J. BOYD.

**Determination of milk proteins. II. Identity of the casein precipitate.** G. M. MOIR (Analyst, 1931, 56, 73—78; cf. B., 1931, 316).—It is shown that the casein precipitated from cows' milk at  $p_H$  4.6 by acetic acid buffered with sodium acetate is chemically identical with that precipitated by acetic acid alone at  $p_H$  4.2, although the amount of material precipitated at the higher  $p_H$  is 1—2% greater than that precipitated at the lower. A. R. POWELL.

**Imitation Liptauer cheese.** W. KOENIG and H. KLUGE (Z. Unters. Lebensm., 1930, 60, 523—526).—Analytical results are tabulated for 8 varieties of Liptauer and similar cheese. Fat (on dry solids) 50.2—77.7%; milk fat in total fat, calculated from the expression (xylene number—1.1)/0.2 (cf. Van Raalte, B., 1927, 153, 569), 2—5%, but in one case 34%;  $n_D^{20}$  (of fat) 40.1—48.0; Reichert-Meissl value 5.4—9.0; Polenske value 2.0—10.0; saponif. value 213—240; solids 41.2—63.3%. All contained paprika and pepper, and 0.5% of potato starch was present in one sample. A red coal-tar dye was found in all but 2 samples. J. GRANT.

**Determination of cholesterol and lecithin and its use in food chemistry, particularly for the evaluation of egg products.** J. TILLMANS, H. RIFFART, and A. KÜHN (Z. Unters. Lebensm., 1930, 60, 361—389).—A solution of 4 mg. of cholesterol in acetone is evaporated with a 2% alcoholic solution of digitonin, and the resulting precipitate filtered on a steam-heated funnel and washed with chloroform, ether, acetone, and finally with water. It is then oxidised by the action for 1 hr. of a 1% solution of potassium dichromate in sulphuric acid, and the iodine liberated after dilution and addition of potassium iodide is titrated with sodium thiosulphate solution. The factors 8.7 (cholesterol) and 7.9 (phytosterol) divided into the thiosulphate equivalent of the chromic acid used give the sterol contents in mg. Egg or pastry products are first dried on sand, and an ethereal extract is used (1) directly, and (2) after

saponification, the values in the latter case being raised by 8–10 mg. For the determination of lecithin an extract of the sample in hot alcohol is evaporated, the residue oxidised with warm sulphuric acid and hydrogen peroxide, and lecithin phosphate precipitated in the cool, neutralised solution with 5 c.c. of 1.5% strychnine nitrate solution and 15 c.c. of an 8% solution of ammonium molybdate in 400 c.c. of 50% nitric acid. The precipitate is filtered from an ice-cold solution, washed with ice-water, and the weight (dried at 100°) divided by 39 gives  $P_2O_5$  with an accuracy of 0.02 mg. Other methods are discussed critically and the results obtained tabulated for 21 egg and pastry products. The egg content may be calculated from the data given, e.g., 58.7 mg. of cholesterol per 100 g. of pastry corresponds with 1 egg per 500 g. of flour. Storage for 1 year produces no decrease in cholesterol or lecithin content, except in home-made products, where this is attributed to exposure to light and to the water content. J. GRANT.

**Spinach saponin.** O. DAFERT [with D. LOY and J. FLESCHNER] (Z. Unters. Lebensm., 1930, 60, 408–409).—The saponin is contained chiefly in the root (haemolytic index 1:1000 for sun-dried and 1:400 for steam oven-dried roots), but is absent from the stalk and seed, and the index of the root rises to a maximum (1:800) after 7 weeks and then decreases. It is extracted in 70% alcohol and the filtered extract concentrated and cooled in ice. The saponin may then be dried, extracted with ethyl acetate, and recrystallised from hot alcohol as pointed white needles ( $C_{42}H_{78}O_{24}$ , index 1:100,000, m.p. 260–262°), soluble in alcohol, alkali, and hot water, but not in organic solvents. No second saponin was obtained, and hydrolysis with alcoholic acid gave a white amorphous sapogenin ( $C_{24}H_{44}O_7$ , m.p. 208–212°), insoluble in water or ether. J. GRANT.

**Physiological action of maté [Paraguay] tea and the possibility of correcting its taste.** A. BICKEL and A. M. LOEW (Z. Unters. Lebensm., 1930, 60, 526–530).—"Mattos" tea, prepared by extraction of dried maté with an aqueous suspension of an ethereal oil, followed by slow re-drying at a low temperature, removes the unpleasant taste and odour, and gives a product comparable in flavour with China tea and having the same caffeine content (0.81%) and a slightly higher extract (18.97% on the dry solids from a 10% aqueous extract) than the original. Gastric juice secretion experiments on dogs (after Pavlov) and kymographic tests gave approximately the same results for both maté and mattos teas. J. GRANT.

**Detection of foreign seed kernels in chocolate and [similar confectionery] pastes.** V. MOUCKA [with R. MÜLLER] (Z. Unters. Lebensm., 1930, 60, 395–402).—The defatted sample is shaken with 4% alcoholic mercuric chloride solution and centrifuged, the sugars are extracted by similar treatment with cold water, and the residue is dried by means of alcohol-ether and mounted in a cold saturated solution of sucrose containing 0.5 g. of iodine and 2 g. of potassium iodide per 100 c.c. The starch then appears dark blue with a light border, the aleurone grains are yellow, whilst globoid, crystalloid, and oxalate inclusions are readily visible in the yellow plasma. The constituents are

identified by the size and nature of the aleurone grains. Finely-ground coconut also shows characteristic long endosperm parenchymatous cells after treatment with chloral hydrate. Photomicrographs are shown in each case for the individual kernels and mixtures (5%) with a paste containing cacao beans, sugar, and cacao butter maintained at 80° for 30 hrs. J. GRANT.

**Ultra-violet absorption of honeys.** J. STITZ and J. KOCZKÁS (Z. Unters. Lebensm., 1930, 60, 420–425).—The Bunsen-Roscoe absorption extinction coefficient ( $e$ ) was determined for  $\lambda$  5500–2350 Å. by means of a sector photometer and quartz spectrograph for layers 1–4 mm. thick between a tungsten-iron arc and a 0.02 mm. slit. Characteristic absorption curves (which are shown) were obtained for 6 varieties of genuine honeys, and show sharp maxima at 2700 Å. ( $e = 0.13$ – $0.145$  for 1 cm. of 4.5–6.5% solution). Addition of water lowers  $e$  markedly; dextrose, laevulose, and sucrose producing similar but relatively small effects, whilst dextrin produces a rise. The total absorption of a honey is not given by the sum of the absorptions of the above constituents unless allowance is made for ash, colouring matter, proteins, etc. (e.g.,  $e$  is higher for commercial than for pure dextrose). J. GRANT.

**Colorimetry in the examination of foodstuffs. I. Colour components and colour changes of paprika powder.** O. VARGA (Z. Unters. Lebensm., 1930, 60, 268–277).—The colorimetric methods of Ostwald have been applied to the colour analysis of paprika powder. This contains only a little white and consists chiefly of black and "full colour." The latter varies from yellow to orange (Ostwald colour degrees 4–6), being redder in the better qualities of powder, in which also the proportion of "full colour" to black is higher. This proportion is also increased by finer milling and decreased by increased oil content. Hungarian and Spanish products are bright fiery-red; Greek are dull brown. In storage paprika powder becomes pale and discoloured. W. J. BOYD.

**Orange juice and orange pulp.** A. HANAK (Z. Unters. Lebensm., 1930, 60, 297–301).—The composition of commercial products used in the preparation of orangeade are discussed and analytical data are given. W. J. BOYD.

**Sterilisation of conserves.** F. KONRICH (Z. Unters. Lebensm., 1930, 60, 233–243).—It is suggested that attention should be paid in canneries to the effect of the size of the pieces of flesh or other material in the packages on the time required for attainment of the requisite temperature for sterilisation. There is a critical temperature (110°) above which the time required for complete sterilisation decreases rapidly. A short treatment at a higher temperature is usually less injurious to flavour than a prolonged treatment at a lower temperature. Complete sterilisation so far as is consistent with quality should be aimed at. The air cock of the steriliser should be at the bottom of the apparatus and the steam inlet at the top. The former should be fully open until 100° is reached and then left only slightly open for 10 min. so that the heavier air is displaced completely. W. J. BOYD.



**Isolation of quinic acid from fruits.** E. F. KOHMAN and N. H. SANDBORN (Ind. Eng. Chem., 1931, 23, 126).—Quinic acid is present to about 1%, on the fresh fruit basis, in prunes and cranberries, and has been isolated by taking advantage of the solubility of its lead and calcium salts compared with that of other acids. During digestion it is converted into hippuric acid and tends to create an acid condition in the system. Grapes appear to contain traces of the acid, but it has not yet been isolated from this fruit. T. McLACHLAN.

**Determination of formic acid in fruit juice.** A. HANAK and K. KÜRSCHNER (Z. Unters. Lebensm., 1930, 60, 278—290).—To 10 c.c. of the juice 0.3 g. of tartaric acid is added and the solution is steam-distilled for 45 min., increase in volume and over-heating of the liquor being avoided. 400 C.c. of distillate are collected, diluted to 500 c.c., and mixed. Then 200 c.c. of this solution are used for titration of the total free volatile acids. A further 100 c.c. or 200 c.c., according to the formic acid content expected, are heated under reflux with *N*-caustic soda, 0.5—1 c.c. being added in excess of the amount required to neutralise the free acid as found by titration, and the solution is warmed for 10 min. to hydrolyse any formic ester present. The solution is then gently evaporated to 30 c.c. after addition of 0.2 g. of sodium carbonate. Enough 0.2*N*-potassium permanganate (usually 3—5 c.c.) is added so that 0.5—1.5 c.c. remain in excess, and, after 45—60 min., 1 c.c. of 10% zinc sulphate solution to promote flocculation of the manganese peroxide. The clear liquid is poured off, the precipitate washed by decantation, and the solution and washings are made up to 50 c.c., filtered by suction, and the filtrate is compared colorimetrically with a standard permanganate solution. The influence of the presence in the juice of other volatile substances which react with permanganate, *e.g.*, sulphurous and salicylic acids, is discussed. W. J. BOYD.

**Antiscorbutic vitamin content of some preserved foods.** G. MACLEOD and L. BOOKER (J. Home Econ., 1930, 22, 588—593).—The antiscorbutic value of grape fruit is: fresh 227, canned 206—162 units per lb. The value for canned pimento is 227 units per lb.

## CHEMICAL ABSTRACTS.

**Effect of storage and canning on vitamin-A, -B, and -C contents of carrots.** D. DOUGLASS and J. E. RICHARDSON (J. Home Econ., 1930, 22, 859—860).—Storage at 40° preserves vitamin-C in carrots, whilst oven-canning destroys it. The vitamin-B, but not -A, content is diminished by oven-canning; storage at 58° or ordinary cooking does not affect vitamins-A and -B.

## CHEMICAL ABSTRACTS.

**Vitamin-B, -G, and -D contents of Arizona dates.** M. C. SMITH (J. Home Econ., 1930, 22, 857).—Maktum dates (227 units per lb.) are richer in vitamin-B than Deglet Noor or Thoory dates. Vitamins-G and -D are present.

## CHEMICAL ABSTRACTS.

**Vitamin-A, -B, -C, and -G contents of watermelon (*Citrullus vulgaris*).** H. E. MUNSELL (J. Home Econ., 1930, 22, 680—685).—The vitamin-A content is 1 unit per g. and the vitamin-C content about 0.33 of that of tomatoes. Vitamins-B and -G are present in small quantities.

## CHEMICAL ABSTRACTS.

**Formic acid.**—See III. Nitrite-sulphite mixtures.—See VII. Edible fats.—See XII.

## PATENTS.

**Preparation of a solid form of lactic acid useful in baking powders and self-rising flours.** E. V. MCCOLLUM and O. S. RASK (U.S.P. 1,771,342, 22.7.30. Appl., 7.1.29).—A mixture of gelatinised starch paste with sufficient lactic acid to yield a product containing at least 30% (preferably 46—48%) of the acid is dried in thin layers by heating at 125—137° *in vacuo*; the product, after grinding, is mixed with sodium bicarbonate and, if desired, starch, flour, etc. L. A. COLES.

**Manufacture of milk products.** A. AXELROD (B.P. 342,083, 16.12.29).—Full-cream or separated milk is pasteurised and treated with peptonising bacteria producing little or no lactic acid. The precipitated constituents are obtained in a finely-divided form by the use of peptonising agents such as an alkali phosphate or citrate. The whole is dried and as such may be used in baking processes. E. B. HUGHES.

**Treatment or preparation of cheese.** APLIN & BARRETT & THE WESTERN COUNTIES CREAMERIES, LTD., and A. E. OTTIKER (B.P. 342,098, 3.1.30).—Cheese is ground and fed into a high-speed disintegrator where the material is caused to travel in a very thin film between surfaces moving at a very high velocity relatively to one another. The process is carried out at not above 38°. It is claimed that such cheese may be pasteurised without causing the fat to separate. E. B. HUGHES.

**Egg products.** A. K. EPSTEIN and B. R. HARRIS (B.P. 340,820, 25.1.30).—Dried egg-whites having better foaming properties are obtained if, before drying, the liquid egg-white is first ripened, preferably after inoculation with acid-producing bacteria, so that the product is acid to phenolphthalein and still alkaline to litmus and no putrefaction has commenced. Fermentable carbohydrates may be added to hasten the process. E. B. HUGHES.

**Processing of fruit to prevent decay.** H. R. BATES, Assr. to CITRUS COMPOUND CORP. (U.S.P. 1,774,310, 26.8.30. Appl., 19.4.29).—To inhibit mould, citrus fruit is wetted with a solution of di- or tri-sodium phosphate, wiped, and dried. E. B. HUGHES.

**Preparation of chocolate and cocoa products containing vitamins.** COMMANDITAIRE VENNOOTSCHAP OP AANDEELEN ONDER DE FIRMA C. J. VAN HOUTEN & ZOON (B.P. 340,580, 23.9.29. Holl., 24.11.28).—Ergosterol, dissolved in cacao butter or a solvent, is irradiated with ultra-violet light, the solvent being removed in an inert atmosphere under reduced pressure, after addition of cacao butter in which the vitamin is stable. The vitaminised fat is used in preparation of chocolate, or mixed cold with cocoa powder of low fat content. E. B. HUGHES.

**Formation of an iodised marine developing medium for production of oysters or other edible marine shellfish rich in iodine.** P. M. R. SALLES and M. R. LOUBATIE (B.P. 341,576, 28.10.29).—Shell-fish are allowed to rot in a tank of sea-water for 5 or 6 days. The shells are removed and the liquor is left for a further



period during which algæ develop at the expense of the organic matter present. It is then circulated, aerated, and iodised, and finally becomes clear and odourless. The live shell-fish are then placed on racks in the tank and owing to the maintained iodine content of the water they absorb iodine directly and indirectly from the crustaceans on which they feed. It is claimed that in 5 days 200 times the normal quantity of iodine can be fixed.

C. JEPSON.

**Cereal food product.** KELLOGG Co., Assees. of E. H. McKAY (B.P. 344,055, 2.12.29. U.S., 5.12.28).

**Roaster.**—See I. Impregnating composition.—See XIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Hydroxylamine method for the determination of ketones.** Carvone in caraway and dill oils. C. T. BENNETT and T. T. COCKING (Analyst, 1931, 56, 79—82).—Two 1.5-g. samples of the oil are placed in each of two stoppered tubes and to each tube are added 10 c.c. of hydroxylamine hydrochloride reagent; the tubes are heated at 75° and their contents titrated with *N*-alcoholic potash until a golden-yellow colour is obtained. An excess of 0.5 c.c. of potash is added to one tube and if the colour becomes a paler yellow the solution in the other tube is titrated further until the two colours match. This figure is then multiplied by 1.008 as a correction factor and the result calculated to carvone. The reagent is prepared by dissolving 6.95 g. of hydroxylamine hydrochloride in 95 c.c. of 90% alcohol, adding 0.4 c.c. of a 0.2% solution of dimethyl-yellow, and neutralising to full yellow with *N*-alcoholic potash.

A. R. POWELL.

**Rate of oxidation of Carstanjen's compound on the addition of an alkali.** A. A. HARWOOD (J. Amer. Pharm. Assoc., 1931, 20, 17—20).—The absorption of oxygen in presence of alkali by Carstanjen's compound under varying conditions has been examined. Under favourable conditions absorption is very rapid, and probably 1 mol. of the compound is oxidised to the quinone by 1 atom of oxygen, alkali sulphite and water being eliminated.

E. H. SHARPLES.

**Detection of artificial balsams in Peru balsam.** J. KOK (Pharm. Weekblad, 1931, 68, 32—33).—The nitric acid reaction with the material soluble in light petroleum should be carried out by allowing one drop of acid to fall on the residue.

S. I. LEVY.

**Decomposition of cocaine solutions by sterilisation or keeping.** S. A. SCHOU and E. HELM (Danske Tids. Farm., 1931, 5, 1—11).—Determinations of cocaine by addition of sodium carbonate and extraction with a mixture of isopropyl alcohol (1 vol.) and chloroform (3 vols.) and of benzoic acid after complete hydrolysis with sodium hydroxide, acidification, and extraction with chloroform enable the amount of hydrolysis which has occurred in solutions of cocaine hydrochloride to be ascertained. There is considerable hydrolysis in buffered solutions of  $p_H$  above 3—4, but pure aqueous cocaine hydrochloride solutions may be sterilised by a current of steam for 30 min., or in presence of 0.001*N*-

hydrochloric acid by heating at 120° for 20 min., without appreciable decomposition.

H. E. F. NOTTON.

**Nicotine contents of Dutch cigars.** A. VAN DRUTEN (Z. Unters. Lebensm., 1930, 60, 501—518).—For the determination of nicotine in tobacco the method of Bodnár, Straub, and Nagy (B., 1928, 546) gives high results and is not recommended, the silicotungstic acid method (Rasmussen) is reliable if suitable precautions are taken and 10 g. of tobacco are available, whilst that of Pfyl and Schmitt (B., 1927, 955), modified as follows, is preferred. The tobacco (3.5 g.) is steam-distilled with 25 g. of sodium chloride and 1 g. of magnesium oxide, the distillate neutralised to methyl-red (which is removed by a drop of bromine water), and nicotine dipicrate precipitated in the cold in the presence of 0.35% of picric acid. The filtered and washed precipitate is titrated with 0.1*N*-sodium hydroxide to phenolphthalein, in the presence of 25 c.c. of toluene, whence the factor 0.243 gives the percentage of nicotine. The nicotine contents of 104 "normal" Dutch cigars varied from 0.5 to 2.7% (mean 1.42%) on the dry tobacco, the amounts being independent of weight and price. Cigars (6) sold as containing "minimum" and "absolutely harmless" amounts of nicotine contained 0.57—1.53%. It is considered that these and "denicotinised" cigars should contain less than 0.5%, and "nicotine-free" cigars only traces.

J. GRANT.

**Importance of the acidity of tobacco in its hygienic evaluation.** A. FAITELOWITZ (Z. Unters. Lebensm., 1930, 60, 518—523).—The degree of mildness of a tobacco is given by the ratio acidity of tobacco : basicity of unneutralised smoke, the former term being determined by titration of a 10% extract with 0.1*N*-sodium hydroxide, and the latter from the volume of 0.1*N*-sulphuric acid neutralised by the smoke from 10 cigarettes. These figures are tabulated, with the nicotine contents of the tobacco and of the neutralised smoke, the free bases in the latter, and the taste of the smoke, for 6 tobaccos in their natural state and after making alkaline. The process of smoking liberates amino-bases which are responsible for irritation of the throat, but the effect of which varies according to the degree of neutralisation by the acidity of the tobacco as the smoke passes through the cigarette. From one half to one third of the nicotine content is found in the neutralised smoke, this proportion being greater for alkaline tobaccos.

J. GRANT.

**Leaf oils of Washington conifers.** V. *Picea sitchensis*. A. J. LEHMAN and E. V. LYNN (J. Amer. Pharm. Assoc., 1931, 20, 29—32; cf. B., 1931, 274).—Immediate steam-distillation of the fresh leaves and twigs of *P. sitchensis* (tideland spruce) gave 0.059% of a light yellow oil having  $d_{27}^{20}$  0.8806,  $[\alpha]_D^{25}$  —5.05°,  $n_D^{20}$  1.4684, acid value 2.04, saponif. value 35.81 (11.81% of bornyl acetate) and containing  $\beta$ -pinene,  $\beta$ -phellandrene (total terpenes more than 40%), camphor, a ketone (semicarbazone, m.p. 231—232°), borneol, terpineol, a sesquiterpene named *picene* ( $d_{25}^{20}$  0.8997,  $n_D^{20}$  1.4990,  $[\alpha]^{25}$  —80.33° in 1.4% alcoholic solution; hydrochloride, m.p. 133°), a lactone (m.p. 112°; from the saponification products), and nonoic and butyric acids.

E. H. SHARPLES.

**Lavender.** A. CHRIS (Parfums de France, 1930, 8, 227—240; Chem. Zentr., 1930, ii, 3469).—At 200 m. the oil content falls rapidly from the commencement of blossoming; at 500—800 m. the maximum yield of oil coincides with full bloom. Variations in the composition are recorded. Oil from buds had  $d_{15}^{20}$  0.8750,  $\alpha_D +15^\circ 20'$ , ester content 23.03, free alcohols 21.92, oxygenated constituents 44.95%. Constants of steam-distillation fractions are recorded. A. A. ELDRIDGE.

**Jack pine.**—See XIII. Tinctures etc.—See XVIII.

#### PATENT.

**Manufacture of the hormone of the anterior lobe of the hypophysis.** SCHERING-KAHLBAUM A.-G. (B.P. 341,038, 4.10.29. Ger., 6.10.28. Addn. to B.P. 313,924; B., 1930, 1131).—The aqueous solution of the precipitated crude hormone obtained from urine is treated with such salts of multivalent metals, particularly of the alkaline earths, the anions of which form alcohol-soluble sodium salts. The precipitated phosphates and sulphates are removed and the hormone is precipitated from the residual liquid with organic solvents. E. H. SHARPLES.

### XXL—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Some properties of chrome alum [photographic] stop baths and fixing baths.** J. I. CRABTREE and H. D. RUSSELL (Brit. J. Phot., 1930, 77, 356—357, 373—375, 388—391, 401—402; cf. Crabtree and Hartt, B., 1930, 686).—Separate chrome alum baths are recommended in preference to chrome alum fixing baths. The maximum degree of hardening is obtained with chrome alum stop baths adjusted to  $p_H$  4.0 for neutral film or to  $p_H$  3.0—3.8 for alkaline film, sulphuric acid being the most suitable acid for maintaining this acidity. At these values the hardening properties are constant with chrome alum concentrations above 0.5%, but in practice a convenient working life and uniform hardening properties are obtained with a 3% bath. Unused baths keep indefinitely, but the addition of developer lowers the hardening properties and adversely affects the keeping properties. Practical exhaustion tests showed that the addition of sulphuric acid at regular intervals increases the hardening properties and prolongs the life of the bath. More uniform hardening properties were obtained at  $30^\circ$  than at  $21^\circ$ . Practical recommendations are given for the preparation of the baths, the time of immersion of the film, the prevention of blisters and stain, etc. J. W. GLASSETT.

**Solarisation. IV. Dependence of solarisation on the method of preparation of an emulsion.** H. ARENS (Z. wiss. Phot., 1931, 28, 341—348; cf. A., 1930, 1534; B., 1930, 929).—An emulsion prepared by adding potassium bromide solution to excess (10%) silver nitrate solution, washing the precipitate by decantation, and immediate peptisation in gelatin solution and ripening shows no solarisation. If, before peptisation, the precipitate is left in the reaction solution (24 hrs.), or, after washing, in water (1 hr.), or freed from excess of silver ions by sodium chloride, ammonia,

etc. (and rewashed), the resulting emulsion will show solarisation. Hence the presence of silver ions retards the acquirement of solarisation properties. Further experiments show that the effect is irreversible, i.e., silver ions cannot remove solarisation properties which the precipitate has once been allowed to acquire. Effects produced by precipitation by adding the silver nitrate to the potassium bromide solution, with or without stirring, are hence explained. J. LEWKOWITSCH.

#### PATENTS.

**Photographic films or plates and methods of forming images thereon.** KODAK, LTD., Assecs. of J. G. CAPSTAFF (B.P. 341,822, 14.5.30. U.S., 17.5.29).—A dye which strongly absorbs the rays to which the emulsion is sensitive is incorporated in the emulsion. By exposing through the support, the outer layers of the emulsion remain unaffected and act as a protective layer to the developed silver image. J. W. GLASSETT.

**Production of sensitised photographic films and copying or duplicating materials.** L. RADO (B.P. 341,003, 7.10.29. Ger., 17.10.28).—The light-sensitive material is incorporated in thin layers of cellulose hydrate, which are firmly cemented to one or both sides of a metal foil, e.g., aluminium. The surface of the cellulose hydrate, after development of the image, is given a protective coating of nitrocellulose, celluloid, or shellac. J. W. GLASSETT.

**Manufacture of kinematograph or like films for colour photography.** I. G. FARBENIND. A.-G. (B.P. 341,948, 23.10.29. Ger., 23.10.28).—A metal or glass roller closely wound with thin wire (40—60  $\mu$  in diam.) is used to impress the casting surface of the film-casting machine so that linear, semi-cylindrical light-refracting elements are directly applied to the surface of the film. Alternatively, the finished film is rendered plastic and impressed with a roller having a negative profile of the original roller. J. W. GLASSETT.

**Anti-halation layers for photographic plates and films.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 342,531, 5.2.30).—Anti-halation layers with increased solubility in alkalis are prepared from dyes of the fuchsone series containing one or more carboxyl groups in the molecule, e.g., aurine-mono- or -di-carboxylic acid. J. W. GLASSETT.

**Light filters.** E. S. CLUDERAY, and RADIOVISOR PARENT, LTD. (B.P. 342,433, 28.11.29).—An infra-red filter is prepared from a sheet (0.018 in. thick) of ordinary ebonite enclosed between two sheets of heat-resisting glass of high infra-red transmission. J. W. GLASSETT.

**Fixing of pictures made on light-sensitive layers containing bleaching-out dyes.** I. G. FARBENIND. A.-G. (B.P. 341,371, 12.5.30. Ger., 11.5.29).—After exposure, the thiourea derivative acting as sensitiser is destroyed by sulphur dioxide, and the decomposition products are removed by wiping the print with fabric moistened with benzene. J. W. GLASSETT.

**Photographic printing processes.** F. J. SHEPHERD, F. F. RENWICK, and ILFORD, LTD. (B.P. 341,924, 23.10.29

and 22.5.30).—A prehardened silver halide-gelatin emulsion layer, with m.p. preferably above 70°, is developed in a non-tanning developer, fixed, washed, and, prior to bleaching, transferred to a buffered stop-bath adjusted to  $pH$  5.0 approx., in order to render the action of the bleaching bath more uniform. After bleaching, fixing, washing, and drying, the ink is applied evenly by means of a roller to the surface of the print, and gradation is developed by rolling the surface with a wet felt or rubber-covered roller. J. W. GLASSETT.

**Photographic reversal processes.** KODAK, LTD., and E. E. JELLEY (B.P. 341,183, 13.11.29).—After passing through the reversal bath, the film is treated with a fogging agent which is only sufficiently concentrated to form development centres on the remaining silver halide grains, and the final positive image is then developed with any suitable developer; alternatively, the fogging and redevelopment treatments may be combined. J. W. GLASSETT.

**Treatment of exposed photographs or the like photosensitive material with gaseous media.** P. HÖRNICKE (B.P. 341,972, 26.10.29).—The exposed material is placed in an evacuated chamber into which the gas is admitted under regulated pressure. Suitable apparatus is described. J. W. GLASSETT.

**Reproducing the artificial grain of a smooth board by photography.** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 341,228, 9.12.29. Ger., 12.12.28).—The contrasts in the grain are increased by treating the surface of the wood, before copying, with water, highly refractive fats or oils, or high-boiling hydrocarbons. J. W. GLASSETT.

## XXII.—EXPLOSIVES; MATCHES.

**Comparison of nitrocellulose and nitroglycerin powders.** ANON (Mém. Poudres, 1931, 24, 1—9).—Samples of BM<sub>17</sub>(B) powder and three nitroglycerin powders after being stored at 60° for nearly 3 years were tested in the close vessel and in a gun at irregular intervals. After 1 year the values for  $(dP/dt)_m$  and  $P_m$  in the close-vessel test showed a general decline, but they are without importance since they do not take into account the "vivacity" of the powders. In the firing trials the values obtained for velocity and pressure were in agreement, and also showed a gradual fall. BM<sub>17</sub>(B) powder during the first six months showed an increase in  $V_0$  and  $P_m$  owing to loss of solvent, but after two years these values approached the original ones, whilst the close-vessel test results indicated a drop. The variations in the content of diphenylamine, chloroform-ether extract, and nitrogen in the nitrocellulose and solvent extract for the respective powders at different periods of hot storage are tabulated. W. J. WRIGHT.

**Cause of ignitions during the pressing of SD powder.** P. FLEURY (Mém. Poudres, 1931, 24, 49—59).—Ignitions during pressing are not due to electrical phenomena, but are caused by the sudden transformation of energy into heat, the amount of heat developed being equal to the work done by the ram on the paste. In an experimental pressing, records were taken of the

travel of the ram, the pressure, the velocity of the ram, and the power (pressure  $\times$  ram velocity), curves being plotted for the values obtained. The power reached a maximum of 146 kg./m. in the first  $\frac{1}{2}$  min. of the ram movement; it then fell to a very low figure, the pressure at this stage being insufficient to extrude the paste, and subsequently gradually increased to about 70 kg./m. for the remaining 15 min. of the operation. The result confirms the experience that ignitions occur before full compression is reached. A constant rate of travel of the ram should therefore be avoided, the interval corresponding to the "danger point" of the curve being sufficiently prolonged. W. J. WRIGHT.

**Testing of lead azide detonators.** J. A. CRESSWICK and S. W. E. PARSONS (J. Proc. Roy. Soc. N.S. Wales, 1929, 63, 85—88).—The use of the sand test of Storm and Cope in the comparison of detonators of different types, fulminate and tetryl-lead azide, leads to inaccurate results, although it serves for the comparison of detonators of a single type. They are best compared by a ballistic method, using a nitroglycerin explosive desensitised with liquid paraffin. This method of desensitisation is preferable to that of picric acid by olive oil, since the additions of diluent are larger and the mixtures more stable. T. H. MORTON.

**Crusher gauge [for explosives].** E. BURLOT (Mém. Poudres, 1931, 24, 60—71).—A description is given of prescribed modifications in the apparatus used by the Service des Poudres. The close vessels are of 25 (instead of 22 $\frac{1}{2}$ ) and 150 c.c. capacity, and plugs and obturation are standardised. By the use of steel with a sufficiently high limit of elasticity, it is possible to employ pressures up to the extreme values given on the "tarage" table. Means are provided for sampling the combustion gases. Details are also given of the recording device. W. J. WRIGHT.

**Nitrated diethylene glycol.**—See III.

## PATENTS.

**Explosive.** R. G. WULFF (U.S.P. 1,772,695, 12.8.30. Appl., 7.3.29).—An explosive capable of losing its power soon after being loaded into a borehole comprises an oxidising agent (chlorate, perchlorate, nitrate, or peroxide) and a liquefied volatile hydrocarbon, such as butane. An absorbent material (silica gel or kieselguhr) may be added. W. J. WRIGHT.

**Nitrocellulose solutions.**—See XIII.

## XXIII.—SANITATION; WATER PURIFICATION.

**Thermophilic digestion of sewage solids.** IV. **Fresh solids and activated sludge.** W. RUDOLFS and H. HEUKELEKIAN (Ind. Eng. Chem., 1931, 23, 67—69).—Small-scale experiments indicate that the time of digestion of both fresh and activated sludges may be reduced to 2.1 days, and possibly less, at 50° (as compared with the previously accepted minimum of 30 days at 28°), and the final sludge has all the characteristics of a ripe, well-digested product. It is recommended that preheated sludge should be added continuously throughout the 24 hrs., and the simultaneously

withdrawn sludge fed to mechanical filters. The increased rate of addition of undigested sludge necessitates the use of a stirring device to bring the fresh and ripe sludges into more intimate contact, to prevent the fresh sludge from rising to the top of the tank, and to aid the steady evolution of the gaseous products. The yield of gas from activated sludge is about 50% of that from fresh solids, calculated on the basis of the volatile matter present. C. JEPSON.

**Effect of temperature on rate of settling of sewage solids.** G. M. RIDENOUR (Sewage Works J., 1930, 2, 245—250).—The rate is maximal at 30°; the effect of temperature is most pronounced in settling periods up to 30 min. The percentage removal of solids in 2 hrs. is the same for all temperatures between 1° and 37°. The settling of fine particles is unaffected. Less flocculation occurs at higher temperatures.

CHEMICAL ABSTRACTS.

**Chloroform [U.S.P.] as sewage preservative increases "oxygen consumed" values.** R. T. HOMEWOOD and H. W. RUF (Sewage Works J., 1930, 2, 251—254).—The increase in permanganate reduction value can be avoided by using chloroform washed with water.

CHEMICAL ABSTRACTS.

**Limitation of phenol coefficient of coal-tar disinfectants.** C. M. BREWER and G. L. A. RUEHLE (Ind. Eng. Chem., 1931, 23, 150—152).—Contrary to previous reports, there is no constant ratio for the phenol coefficient between *B. typhosus* and *S. aureus*. It may vary from 2 to 20 with disinfectants forming cloudy solutions with water and from 1.3 to 2.4 for those forming clear solutions. It is therefore impossible to calculate the *S. aureus* coefficient from the *B. typhosus* figure in the case of coal-tar disinfectants, and it is probable that this is true of other organisms.

T. McLACHLAN.

**Testing of Admiralty disinfectant fluid.** T. C. PATTERSON and R. C. FREDERICK (Analyst, 1931, 56, 93—104).—The method consists in preparing a 1% dilution of the fluid with sterile artificial sea-water and, after 24 hrs., determining its germicidal action in the presence of organic matter, against a particular strain of *B. typhosus*, as compared with phenol. As the accuracy of the results depends to a large extent on the exact following of the rather lengthy method described, the original must be consulted for full details of the procedure.

A. R. POWELL.

**Hydrogen sulphide poisoning.** L. B. ALLYN (Ind. Eng. Chem., 1931, 23, 234).—The death of two people by hydrogen sulphide is recorded: both bodies were deeply cyanosed. A dilution of 1 in 400 of the gas killed an albino rat in 40 sec.

T. McLACHLAN.

**Technique of stream-pollution investigations.** F. W. MOELMAN, T. L. HERRICK, and H. G. SWOPE (Ind. Eng. Chem., 1931, 23, 209—213).—In sampling the flow of a large river it is advisable to ascertain the correct location of each sampling point by means of a cross-sectional survey which includes determinations of current velocity and depth, the dissolved oxygen content and biochemical oxygen demand of the water, and the condition of the stream bed. The variations in the oxygen

content and demand due to pollution and possibly also to the effect of daylight on green and blue-green algae indicate whether a single daily sample will suffice or whether one compounded of a number of samples taken at shorter intervals is required. Sludge deposits lower the dissolved oxygen content of the adjacent water, especially when they are disturbed, e.g., by fermentation gas released by a reduction in atmospheric pressure. The authors determine the dissolved oxygen by the Winkler method, using the permanganate-oxalate modification only when the nitrite content exceeds 0.1 p.p.m. In the Sanitary District of Chicago variations in tap water derived from deep wells have led to the regular use of a synthetic diluent containing 300 p.p.m. of sodium carbonate.

C. JEPSON.

**Some interrelationships of plankton and bacteria in natural purification of polluted water.** C. T. BUTTERFIELD and W. C. PURDY (Ind. Eng. Chem., 1931, 23, 213—218).—Experiments with *B. aerogenes* and the plankton *Colpidium* in pure culture indicate that the oxygen demand of living organisms is small unless they are in a state of active increase. Bacteria, by absorbing dilute food and thus concentrating it in their bodies, become in themselves sufficient to stimulate plankton growth. The function of the plankton appears to be to maintain the bacterial population below its limiting value so that compensatory bacterial multiplication is stimulated and the continuation of oxidation phenomena is maintained, as the presence in the medium of a definite number of bacterial cells, living or dead, prevents further multiplication. These conclusions are supported by the observed results of a reduction of the bacterial population by artificial means, e.g., pasteurisation, chlorination, or filtration through a Berkefeld filter.

C. JEPSON.

**Effect of sunlight and green organisms on re-aeration of streams.** W. RUDOLFS and H. HEUKELEKIAN (Ind. Eng. Chem., 1931, 23, 75—78).—In running streams the dissolved oxygen content increases during daylight to a maximum during the afternoon and decreases during darkness to a minimum just before sunrise. Similar variations were noted in a tidal section of the R. Delaware, but were somewhat affected by the change in tides. Laboratory experiments with river water containing green algae showed that the dissolved oxygen content depends on the number of organisms present and the relative times of exposure to darkness and light; direct sunlight and temperature were not found to be important factors. In stream-pollution surveys, samples should be taken over a 24-hr. period, as otherwise the dissolved oxygen found will be too high or too low according to the time the sample was taken.

C. JEPSON.

**Precautions needed in the ammonia-chlorine treatment of swimming pools.** L. L. JENNE and H. R. WELSFORD (Ind. Eng. Chem., 1931, 23, 32—34).—By means of this combined treatment a higher residuum of chlorine may be maintained without producing any offensive taste, odour, or irritation. Care is needed in making the o-tolidine test, as high results may be recorded owing to the presence of nitrites in amounts

up to 1.0 p.p.m. and thus create a false security. In carrying out the test the sample should be finally dechlorinated with thiosulphate and the loss in colour noted as equivalent to the chlorine content. The excessive nitrites in the swimming-pool water may be removed by cutting off the supply of ammonia and increasing the chlorine dosage. C. JEPSON.

**Efficiency of [water] chlorination at Chicago.** H. H. GERSTEIN (J. Amer. Water Works' Assoc., 1931, 23, 53—62).—In spite of its widely varying characteristics, Chicago's supplies of raw water from L. Michigan have been satisfactorily purified by chlorination alone. The efficiency of the process is dependent on the strict supervision maintained, as there appears to be a definite relationship between the bacterial qualities of the raw and chlorinated waters, which is influenced by the extent of the turbidity present. Owing, however, to the increasing pollution of the lake, it is considered that chlorination alone will not produce a safe water in the near future unless excessive amounts of chlorine be used. C. JEPSON.

**Prechlorination in relation to the efficiency of water-filtration processes.** H. W. STREETER and C. T. WRIGHT (J. Amer. Water Works' Assoc., 1931, 23, 22—52).—Parallel tests carried out in an experimental rapid sand-filtration plant showed that prechlorination of raw water, when properly controlled, is a useful means of reinforcing the bacterial efficiency of plants of this kind. A low residuum of chlorine (0.05—0.1 p.p.m.) in the applied water proved more satisfactory than superchlorination. On the other hand, the bacterial efficiencies of the filtration and postchlorination processes were measurably reduced and the length of filter runs was not appreciably increased although the bacterial content and the biochemical oxygen demand of the filtering medium were appreciably reduced. C. JEPSON.

**Residual alum in North Carolina filtered waters.** J. W. KELLOGG (J. Amer. Water Works' Assoc., 1931, 23, 92—102).—The presence of alum in filtered water, though probably harmless to health, is undesirable for certain industrial purposes, and may be due either to faulty coagulation or desolution of the floc during secondary lime treatment for corrosion prevention. Examination of effluents from plants in North Carolina showed that whereas in some cases the alum content never exceeded 0.05 p.p.m., there were certain coloured waters which failed to produce a colourless effluent without excess of alum in solution. Details of the "jar test" for determining the optimum dosage and the hæmatoxylin method of determining alum in solution (B., 1924, 397) are quoted. C. JEPSON.

**Determination of nitrate in drinking water.** K. SCHERINGA (Pharm. Weekblad, 1930, 67, 1362—1363).—Sodium salicylate is added to the water and the residue after evaporation treated with sulphuric acid; after addition of alkali, the nitrate content is determined colorimetrically. A correction factor is used if much chloride is present. S. I. LEVY.

**Recent developments in boiler water treatment.**

A. W. CHAPMAN (Fuel, 1931, 10, 64—69).—The problem of scale formation and its prevention, particularly by treatment of the water with sodium phosphate, is discussed (cf. Hall and others, B., 1928, 70; 1929, 342, 962; Partridge and White, B., 1929, 874; Partridge, B., 1930, 1133; Coates, J.S.C.I., 1930, 206 T).

A. B. MANNING.

**Tannery effluent. I. Effect of various gases on the nitrogen distribution.** E. R. THEIS and P. KRATZ (Ind. Eng. Chem., 1931, 23, 69—71).—When tannery soak effluent is treated with sodium nitrate (1000 p.p.m.) nitrogen is evolved under acid conditions, but gases are absorbed when the reaction is alkaline. When a stream of oxygen is passed through the soak liquor ammonia is produced, indicating the deamination of amino-acids, the effluent becomes colloidal in character, and there is very little formation of volatile sulphides. In presence of hydrogen the production of hydrogen sulphide is encouraged, deamination ensues with subsequent reduction, and volatile acids are produced. C. JEPSON.

PATENTS.

**Destruction of algae and fungi in water or sewage.** G. ORNSTEIN (B.P. 340,218, 20.9.29).—For this purpose the simultaneous addition of chlorine water and a solution of copper salt is much more effective than either used alone, even when each is added in much greater quantities. A form of apparatus is claimed in which, by means of an arrangement of siphons, proportional doses of the two solutions may be added at prearranged intervals. [Stat. ref.]

C. JEPSON.

**Sterilisation of water by means of chlorine.** R. ADLER (B.P. 340,202, 22.6.29).—The gradual deactivation of carbon used for the removal of excess of chlorine from water is caused, in part, by the loading of the carbon surface with foreign substances carried by the water and, in part, by the products of a by-reaction between chlorine and the carbon, consisting mainly of carbon dioxide, but to a smaller extent of lower oxidised hydrogen- and oxygen-containing carbon compounds (termed "mellogens"). The mellogens may be removed and the carbon reactivated by treatment with an alkaline solvent, e.g., sodium carbonate, together with a strongly active oxidising agent, e.g., chlorine water, followed by subsequent removal of all alkali from the carbon, either by washing, or by precipitation with, e.g., calcium chloride. C. JEPSON.

**Removing [dissolved] gases from boiler-feed water and the like liquids.** SOC. DES CONDENSEURS DELAS (B.P. 341,829, 19.5.30. Fr., 25.5.29).—The gases are released from the heated liquid by reducing the adjacent atmospheric pressure to a value below the vapour pressure of the water etc. at that temperature. In the device claimed, the heating chamber and the pressure-reducing chamber are connected by means of a siphon through which the water flows and which compensates by means of a liquid column for the difference in pressure between the two chambers.

C. JEPSON.

**Zeolites. Oxygen for respiration.**—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAY 8 and 15, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Measurement of viscosity.** P. WOOG (Bull. Inst. Pin, 1931, 23—24, 41—47).—Details are given of the methods available for the measurement of viscosity in arbitrary and in absolute units. The units of absolute and kinematic viscosity ("poise" and "stokes," respectively) are defined and their wider usage is recommended. S. S. WOOLF.

**[Hydrostatic] pressure of foam as a measurable quantity.** K. SCHIEBL (Chem.-Ztg., 1931, 55, 169—170).—The saturation of limed beet syrup with carbon dioxide produces foaming, the height of the foam being proportional to the alkalinity of the liquor. If this process is carried on with batch-working it can be controlled by the observation of hydrostatic pressure at the bottom of the vessel, and at a point above the original surface of the liquid. Time-pressure curves show the gradual rise of the foam and its partial settlement up to completion of the reaction. The method can also be adapted to continuous working, and it is thought that it may be applicable to other industrial processes. C. IRWIN.

**Application of legal requirements in the building of steam boilers to the chemical industry.** SCHEFFEL (Chem. Fabr., 1931, 109—111).—Anomalies in German law relative to steam boilers and other pressure vessels such as autoclaves are criticised. A uniform code of specifications for all vessels required to withstand pressure and temperature is advocated. Points discussed include the use of cast steel, of stainless steel and non-ferrous metals, the strength of welded joints, expansion allowances, and safety factors. C. IRWIN.

**Necessity for the chemical supervision of boiler feed water.** H. PROSKE (Chem.-Ztg., 1931, 55, 226—227).—An example is quoted showing the necessity for careful adjustment of the lime and soda used in the Reisert-Derveaux method of softening boiler feed water. In the case cited too large an excess of soda was used and insufficient time was allowed for the precipitate to settle; as a result undue foaming occurred in the boiler and a deposit of soluble salts formed in the superheater tubes which finally caused the complete stoppage and bursting of a tube. The deposit contained 67%  $\text{Na}_2\text{SO}_4$ , 29%  $\text{Na}_2\text{CO}_3$ , 1%  $\text{NaCl}$ , and 1.2%  $\text{Fe}_2(\text{SO}_4)_3$  and, except for a small amount of silica and calcium sulphate, was completely soluble in water. The same water, when properly softened, behaved quite satisfactorily in a similar type of boiler. A. R. POWELL.

**Method of representing the analytical results obtained in the examination of boiler feed water.** J. LEICK (Z. angew. Chem., 1931, 44, 100—102).—It

is recommended that the results should be expressed in millival (1 val  $\equiv$  equivalent weight in g./litre); as most of the titrations are made with 0.1N-reagents the number of c.c. required in the titration of 100 c.c. of water gives directly the amount of the substance present in millival. By multiplying the results by the g.-equiv. of the substance the amount of the substance present in mg./litre is obtained. A. R. POWELL.

**Nomograms in chemical technology.** A. SULFRIAN (Chem.-Ztg., 1931, 55, 209—211).—The applications of nomograms in industrial chemistry are reviewed. A nomogram for use in the control of boiler and domestic water, which facilitates conversions from one to another of the various scales of hardness, calculations from analytical data, and the evaluation of the quantities of reagents required for conditioning any type of water, is reproduced and explained in detail. Various methods of expressing the composition of a specimen of water are discussed, and it is recommended that all quantities be expressed in milliequiv. per litre. H. F. GILLBE.

**Nomography.** O. LIESCHE (Chem. Fabr., 1931, 4, 122).—The nomogram described shows the weight to which a solution must be evaporated, or a solid dried, in order to increase the percentage content of non-volatile matter to a predetermined value.

H. F. GILLBE.

**Horizontal tank chart.** W. F. SCHAPHORST (Ind. Eng. Chem., 1931, 23, 314—315).—A nomogram is given for determining the volume (gals.) of liquid at any depth in tanks of 2—200 in. in diam. and up to 100,000 in. long. W. J. WRIGHT.

**Inexpensive crystal or molasses separator.** R. H. KING (Ind. Eng. Chem., 1931, 23, 300).—The device comprises a container constructed from  $\frac{3}{4}$ -in. pipe, having a perforated plug and sieves for drainage at the lower end, and a plug with a central hole at the upper end through which air at any desired pressure is admitted. The efficiency of crystallisation of the liquid is determined from the ratio of the concentration of the product in the original magma to that in the mother-liquor after crystallisation. W. J. WRIGHT.

**Control of distillation columns.** M. PÉRARD (Chim. et Ind., 1931, 25, 286—290).—For each column the temperature on a particular plate near the top corresponding to the desired removal of a constituent, e.g., alcohol from a distillery effluent, is determined by experiment, and this temperature is maintained by automatic control of the steam supply. When live and exhaust steam are used they should be mixed before entering the column, the live-steam supply being automatically regulated. A preheating condenser and a water-

\* The remainder of this set of Abstracts will appear in next week's issue.

cooled condenser in series are indicated. The speed of the vapour through the column should not be too great, otherwise the plate efficiency will be reduced and priming will result.

D. K. MOORE.

**Heat transmission to water flowing in pipes. Effect of tube length.** A. E. LAWRENCE and T. K. SHERWOOD (Ind. Eng. Chem., 1931, 23, 301–309).—Experiments were carried out on heating water flowing through a copper tube (inside diam. 0.593 in.) at velocities of 0.62–22.4 ft./sec., the tube being heated by means of a steam-jacket, and its surface temperature determined by means of thermocouples. Data are tabulated for pipes 11.09, 9.03, 6.03, and 2.91 ft. in length. The calculated film coefficients for the water side fall within narrow limits, but are not proportional to the pipe lengths. It is concluded that for turbulent flow the effect of pipe length on the film coefficient of heat transmission is negligible, and this is confirmed by graphical analysis of the data on the overall coefficients of heat flow from steam to water. The film coefficients for condensing steam on the outside of the pipes agreed with the Nusselt equation.

W. J. WRIGHT.

**Flax residues.**—See V.

#### PATENTS.

**Heat-treatment furnaces.** ELECTRIC FURNACE Co., LTD., and D. F. CAMPBELL (B.P. 343,306, 7.2.30).—The furnace is electrically heated and is operated in a horizontal position and then pivoted to a vertical position so that the goods may be discharged directly into a quenching bath without contact with the air; as an additional precaution, goods, such as drills or rods, may be inserted into nickel-chromium tubes.

B. M. VENABLES.

**Furnaces and/or apparatus for the annealing or heat treatment of metal and other goods.** A. SMALLWOOD and J. FALLON (B.P. 343,187, 22.11.29).—In a continuously operating apparatus having a slowly moving conveyor, the latter is provided with upstanding vanes which almost make contact with the walls of zones at each end of the furnace, to which zones inert gas is supplied, forming seals preventing the ingress of air.

B. M. VENABLES.

**Operation of reversible regenerative furnaces.** W. ALBERTS and P. ZIMMERMANN (B.P. 344,289, 9.5.30. Ger., 21.5.29).—A connexion having an adjustable valve is provided between the upper parts of the air and gas uptakes, so that a proportion of the waste gases leaving by the air port may be diverted into the gas regenerator; the connexion is useful when the gas ports are restricted for the use of rich gas.

B. M. VENABLES.

**Rotary kilns.** M. VOGEL-JØRGENSEN (B.P. 343,513, 18.11.29. Denm., 26.10.29).—The low thermal efficiency of a rotary kiln compared with that of a shaft kiln is considered to be largely due to poor heat transmission from the gases and walls to the material. A rotary kiln is therefore provided at the calcining zone, and, if desired, also at the preheating zone, with outwardly extending chambers (bulb-shaped in cross-section) in which the material is lifted and showered across the path of the gases. Alternating with these chambers, in positions such that the showering material does not enter

them, are other openings which serve for the outlet of gases of combustion and are regulable by dampers operated while the kiln is in motion.

B. M. VENABLES.

**Apparatus for recovery of dry-distillation material.** L. HONIGMANN and F. BARTLING (U.S.P. 1,774,554, 2.9.30. Appl., 2.6.26. Ger., 2.6.25).—Dust-like or granular material from dry-distillation processes in which a revolving hearth is used is transported pneumatically to a closed chamber provided with baffles and an outlet pipe. Steam may be injected into the chamber to induce separation of dust without producing condensation of the distillation products. The separated dust is carried by means of a vertical, revolving worm into a cooling chamber communicating with the bottom end of the separating chamber, and may be removed continuously from the lower end of the cooling chamber by a second revolving worm.

H. E. BLAYDEN.

**Furnace-wall structure.** F. J. TONE, Assr. to CARBORUNDUM Co. (U.S.P. 1,775,414, 9.9.30. Appl., 21.1.26).—Water-tubes coated with a silicon carbide refractory are built into the lining of a furnace which is constructed of blocks of that material, these being mechanically interlocked with the tubes; the wall of the furnace is thereby supported throughout its entire area and slipping or movement prevented.

C. B. MARSON.

**Heat-insulating material made of a metal or of textile fibres.** C. KOHLER (B.P. 343,632, 5.12.29. Switz., 12.12.28).—The material is made of textile or metallic tubular fibres which are evacuated without collapsing the walls; the material is also enclosed in an airtight shell.

B. M. VENABLES.

**Transference of heat from solid surfaces to liquids and semi-liquids.** E. METCALFE-SHAW (B.P. 342,830, 3.12.29. U.S., 3.12.28).—The liquid is spread in a thin layer on a heated moving surface and is kept turning over by a number of plough-like devices, preferably formed on the ends of wires having a certain amount of resilience and arranged in such a way that the space cleared by one plough is covered by the tilth from the next.

B. M. VENABLES.

**Processing plant [for canned goods etc.].** CARRIER ENG. Co., LTD., and W. S. YARROW (B.P. 342,722, 4.11.29).—The apparatus comprises a fixed cylindrical chamber within which a drum of smaller diameter rotates slowly. The drum is provided with longitudinal ribs interrupted to allow the intercalation of circumferential ribs on the interior of the fixed casing; in one longitudinal line spaces are left in all the circumferential ribs, and at the ends of this line charging and discharging means for one can at a time are provided. Every cell contains a can and each can goes round a complete circle and is then pushed to the next circle and so on through every circle, the cooking being effected by steam or other hot fluid in the chamber.

B. M. VENABLES.

**Heat-exchange apparatus.** A. D. HARRISON (B.P. 342,959, 10.4.30. U.S., 10.4.29).—An exchanger of the filter-press type is described. Circular plates with uninterrupted packing rings are utilised.

B. M. VENABLES.



**Construction of air preheaters or other heat-exchange apparatus.** R. P. WALLIS (B.P. 343,600, 26.11.29).—The oval tubes of a heat exchanger are arranged in staggered rows in such a manner that the passages for the outer transversely flowing fluid are of constant cross-section, which area may have any desired relation to the cross-section available for the inner fluid. The ratio of the axes of the elliptical tubes is made as large as is possible consistent with preserving enough flow area, so as to reduce the distance of any particle from the tube walls. B. M. VENABLES.

**Method of heat exchange between viscous liquids.** H. BOOTH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 343,231, 10.12.29).—A comparatively non-viscous liquid, *e.g.*, water, under a pressure suitable to the temperature is continuously circulated between two exchangers, flowing inside the tubes at such a speed that the flow is always turbulent. The viscous fluids (*e.g.*, oils for cracking purposes) flow in a zig-zag manner outside the tubes of their respective exchangers. B. M. VENABLES.

**Minimising the formation of scale and similar deposits in steam boilers, evaporators, condensers, boiling pans, coolers, etc.** W. THALHOFER (B.P. 343,025, 8.11.29. Austr., 1.12.28).—The electro-osmotic conditions existing in the scale are changed at arbitrary intervals of not less than one week by the addition of chemical substances to the feed water alternately with additions of other substances or with periodical use of electric currents, with the result that the scale already formed is caused to drop off. B. M. VENABLES.

**Water-cooling towers.** L. G. MOUCHEL & PARTNERS, LTD., and A. T. J. GUERITTE (B.P. 343,288—9 and 343,415, [A, B] 23.1.30, [C] 18.12.29).—The patents refer to cooling towers constructed of brick and/or concrete. In (A) a profile is described, in (B) a method of construction partly in brick and partly in reinforced concrete, in (C) a method of reinforcing the concrete. B. M. VENABLES.

**Drying of wet materials [*e.g.*, wood].** AKTIEBOLAGET FRIBERG'S HÖGVACUUMPUMP (B.P. 343,981, 27.11.29. Swed., 29.11.28).—The material to be dried is heated by direct steam in a closed chamber which is afterwards evacuated to remove air etc. Then steam is readmitted, but the temperature is not allowed to rise above 60°, and during part at least of the second steaming period the pressure must be below 200 mm. Hg. The chamber is then again evacuated until the temperature of the material falls to 35–40°. The second steaming and evacuation process may be repeated as often as is necessary and the use of superheated steam is sometimes desirable. B. M. VENABLES.

**Apparatus for extracting liquid from finely-divided material.** L. ALTPETER, and GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 344,293, 19.5.30).—For the dehydration of finely-divided coal or similar material which retains too much moisture when treated in a centrifuge of the decanting type, thus clogging the meshes of the basket of an extractor of the strainer type, a centrifuge with impervious walls is converted into a strainer by the provision through the walls of devices through which the liquid can leave from points

of large radius, but only in an inwardly inclined direction so that the heavier solid matter does not enter the discharge passages. B. M. VENABLES.

**Dehydrator.** C. DUNNING (U.S.P. 1,775,036, 2.9.30. Appl., 13.12.26).—The apparatus, suitable for delicate liquids, comprises a casing, through which heated or unheated air is blown, containing a sort of water-wheel of considerable axial width, the radial vanes of which are composed of wire-mesh or other perforated material, and each terminates in a lifting trough the liquid from which is spilled over the mesh work after the vane concerned has reached the horizontal. The bottom of the casing is curved to the shape of the wheel over an arc of about 90° and has a subsidiary trough to draw off the liquid when sufficiently concentrated; during most of the period of concentration the supply of liquid is constant to maintain the depth of the bath. B. M. VENABLES.

**Utilising the waste heat of moisture in drying apparatus which is charged and discharged continuously.** W. E. EVANS. From MADRUCK GES. F. MASCHINELLE DRUCKENTWÄSSERUNG M.B.H. (B.P. 343,593, 23.8.29. Cf. B.P. 338,950; B., 1931, 329).—It is intended to dry peat, lignite, etc. in the complete absence of air so that the water vapour produced can be completely condensed at a temperature only slightly below 100°, with consequent improvement in the re-utilisation of the latent heat. The drying zone is operated at a pressure slightly above atmospheric and the charging and discharging apparatus at the ends of the dryer are maintained full of the material from which the air is expelled by steam diffusing outwards. The bulk of the steam is led off by pipes to a heat-exchanging condenser and the resistance of the pipes etc. automatically produces an increase in pressure. B. M. VENABLES.

**Grinding mills.** BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 343,257 and 344,124, 2.1.30. Addns. to B.P. 334,058; B., 1930, 970).—Modifications to the apparatus described in the prior patent are described. Thus, *e.g.*, in (A) the screen within the upper ring of balls is replaced by a conically shaped, imperforate plate, and in (B) the air is led at high velocity inside the lower ring of balls and at a lower velocity outside the upper ring. B. M. VENABLES.

**Mills for grinding paints, enamels, inks, and other viscous substances.** S. SMITH (B.P. 344,316, 24.6.30).—A roller mill in which the doctors are enclosed in a casing is described. B. M. VENABLES.

**Hammers, beaters, or the like for pulverising mills.** GEN. ELECTRIC Co., LTD., and D. H. WAYMARK (B.P. 342,605, 12.9.29).—A form of hammer head which can be adjusted outwards to compensate for wear is described. B. M. VENABLES.

**Pulverisation of solid materials.** M. MADORE (B.P. 343,759, 28.2.30).—The material is held centrifugally in a layer on the interior of a drum rotating in one direction, and crushing is effected by rollers rotating about fixed axes in the other direction. B. M. VENABLES.

**Crushing of ores [etc.].** J. G. CLOKE (B.P. 343,130, 11.11.29).—Fuel and other minerals, cereals, etc. are crushed by a number of mullers arranged in line in a trough or plane surface of which only that portion near the feed end is roughened. The mullers are fixed to a single frame which in turn is connected to the means for reciprocating it by a link from one point only, so that the ends are free to rise. Spaces are provided above the mullers through which water carrying finely-crushed ore can pass from end to end without further crushing, except that the passage is stopped at at least one intermediate point so that all material must pass under at least one muller. B. M. VENABLES.

**Production of steel abrasive materials.** S. D. LAYTON (B.P. 343,319, 24.2.30).—Steel "shot" is broken up into angular particles by crushing between rolls. B. M. VENABLES.

**[Dry] separation of intermixed divided materials.** A. E. WHITE. From R. PEALE, W. S. DAVIES, and W. S. WALLACE (B.P. 343,906, 23.10.29).—The apparatus is intended for the separation of materials that differ greatly in size but not much in sp. gr. It comprises two reciprocating tables, operated in tandem by the same mechanism but with independent control of the slope and stroke; the air supplies to the pervious decks are also separately controlled, though they may be derived from the same source. Provision may be made for a selected product from the first table to fall directly on to the second. B. M. VENABLES.

**Jigs and like concentrators and classifiers.** W. A. HARRIS (B.P. 342,682, 4.10.29).—In a multi-compartment jig the products are discharged through side or end chambers having adjustable, serrated, sliding doors to control the inlet apertures and mechanically operated or spring-controlled outlets which are also above the screen bed. B. M. VENABLES.

**Porous medium [perforated rubber sheet] for aerating apparatus [for pulp].** F. P. EGEBERG, Assee. of W. T. MACDONALD (B.P. 344,238, 17.3.30, U.S., 18.3.29).—A method of making small, clear perforations through rubber is described. In the case of rubber sheet (0.06—0.1 in. thick), this is sandwiched between sheets of stiff paper and cut by a punch, the pieces of rubber removed being dumb-bell shaped. Claim is made for the use, as an air-dispersing diaphragm, of sheet rubber (or other elastic material), supported by means such as perforated metal and having 50—500 apertures per in.<sup>2</sup>, each having a clear diam. of 0.005—0.02 in. B. M. VENABLES.

**Filtering apparatus.** PATERSON ENG. CO., LTD., and R. WHITEHEAD (B.P. 343,660, 24.12.29).—A system of pipes for the withdrawal of filtrate from, and the supply of wash-water and air simultaneously to, a filter bed is described. B. M. VENABLES.

**Filters or strainers for petrol or other liquids.** A. G. FENN, and ANGLO-AMERICAN OIL CO., LTD. (B.P. 343,219, 5.12.29).—A strainer suitable for attachment to the delivery end of a hose and easily opened for cleaning is described. B. M. VENABLES.

**Centrifugal machines.** T. BROADBENT & SONS, LTD., and J. V. LEVETT (B.P. 344,224, 5.3.30).—An

underdriven type of machine is described. The basket and motor form a rigid unit which is flexibly supported on the stand by a rubber buffer. B. M. VENABLES.

**Purification of liquids.** R. M. BERLINE (B.P. 317,453, 16.8.29, Luxemb., 16.8.28).—The purification of a liquid such as used transformer or lubricating oil, where the solid impurity is heavier and the liquid impurity more volatile than the stock, is effected in a centrifuge provided with an airtight casing at the discharge end in which the issuing fluid is subjected to a vacuum or to a current of inert gas while in a highly dispersed state. The gas may be circulated in a closed circuit through the casing, a condenser, and a reheater, or, alternatively, the raw oil may be heated. If the quantity of impurities is large the crude liquid may be pretreated in the centrifuge under atmospheric conditions. [Stat. ref.] B. M. VENABLES.

**Means for separating particles from free water.** C. B. THORNE (B.P. 343,327, 26.2.30).—The apparatus comprises a slowly rotating drum which is slotted and provided with a surrounding framework supporting filter medium in a number of V-shaped pieces which bridge the slots. The white-water or other prefilter is supplied to the interior of the drum and the collected pulp flushed from the filter, backwards through the slots, into a trough below the upper circumference of the drum. The drum is rotated intermittently, as required, by the incipient clogging of the then lower part, by means of a ratchet wheel operated (or not) by a hit-and-miss pawl controlled by a float. B. M. VENABLES.

**Evaporators.** O. ENGISCH (B.P. 344,125, 2.1.30, Addn. to B.P. 308,622; B., 1930, 399).—Pivoted, double-edged scrapers are placed between the deep corrugations of the rotating heating element described in the prior patent. B. M. VENABLES.

**Evaporators intended particularly for generating high-pressure steam.** K. BAUMANN (B.P. 342,815, 21.11.29).—A form of connexion between a tube-plate and an end-cover is described. B. M. VENABLES.

**Evaporation and crystallisation of liquids and drying of materials.** E.M.S. INDUSTRIAL PROCESSES, LTD., R. A. STOKES, and E. G. L. ROBERTS (B.P. 343,057, 11.11.29).—The apparatus comprises a number of troughs abreast, the material being assisted over the ridges by paddles. Heat exchange is provided for by jacketing the troughs throughout, and, in the case of some individual troughs, by having jackets or spaces in the paddles or their shafts. B. M. VENABLES.

**Crystallisation of salts.** A.-G. DER MASCHINEN-FABR. ESCHER WYSS & Co., Assecs. of F. KRÄMER (B.P. 342,942, 19.3.30, Ger., 21.3.29).—A graining evaporator is arranged to afford rapid circulation round the heater. Crystals of a finer grain than is desired, that drop out of the circulation into a quieter zone, are drawn out by a pump, together with the minimum quantity of brine, and returned to the upper part of the evaporator. When the crystals become coarse enough they are drawn out from time to time through the bottom of the apparatus. B. M. VENABLES.

**Crystallisation.** E. HOLLAND-MERTEN (B.P. 343,553, 19.11.29).—A crystalliser of the rocking-trough type is

provided with means for the independent regulation of the rate and level of inflowing liquid, of outflowing liquid, and of outgoing crystals. B. M. VENABLES.

**Apparatus for carrying out distillation processes.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 342,741, 9.8.28).—In a distillation process involving the bubbling of a scrubbing gas through the liquid in the still, particularly when operating under reduced pressure, the scrubbing gas is used over and over again, being circulated from the upper part of the receiver to the lower part of the still by a pump. B. M. VENABLES.

**Continuous distillation apparatus for the refining of oils and fats and for like purposes.** BIRMINGHAM ALUMINIUM CASTING (1903) Co., LTD., P. PRITCHARD, and G. W. LACEY (B.P. 342,921, 25.2.30).—The apparatus comprises a vertical cylindrical vessel subdivided by a number of radial partitions. The liquid flows in series through all the compartments thus formed, but the vapour space is common to all. In addition to external heat, steam or other gas is injected through passages and fine apertures formed in the thickness of the partitions. B. M. VENABLES.

**Condensing and cooling of vapours.** E. B. GRUSELLE (B.P. 344,328, 26.7.30).—An ejector condenser is described. In addition to the supply to the ejector, water is sprayed on the underside of a conical baffle against which the incoming vapour impinges; provision is made for passing a refrigerant through a coil in the sump. B. M. VENABLES.

**Bubble-tower grid.** L. E. WINKLER and F. C. KOCH (U.S.P. 1,774,581, 2.9.30. Appl., 1.8.27).—A bubble cap with serrated edges is surmounted by a horizontal perforated plate or grid below the surface of the liquid to break up the rising bubbles. B. M. VENABLES.

**Preparation of emulsions.** E. B. MYERS, Assr. to H. W. DIX (U.S.P. 1,774,609, 2.9.30. Appl., 6.9.28).—The substance to be emulsified is ground in sulphite waste liquor between flexibly supported grinding surfaces, revolving preferably in opposite directions. Several stages with or without a preliminary ordinary grinding may be used. B. M. VENABLES.

**Flaker for production of relatively thick flakes of a congealed liquid.** E. A. TAYLOR, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,775,177, 9.9.30. Appl., 13.10.27).—A cooled drum dipping into a bath of, e.g., molten soap or sodium sulphide is rotated under such conditions that only a thin layer is formed at each revolution, but the scraper is put into engagement only for exactly one revolution out of several. B. M. VENABLES.

**Gas-cleaning apparatus.** J. H. LAWRENCE, Assr. to METROPOLITAN ENG. CORP. (U.S.P. 1,774,604, 2.9.30. Appl., 27.3.25).—The gases are directed against the surface of water to collect and quench cinders or large particles, and are then subjected to electrical precipitation. B. M. VENABLES.

**Dust separators.** F. W. WILSON, and TILGHMAN'S PATENT SAND BLAST Co., LTD. (B.P. 344,255, 1.4.30).—A system comprising a bag filter and exhaust fan is provided with five valves so that a reverse current of

atmospheric air may be blown through the bags to dislodge the collected dust. B. M. VENABLES.

**Removal of solid particles from hot or corrosive gases.** J. P. BAXTER, E. STEEL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 344,227, 6.3.30).—The gases are filtered through the walls of a hollow prism of porous mineral substance which is contained in a housing of acid-resisting material constructed in such a way that a reverse current of gas may be applied to each wall of the prism in turn. The dislodged dirt falls into a hopper and is removed therefrom at intervals by a blast of inert gas. B. M. VENABLES.

**Air liquefaction.** I. H. LEVIN, Assr. to GAS INDUSTRIES Co. (U.S.P. 1,775,434, 9.9.30. Appl., 1.6.25).—An exchanger for a fluid which is likely to congeal comprises a vertical bundle of tubes provided with restrictions at their upper ends to increase the velocity and pressure-drop of the inner cooled fluid at or near its outlet. The outer fluid flows downwards in a zig-zag manner. B. M. VENABLES.

**Gas analysis.** AKTIEB. CARBA (B.P. 342,995, 2.8.30. Swed., 3.8.29).—The apparatus is particularly suitable for the determination of both oxides of carbon in flue gases. Before reaching apparatus of known type for the continuous determination of carbon dioxide the sample of gas is passed through a combustion chamber which is heated electrically and unheated during alternate periods. The automatic switch may be controlled either by time or by the temperature of the combustion chamber itself, and if combustion is arranged to take place at two temperatures, e.g., 250° and 700°, the quantity of combustible constituents other than carbon monoxide may also be measured. B. M. VENABLES.

**Diffusion apparatus for detecting and indicating the presence and proportion of absorbable gases.** H. R. WEBSTER (B.P. 342,949, 2.4.30).—A chemical absorbent is placed within a porous cylinder; one end-closure of the cylinder affords means for easy renewal of the absorbent, the other embodies a thin diaphragm or other means for measuring the partial vacuum. B. M. VENABLES.

**Apparatus for automatically testing gases [volumetrically].** C. A. HARTUNG (B.P. 343,874, 21.8.29).—More than one constituent of a mixture of gases may be determined by the same reacting gas by maintaining the temperature of the reaction chamber and contact substance (e.g., by an electrically heated wire spiral) between two limits, the lower determined by the temperature at which the desired reaction starts, and the upper by the commencement of undesired reactions. E.g., carbon monoxide may be oxidised at 250°, and in the remainder of the same sample hydrocarbons may be oxidised at 800°. B. M. VENABLES.

**Catalytic gaseous reactions.** J. L. BRILL (B.P. 342,854, 31.12.29. U.S., 31.12.28).—In carrying out exothermic gas reactions at temperatures higher than the external gas conduits can withstand, and using a main catalyst which is not active at such lower temperature, the pressure-resisting vessel may be made of metal superior to that necessary for the drawn pipes and may also be kept cool by the entering gases. The initial rise of temperature is obtained by the use of an auxiliary

catalyst which effects an exothermic reaction, not necessarily the finally desired reaction, at a lower temperature. Two methods of arranging the catalysts are described.

B. M. VENABLES.

[Carrying out] catalytic gas reactions [e.g., synthesis of ammonia]. DU PONT AMMONIA CORP., Assees. of R. WILLIAMS (B.P. 344,119, 31.12.29. U.S., 31.12.28).—In a process wherein the poisoning of the main catalyst is prevented by a subsidiary reaction in an auxiliary catalyst (which may sometimes be of the same nature as the main catalyst) the heat of the purifying reaction is not usually sufficient to maintain the temperature of the auxiliary catalyst if the feed gases were passed through it first. To avoid the separate heating of the auxiliary catalyst the process is worked on the following cycle: the gaseous reactants are passed through the hot main catalyst, the make-up unpurified gases are added, and the whole is passed through the auxiliary catalyst, the desired products being then removed in external apparatus and the residual gaseous reactants returned.

B. M. VENABLES.

Gaseous reactions taking place in the presence of steam or water vapour. O. PIETTE, and UNION CHIM. BELGE, Soc. ANON. (B.P. 342,701, 4.11.29).—When carrying out gaseous catalytic reactions in the presence of excess of steam, the steam condensed from the cooling emergent gases is transferred to the entering gases in the stages of a heat exchanger, the transfers being made from and to points at substantially the same temperature; the pressure of the hot and wet gas at the inlet end of any transfer pipe is slightly above that of the comparatively cool and dry gas at the outlet.

B. M. VENABLES.

Sp. gr. indicators or recorders. J. L. HODGSON (B.P. 343,218, 5.12.29).—The apparatus comprises a pair of displacers balanced on a scale beam and submerged in the liquid of which the sp. gr. is to be determined. The liquid is continuously renewed and may be under pressure, in which case the swing of the beam may be transmitted to the outside by magnetic means. One displacer is much larger than the other and is hollow. It is filled with liquid of suitable coefficient of expansion in relation to the liquid under test, and, when the temperature rises, the excess liquid is expelled to another vessel situated so as to have no moment, i.e., surrounding or under the pivot.

B. M. VENABLES.

Furnace roofs. E. BLOCK (B.P. 344,724, 19.5.30). SCHEIDHAUER & GIESSING A.-G. (B.P. 344,754, 4.7.30. Ger., 17.7.29).

Air- and water-cooled walls for furnaces and the like. AMER. ENG. CO. (B.P. 342,806, 19.11.29. U.S., 5.7.29).

Mechanisms for opening the doors of furnaces, retorts, etc. M. B. WILD & CO., LTD., and J. A. TURNER (B.P. 344,546, 14.12.29).

Refrigerating machines. E. KLEPETAR and A. SCHOENFELD (B.P. 344,706, 25.4.30. Austr., 19.7.29).

Lubricating devices for centrifugal separators. ARTIEB. SEPARATOR (B.P. 345,097, 20.3.30. Swed., 22.3.29).

Moving-chamber [measuring] apparatus for granular and pulverulent materials. C. W. O'LEARY (B.P. 343,987, 31.8.29).

Drum-type mill.—See V. Catalyst carriers.—See VII. Lehr.—See VIII. Fireproof paste.—See XIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Inflammation of coal dusts: effects of the presence of firedamp. T. N. MASON and R. V. WHEELER (Safety in Mines Res. Bd., 1931, Paper No. 64, 33 pp.).—As a result of tests on the dusts from 7 coals of volatile matter content (ash-free dry basis) 13–44% in the Buxton explosion gallery, it is shown that (i) the inflammability of a coal dust, determined experimentally, can be expressed as the least proportion,  $S$ , of incombustible matter required to be added to suppress inflammation, or as the ratio  $100/(100-S)$ ; (ii) there is a fairly regular relationship between the volatile matter content of a coal ( $V$ ) and its inflammability: the index of inflammability,  $100/(100-S)$ , is given by the derived expression  $V/12.5$ , i.e.,  $S = 100 - 1250/V$ , an expression which enables calculation to be made for any coal dust, of known volatile matter content, of the proportion of incombustible matter required to be added to suppress inflammation; (iii) from the experimental results with 7 coal dusts, and up to 4% of firedamp in the air, for each 1% of firedamp the proportion of incombustible dust required to suppress inflammation must be increased by  $(100-S)/6$ ; (iv) the additional amount of incombustible dust required to suppress inflammation of coal dusts of known volatile matter content for each 1% of firedamp in the air can be derived from the relationships given above, and is  $S$  (additional)  $= 208/V$ .

C. B. MARSON.

The hydrogen in coal. A. ECCLES, G. H. KENYON, and A. McCULLOCH (Fuel, 1931, 10, 4–15. Cf. Marsh, McCulloch, and Parrish, J.S.C.I., 1929, 48, 167 T; Eccles and McCulloch, B., 1930, 933).—A study has been made of the chlorination and subsequent distillation of four British coals of different rank. The finely-divided coals were subjected to the action of a current of chlorine for 50 hrs. and the products were stored in a vacuum desiccator over solid potassium hydroxide until they had attained a constant weight. The percentage increases in weight varied from 31% to 66%, and the increases in volume from approx. 25% to 60%. The chlorinated coals were distilled in 100° stages up to 900° in a laboratory assay apparatus, and the volume and composition of the gases evolved at each stage were determined. No tar was evolved. No paraffin hydrocarbons could be detected in the gases evolved below 500°, and in those evolved at higher temperatures methane was the only hydrocarbon present. Hydrochloric acid began to be evolved below 200°, and the evolution continued up to 500–600° and then ceased except with one coal ("Welsh Main"), which continued to evolve hydrochloric acid, but at a diminished rate, up to 900°. The results obtained with the other three coals can be summarised as follows: (a) the percentage of the total hydrogen evolved as hydrochloric acid and water during chlorination, and as hydrochloric acid

during distillation, was considerably greater with a bituminous and a sub-lignitic coal than with an anthracite; (b) the percentage of the total hydrogen evolved as water above 200° increased in the same order as the oxygen contents of the coals; (c) the percentage of the total hydrogen evolved as methane was approximately the same with all three coals; (d) the percentage of the total hydrogen evolved in any form above 600° was of the same order as the proportion remaining in the coke produced by the direct carbonisation of the coal at 600°; (e) the hydrogen contents of the cokes obtained by distilling the chlorinated coals at 900° were less than those of the cokes obtained by direct distillation of the coals at the same temperature. Chlorination and distillation caused a concentration of the carbon in the coke at the expense of the gaseous and liquid hydrocarbons obtained when the coal is distilled in the usual manner. The abnormal behaviour of one coal is attributed to its high content of pyritic sulphur, the presence of pyrites modifying the action of the chlorine on the coal.

A. B. MANNING.

**Combustion tests with Illinois coals.** A. P. KRATZ and W. J. WOODRUFF (Univ. Ill. Eng. Exp. Sta. Bull., 1930, No. 313, 58 pp.).—Performance characteristics in boiler tests are recorded.

#### CHEMICAL ABSTRACTS.

**Primary oxidation of bituminous coals.** W. FUCHS and O. HORN (Brennstoff-Chem., 1931, 12, 65—67).—By treating the coal with nitric acid ( $d$  1.4) at 90—94° the humins have been converted into products having the properties of hydroxycarboxylic acids. These form potassium salts on treatment with alcoholic potassium acetate, and ether-esters on treatment with methyl alcohol and hydrochloric acid, or with diazomethane in ether. The primary oxidation products are partly soluble in organic solvents, but are not always peptisable by aqueous sodium hydroxide. Treatment with alkali and then with aqueous pyridine converts them almost completely into water-soluble products; on acidification of the solution with hydrochloric acid, acids of higher basicity than that of the primary products are precipitated. The oxidation products from anthracite and boghead coals cannot be converted into water-soluble products by this treatment.

A. B. MANNING.

**Action of various solvents on coal.** J. C. PEW and J. R. WITHROW (Fuel, 1931, 10, 44—47).—An Ohio coal was extracted with various organic solvents. The monoalkyl ethers of ethylene and diethylene glycols dissolved 10—12% of the coal. The dialkyl ethers had a considerably lower solvent action, whilst that of the esters of the monoalkyl ethers was very slight. Glycol itself had no solvent action. A solution of potassium hydroxide in ethylene glycol monoethyl ether extracted over 30% of the coal, which was of the same order as the amount extracted by pyridine. The residue showed a high affinity for oxygen. A study of the action of these solvents on a variety of coals should yield useful information on their coking properties.

A. B. MANNING.

**Heats of coking and of decomposition of coal.** III. E. TERRES and K. VOITURET (Gas- u. Wasserfach, 1931, 74, 97—101, 122—128, 148—154, 178—183).—

The methods used previously for determining the heat of coking (cf. B., 1927, 177; 1928, 508) have been modified to allow of more accurate calibration of the apparatus. The method consists essentially in heating the coking system electrically by a current of constant magnitude until thermal equilibrium is established. The thermal capacity of the empty coking system in the range 600—1100° is then directly proportional to the temperature and is independent of the time and amount of heat required to reach the desired coking temperature. The coking system is contained in a water-jacketed calorimeter and is heated to the desired coking temperature and maintained at that temperature until thermal equilibrium is established in the bomb constituting the coking system. The total heat added (a) to the system during this period is found by measuring electrochemically the total current used. The heat absorbed (b) by the calorimeter is observed, and the heat content (c) of the coking system at the coking temperature used is deduced from previous calibration experiments in which quartz powder or graphite is used. The heat utilised in coking the air-dried coal (10 g.) is then given by  $a-b-c$ . It is necessary to measure the heat content of the gaseous products when the heat of decomposition of the coal is to be determined. Fifteen coking and gas coals were examined by the method described. Each coal gave a characteristic curve for the heats of coking at temperatures between 600° and 1100°. No relationship was observed between the results of analysis of the coals and the heat of coking at various temperatures. Increase in the time of storage resulted in increased heat of coking due to a decrease in the exothermic reactions accompanying carbonisation. The heat of coking of moist coal could not be calculated from the heat of coking of air-dried coal. The methods in use for determining the heat of decomposition of coals are discussed. The values for the heat of decomposition of 28 coals (including the English coals, Boldon and Easington) in the range 600—1100° were determined. A relationship was shown to exist between heat of coking and heat of decomposition for the coals examined. It is concluded that the heat of coking of a coal which undergoes neither exothermic nor endothermic reactions during carbonisation amounts to 325 kg.-cal. per kg. of "pure coal." Coals undergoing exothermic reactions have smaller, those undergoing endothermic reactions larger, heats of coking. Large negative values of heats of decomposition are possible by carbonisation at 1000°, but the positive value of heat of decomposition rarely exceeds 50 kg.-cal. per kg. of "pure coal."

H. E. BLAYDEN.

**Compressed low-temperature coke.** E. ROSER (Brennstoff-Chem., 1931, 12, 86—87).—The finely-ground coal was charged into vertical cylinders of highly heat-resistant steel (30 cm. diam.; 280 cm. high), and compressed therein to a bulk density of about 1.0. The charged cylinders were placed in the chamber of an ordinary coke oven and the coal was carbonised therein at 750°. Strong, uniform cokes were produced, even when carbonising a 1:1 blend of coking coal and anthracite. The yields of gas and tar were not determined.

A. B. MANNING.

**"Melting" of coal during coke formation.** R. G. DAVIES and R. V. WHEELER (Fuel, 1931, 10, 100—108).—The plastic flow of a number of French and British coals has been studied by Audibert's method (B., 1927, 383). Although this method has been found of great value in the investigation of the phenomena of coking, Audibert's conclusions relative to the "melting" of coal during coke formation have not been confirmed. The pellet of coal does not become perfectly fluid between the softening and intumescence points; moreover, the apparent homogeneity of the heated pellets is due to surface flow occurring when the specimen is polished. If the surface is subsequently etched the pellet is seen to be heterogeneous. A. B. MANNING.

**Determination of the hardness of coke.** F. G. HOFFMANN (Brennstoff-Chem., 1931, 12, 61—65. Cf. Dörfinger, B., 1927, 898; Wolf, B., 1928, 177).—The influence of fissures and internal strains in the coke on the determination of its hardness by the trommel test, the shatter test, and Wolf's pressure test is discussed, and a distinction is drawn between the true hardness of the coke and its "lump strength." Thus a highly fissured coke might possess a low "lump strength" combined with great hardness. The most suitable method for evaluating both these factors consists of a trommel test followed by at least a double-sieve separation of the material, in which, *e.g.*, the percentages over 40 mm. and under 10 mm. are determined. Before subjecting the coke to the test a preliminary sieving over the 40-mm. sieve is recommended.

A. B. MANNING.

**Origin and decomposition of organic sulphur compounds under gas-making conditions, with particular reference to the rôle of the carbon-sulphur complex.** J. C. HOLTZ (Fuel, 1931, 10, 16—30).—The gas produced on cracking a high-sulphur oil (3.6% S) at 650° contained no carbon disulphide; this, however, was formed in increasing amounts as the cracking temperature was raised above 650°. Carbon disulphide was formed also when a sulphur-free oil was cracked at 850° in the presence of a gas containing hydrogen sulphide; part of the latter, however, was converted into sulphur compounds other than carbon disulphide. When nitrogen containing hydrogen sulphide was passed over sugar charcoal at 850° neither hydrogen sulphide nor carbon disulphide could at first be detected in the outlet gas; after a time a gradually increasing amount of hydrogen sulphide was detected in the gas, and after a further interval carbon disulphide appeared among the reaction products; at the end of the experiment the charcoal contained 2.8% S. When the high-sulphur gas oil was cracked in a tube packed with pumice the sulphur was at first absorbed by the layer of deposited carbon and was later evolved as carbon disulphide. These phenomena are attributed to the formation of a solid carbon-sulphur complex. The influence of various factors on the equilibrium conditions reached when nitrogen containing hydrogen sulphide was passed over charcoal was studied. A rise in the temperature or an increase in the concentration of the hydrogen sulphide caused an increase in the concentration of the total organic sulphur as well as of the carbon disulphide in the outlet gas. The presence of

carbon monoxide or oxygen caused an increase in the concentration of organic sulphur, the increase taking place principally in compounds other than carbon disulphide. Addition of hydrogen caused a decrease in the concentration of organic sulphur. Addition of water vapour did not affect the total organic sulphur, but decreased the concentration of carbon disulphide.

A. B. MANNING.

**Application of glass filters in gasworks' practice.** H. BRÜCKNER (Gas- u. Wasserfach, 1931, 74, 121—122).—The determination of ammonia in gases (*e.g.*, coal gas) by absorption in standard sulphuric acid may be effected with accuracy and economy of time by using washing vessels in which the gas enters the washing liquid through a sintered glass filter plate, which allows of efficient washing of a rapid stream of gas. The results of the application of this method to the control of ammonia washers are given. Sintered glass washing vessels may also be used in the determination of hydrogen sulphide in gas by absorption in iodine solution and for the saturation of a gas with the vapour of a liquid, but not for reactions in which precipitation occurs.

H. E. BLAYDEN.

**Determination of tar fog in coal gas, using glass filters.** H. BRÜCKNER (Gas- u. Wasserfach, 1931, 74, 183—184).—The most efficient separation of the tar is effected by passing the gas (at a rate of 80—120 litres/hr., according to the tar content) through a weighed dry filter consisting of a pad of cotton wool contained in a tube sealed on to a bulb containing two separated, sintered glass filters. The amount of gas passed through the filter is measured by a meter. The filter is finally dried to a constant weight by passing through it a current of dry air at 50—60°. The increase in weight of the filter gives the amount of tar separated. The filter is readily cleaned by the usual solvents or chromic acid mixture.

H. E. BLAYDEN.

**The tetralin process [for gas].** G. WEISSENBERGER (Gas- u. Wasserfach, 1931, 74, 154—156).—For efficient action the tetralin process requires complete dispersion of the tetralin in the gas. Examples of two types of apparatus for this purpose are described. One type involves vaporisation of the tetralin (*e.g.*, by means of electrical heating elements immersed in the liquid tetralin contained in a suitable reservoir) and injection of the vapour into the gas mains; in the other type the liquid tetralin is injected as a fine spray into the gas-mains by means of an injector actuated by compressed gas. The amount of tetralin delivered to the mains is determined by the rate of heating in the one case and by the amount of suction at the injector in the other, the amount required being:  $x = 5m(A + 8)/100,000$ , where  $x$  is the amount of tetralin required in kg. per day,  $m$  is the quantity of gas in cub. m. per day, and  $A$  is the naphthalene content in g. per 100 cub. m.

H. E. BLAYDEN.

**Simple waste-gas diagram for excess air.** L. ZIPPERER (Gas- u. Wasserfach, 1931, 74, 199—200).—Directions are given for the construction of a diagram on the basis of the composition of the waste gas resulting from theoretically complete combustion of the gas under consideration. The excess of air corresponding to a given percentage of carbon dioxide in the dry



waste gas and the proportions of the individual constituents of the dry waste gas may be determined from the graph.

H. E. BLAYDEN.

**Recovery of phenol from gas liquor.** C. SCHÖNBURG (Brennstoff-Chem., 1931, 12, 69—71).—The liquor is extracted with tolyl phosphate, and the phenol is recovered by distillation in steam under reduced pressure. Tolyl phosphate possesses the advantages over benzene of a higher solubility for phenol (the distribution coefficient of phenol between tolyl phosphate and water being about 20 times that between benzene and water), a lower solubility in water or gas liquor, and a much lower vapour pressure (b.p. 280—285°/10 mm.). Moreover, the method of recovering the phenol is simpler, involving only a distillation instead of an extraction with sodium hydroxide solution. Economically considered, these advantages greatly outweigh the increased cost of the solvent.

A. B. MANNING.

**Tar removal by electrostatic precipitation (Simon-Carves system).** V. F. GLOAG and J. P. V. WOOLAM (Fuel, 1931, 10, 137—141).—The plant comprises a transformer, a static copper oxide rectifier, and a precipitating chamber built up of 6-in. steel tubes down the centres of which hang the high-tension electrodes. The tar is precipitated on the sides of the steel tubes, which are earthed, and collects at the bottom of the chamber. The power consumption is less than 1 kw. per 100,000 cub. ft. of gas, and the back-pressure thrown by the plant is less than  $\frac{1}{4}$  in. W.G.

A. B. MANNING.

**Mechanism of the action of the electrical discharge on methane.** K. PETERS and O. H. WAGNER (Brennstoff-Chem., 1931, 12, 67—68; cf. B., 1930, 848).—By suitably varying the experimental conditions it is possible to produce ethylene, ethane, and higher hydrocarbons, in addition to acetylene, by the action of the electrical discharge on methane under reduced pressures. The maximum efficiency of production of these hydrocarbons, however, is low. As the load is increased the colour of the discharge changes suddenly from pale blue to yellow. Before this change occurs the spectrum of the discharge shows the CH bands, and also, owing to the presence of nitrogen, the CN bands; after the change the C:C bands (Swan spectrum) and C<sup>+</sup> bands are observed in addition. The mechanism of the reaction is discussed in relation to these phenomena.

A. B. MANNING.

**Sakhalin crude oil.** S. S. NAMETKIN and S. S. NIFONTOVA (Neft. Choz., 1930, 19, 421—422).—A Chakrui asphalt-base crude oil, containing no gasoline and little kerosene, *d* 0.943, *E*<sub>50</sub> 4.38, and a Lyangrui crude oil, *d* 0.951, *E*<sub>50</sub> 3.6, are described.

CHEMICAL ABSTRACTS.

**Distillation of heavy bottom oils by the Pengu-Gurvich-Nersesov method.** G. TER-GRIGORYAN (Azerbaid. Neft. Choz., 1930, No. 11, 74—82).—The plant is described, and yields and composition of products are recorded.

CHEMICAL ABSTRACTS.

**Significance of the Edeleanu refining process as a source of transformer, switch, and turbine oils.** F. FRANK (Erdöl u. Teer, 1930, 6, 357—358, 375—376,

392—399; Chem. Zentr., 1930, ii, 3882—3883).—The oils may be refined and the decomposition controlled so as to convert oils not formerly applicable for all purposes (such as those of American origin) into valuable transformer or turbine oils. A number of oils from widely differing origin were treated by the process and the resulting oils of high value investigated. The results are summarised graphically and in tables. Special attention is directed to the ageing phenomena produced in these oils by the usual methods, and the relation of these phenomena to the behaviour of the oil in practice is discussed.

H. E. BLAYDEN.

**Significance of the Edeleanu refining process for users of transformer, switch, and turbine oils.** F. FOERSTER (Petroleum, 1931, 27, 60—65).—Objection is raised to the adoption of Edeleanu oil for transformer purposes on the basis of insufficient evidence of its merits. The investigations of Frank (cf. preceding abstract) are criticised adversely. It is considered that the method of comparison used by this author is open to objection. Revision of Frank's results of examination of the effect of catalysts, such as lead, copper, etc. indicate that the Edeleanu oil is inferior in some respects to the Russian oil with which it was compared.

H. E. BLAYDEN.

**Refining of motor benzols and other fuels. The Instill process.** ANON. (Gas World, 1931, 94, Coking Sect., 26—30).—In the Instill process, which endeavours to eliminate the use of large amounts of sulphuric acid, naphthalene and wash oils are removed by distillation, and the bases by sulphuric acid (*d* 1.30) in a continuous pyridine scrubber. The base-free spirit passes to the washer, and is heated to 55°, during which period 2—3% by wt. of "Instill refining medium" is added to the charge. Agitation is continued for 90 min. The benzol is then run through a filter and continuous neutraliser to the still and rectified. The extra plant required for the process is described. The refining medium consists of a mixture of ferric sulphate, absorbent earth, and free sulphuric acid. The benzol obtained conforms to N.B.A. specifications. In motor trials Instill benzol gave superior results to acid-washed spirit obtained from the same crude spirit; it contains more unsaturated hydrocarbons and less free sulphur. Cost sheets of the Instill and acid process are given.

A. H. EDWARDS.

**Removing naphthenic acids [from oil].** Y. EMMUIL (Azerbaid. Neft. Choz., 1930, No. 11, 90—95).—In the apparatus described 85—90% of the naphthenic acids may be extracted from the oil by means of a caustic solution. The naphthenic acid contents of various fractions of Baku petroleum are recorded.

CHEMICAL ABSTRACTS.

**Lacquer diluents.**—See XIII. Coal as fertiliser.—See XVI.

PATENTS.

**Coal-washing machines.** G. NORTON (B.P. 344,082, 7.12.29).—A machine of the washer-box type is fitted with one or more air-compressing cylinders which control directly the supply of air to or from the chambers of the box. Suitable compressor designs and mountings are described in detail.

R. H. GRIFFITH.



**Drying and pulverising of coal.** W. RUNGE and E. A. PACKARD, Assrs. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,775,324, 9.9.30. Appl., 15.1.26).—Two separators, two fans, a feeder, and a mill are arranged in two circuits so that coarse or already pulverised coal can be dried in waste flue gases (diluted, if need be, with fresh air) and, after grinding if necessary, transported away in fresh air. B. M. VENABLES.

**Production of fuel to selected specifications.** A. A. ROBERTS (U.S.P. 1,770,627, 15.7.30. Appl., 28.6.29).—Blocks of artificial fuel are prepared by subjecting moist mixtures of powdered carbonaceous materials (*e.g.*, peat, lignite, coal, or wood waste), small amounts of a cementing material (such as a hydraulic cement), and an accelerator of combustion (*e.g.*, magnesium chloride) to pressure in a cold mould, preferably slightly conical. The hardness and porosity of the block are under control. H. E. BLAYDEN.

**Obtaining a [finely-divided] carbonaceous product.** G. ANTONOFF and J. FREEDLAND (B.P. 342,653, 23.10.29).—Organic matter, such as wood shavings, is heated in an autoclave with dilute sulphuric acid for a short time, under a pressure of a few atm. The product, after washing, is oxidised by nitric acid, chlorine, or a similar reagent, after which it will dissolve in ammonia. Addition of acid to the filtered solution precipitates a moist colloidal material, which is then mixed with a large excess of phosphoric acid and carbonised at 400–450°. Treatment with water then separates an extremely finely-divided charcoal or carbon, which may have an apparent density as low as 0.078. Modification of the process may involve pretreatment with alkali and drying in admixture with sodium carbonate or sulphur in place of phosphoric acid. R. H. GRIFFITH.

**Low-temperature carbonisation.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 343,435, 11.11.29 and 11.8.30. Addn. to B.P. 301,975; B., 1929, 119).—Bituminous material is carbonised by contact with hot gases, the process being carried out by passing a stream of gas through small apertures in a number of superimposed grates mounted in a vertical furnace. The raw material is fed to the top grate and is gradually carried across it to a sluice through which it passes to a lower level; in this way it is continually agitated by the rising gas and gradually subjected to rising temperatures. The tars and gases produced at different levels may be withdrawn and condensed separately, or may be led into a common offtake. R. H. GRIFFITH.

**Retorts for carbonisation of coal and similar materials.** CHAMBER OVENS, LTD., A. H. LYNN, and N. J. BOWATER (B.P. 342,720, 4.11.29).—Superheated steam is raised, in coke-oven and similar carbonising apparatus, by a tubular system which is built inside the brickwork, at the top or in the intermediate walls of the setting. The steam thus provided is employed in making water-gas in the retorts; provision is also made for cooling the charging floors. R. H. GRIFFITH.

**Apparatus for the carbonisation of fuel.** E. RAMMELKAMP, Assr. to CHEM.-TECHN. GES.M.B.H. (U.S.P. 1,775,570, 9.9.30. Appl., 10.9.27. Ger., 21.9.26).—

Narrow, vertical heating and distillation chambers are arranged radially about a vertical axis and inside a closed chamber. The material to be carbonised (*e.g.*, coal) falls from a hopper above the chamber into a charging space containing a vertical cylinder which rotates about an eccentric axis so as to push the coal through the distillation chambers. The solid and gaseous products escape through an annular space surrounding the heating and distillation chambers and may be suitably collected. H. E. BLAYDEN.

**Extracting values from coals and the like.** F. C. GREENE and I. F. LAUCKS, Assrs. to OLD BEN COAL CORP. (U.S.P. 1,775,280, 9.9.30. Appl., 9.4.19).—A vertical, cylindrical, externally heated retort is provided internally with a helix mounted on a rotating tubular shaft, the diameter of which increases gradually to a maximum at the middle of the retort, and then decreases. The material to be distilled is carried from the top to the bottom of the retort, is compressed in the reduced space at the middle of the retort while in a plastic condition, and is discharged at the bottom of the retort. The products of distillation pass through holes in the tubular shaft and are collected. The solid products may be subjected to a secondary distillation. H. E. BLAYDEN.

**Distillation of solid carbonaceous material, and manufacture of gas.** H. NIELSEN and B. LAING (B.P. 342,817, 22.11.29. Addn. to B.P. 292,060; B., 1928, 593).—The process of the prior patent is applied to a coking coal, ranging in size from  $\frac{1}{2}$  cub. in. to 2 cub. in., after preliminary oxidation: the resulting semi-coke is used as a source of water-gas. R. H. GRIFFITH.

**Low-temperature distillation of carbonaceous material.** A. KATH (B.P. 342,983, 6.5.30).—An inclined rotary kiln is provided with a large number of tubes passing down its length, through which powdered coal or similar material is carried by gravity. Heating of the charge is effected by circulation of gases through the annular spaces between the tubes and the outer shell of the kiln, and also by other gases which pass independently through the coal. Means are provided for regulating the flow of materials through the system, and for controlling the temperature of the internal and external gas streams. R. H. GRIFFITH.

**Low-temperature distillation or carbonisation of carbonaceous material.** C. HONNAY (B.P. 344,301, 23.5.30).—An endless conveyor of wire gauze or perforated metal sheet is fitted with rows of transverse scrapers, mounted in a zig-zag fashion, which pass through and over a thin layer of coal heated on the grate of a gas-fired horizontal oven. In this way the coal is carried continuously forward, with thorough stirring, and the gases or vapours produced in the distillation escape in an upward direction. Dust particles are thus largely removed from the crude gas by passage through the lower part of the gauze, and the sensible heat of the distillation products is employed in a pretreatment of fresh coal, which is carried into the hot zone on the upper part of the same conveyor. R. H. GRIFFITH.

**Apparatus for destructive distillation of cereal husks and other vegetable waste.** P. (GRAF) VON

RADOLIN, Assr. to G. SAUERBREY MASCHINENFABR. A.-G. (U.S.P. 1,770,636, 15.7.30. Appl., 19.5.23).—Cereal husks etc. are passed between compression rollers into an airtight chamber, where they are dried by an opposing current of hot distillation gases; the product falls on a heated rotating drum provided with flanges which carry the resulting carbonised material to the lower part of the chamber, where it may be discharged without allowing air to enter the apparatus. An off-take pipe connected to the chamber withdraws the gaseous distillation products, a portion of which is recirculated in order to dry the material to be carbonised. The carbonised product may be combined with the tarry products of distillation to produce briquettes.

H. E. BLAYDEN.

**Smoke-generating apparatus and compositions therefor.** C. BOULANGER (B.P. 343,023, 7.11.29. Fr., 10.11.28).—A composition of hexachloro-benzene, -ethylene, and -toluene, carbon tetrachloride, powdered zinc, and sodium nitrate, and an accelerating composition containing also calcium silicide, are claimed.

W. J. WRIGHT.

**Production of carbon and hydrogen chloride.** J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 343,477, 10.10.29).—Hydrogen chloride and carbon are obtained by treating a partly or wholly chlorinated hydrocarbon at a high temperature with a hydrocarbon, a chloro-derivative thereof, or chlorine. The proportions of the various reactants should be chosen so that the numbers of gram-atoms of chlorine and of hydrogen are approximately equal. (Cf. following abstract.)

R. H. GRIFFITH.

**Production of carbon [black].** J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 343,676, 7.1.30).—Hydrogen chloride, which is produced when chloro-derivatives of hydrocarbons react with hydrocarbons, is recovered and used for preparing fresh quantities of chlorinated hydrocarbons. This may be effected by electrolytic production of chlorine, or by an indirect method in which cuprous chloride is converted into the cupric compound and the latter reacts with a hydrocarbon (cf. preceding abstract).

R. H. GRIFFITH.

**Purification of carbon black.** J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 343,108, 10.10.29).—Crude carbon, obtained by the interaction of chlorine or a chlorinated hydrocarbon with a hydrocarbon, is purified by heating in a stream of gas. The solid is admixed by injector action of the gas, and the mixture is passed vertically through a heated zone at such a rate that deposition of carbon by gravity does not occur. If the gas is inert the temperature may be as high as 900°, and the time of treatment is correspondingly very short, but with gases containing combined oxygen the temperature must not exceed 500°. If free oxygen is present the maximum permissible is only about 150°.

R. H. GRIFFITH.

**Manufacture of adsorbent carbons.** O. L. BARNEBEY (U.S.P. 1,774,585, 2.9.30. Appl., 6.11.24. Renewed 14.12.27).—Adsorbent carbon is manufactured by partly carbonising carbonaceous materials (e.g., coal, oil shales, wood waste) at 350–900°, extracting the product with a suitable solvent (e.g., pyridine), heating the

residue with oxygenated gases at 500–900°, and extracting a substantial proportion of ash constituents by treatment with caustic soda solution (e.g., 5% solution) and with dilute (2%) hydrochloric acid.

H. E. BLAYDEN.

**Destructive hydrogenation of coal, tar, mineral oils, and the like.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 344,165, 31.1.30).—Waste gases obtained in cracking or destructive hydrogenation of tars, oils, etc. are passed through an electric arc (cf. B.P. 294,494 and 332,057; B., 1928, 738; 1930, 893) and thus serve as a source of the hydrogen required for the hydrogenation. The crude reaction gas is freed from carbon dioxide and hydrogen sulphide, and passed through a suitable arc furnace; steam or carbon dioxide may be added, and useful by-products such as acetylene, ethylene, or carbon black may be obtained as well as hydrogen.

R. H. GRIFFITH.

**Extraction of oils from solid residues obtained in the destructive hydrogenation of coal and the like.** K. GORDON, H. HARPER, W. I. JONES, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 342,840, 18.12.29).—Residues from destructive hydrogenation processes are treated with a countercurrent stream of hot inert gas, or steam, while passing through a vessel heated externally by another stream of gas. The temperature of the gas in contact with the oily mixture is regulated to about 350° by diluting it with cool gas that remains after condensation of oils leaving the plant. In a continuous process about 80% of the oil may be recovered.

R. H. GRIFFITH.

**Gas-producing oven.** C. OTTO (U.S.P. 1,770,285, 8.7.30. Appl., 5.3.29. Ger., 25.11.25).—The vertical heating flues of a horizontal oven are provided with fuel-gas inlets at various heights above the bottom of the flues. Air is supplied at the bottom of the flue and the distribution of heat in the flue may be controlled by supplying gas independently or conjointly to the various inlets. Producer or coke-oven gas may be used as the fuel gas.

H. E. BLAYDEN.

**Gas producers.** C. WHITFIELD (B.P. 342,646, 3.8.29 and 1.1.30).—The grate of a gas producer is constructed so that the quantity of air supplied to different parts of it is separately controlled, e.g., by altering the openings to a number of independent air chambers built under various sections of the fuel bed.

R. H. GRIFFITH.

**Gas producer plant.** W. CLIMIE and J. E. DUNLOP (B.P. 343,065, 13.8.29).—A cylindrical, vertical gas producer is fitted with a rotary cover plate which carries a circular toothed rake resting on the top of the fuel. This rake is supported by tubes through which water circulates, and is slidable in sleeve tubes and water-cooled; differences in the level of the fuel bed are thus allowed for. Fresh fuel is supplied by a wide central duct associated with a hopper, and water-seals are provided for the rotary cover and for the mechanism which admits fresh fuel.

R. H. GRIFFITH.

**Manufacture of producer gas.** J. U. MACDONALD (U.S.P. 1,771,133, 22.7.30. Appl., 15.1.23).—The producer consists of a suitable refractory chamber enclosing

a vertical column of coke through which the air for combustion passes downwards either from ports at the top of the producer or from an inlet pipe communicating with a heat exchanger. The resulting gases pass through a centrally placed, vertical outlet pipe at the bottom of the producer. The outlet pipe may be rotated on a vertical axis to agitate the fuel, and is furnished with a water-seal. The lower part of the producer is provided with a water-sealed ash-pan and the coke column is maintained by gravity feed from a hopper. Provision is made for the introduction of steam or oil to the coke column.

H. E. BLAYDEN.

**Manufacture of water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of C. S. CHRISMAN (B.P. 344,205, 25.2.30. U.S., 6.5.29).—In a process for the complete gasification of coal, the whole of the water-gas is made by steaming in an upward direction, and its sensible heat is all available for the treatment of uncarbonised coal. This is made possible, without excessive cooling of the bottom of the generator, by admitting the steam through a central tuyère at an appreciable distance above the base of the fuel bed. During the blow period air is admitted at the fire bars and also through the projecting tuyère, so that the fuel bed is heated through a larger zone than is normally the case.

R. H. GRIFFITH.

**Production of a mixed gas containing carbon monoxide and hydrogen.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 343,160, 15.11.29. Ger., 15.11.28).—A generator for the production of mixtures of carbon monoxide and hydrogen consists of a column of coke placed between the poles of a high-frequency electric generator to which steam is supplied. The composition of the resulting gas can be controlled by varying the current frequency and voltage, and also by the quantity of steam added; the amount of carbon monoxide formed is appreciably less than that produced in a normal water-gas plant.

R. H. GRIFFITH.

**Manufacture of [coal] gas.** HUMPHREYS & GLASGOW, LTD., and A. R. GRIGGS (B.P. 342,958, 8.4.30).—In a system for the complete gasification of coal, the carbonisation section is surrounded by a recuperator, which is connected to the gasification section by pipes and valves. The recuperator is heated by burning blow-gases in it, and then serves to superheat steam, passed in a reverse direction, on its way to the fuel bed. An additional external heat-storage vessel may be used as a carburettor or, if this is not required, as a further source of superheated steam and for preheating circulation gases.

R. H. GRIFFITH.

**Recovery of hydrogen from gas mixtures containing hydrocarbons.** GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 317,731, 19.8.29. Ger., 20.8.28).—Gases containing hydrocarbons, such as coal gas, are used for the production of hydrogen by a combined process of catalysis and refrigeration. Initial purification and condensation remove ammonia, hydrogen sulphide, benzol, etc., and a further cooling operation yields a mixture of liquid methane and its homologues. This is expanded, mixed with 4 vols of steam, and passed over a nickel-alumina catalyst at 500°; 75% of the gas is converted into hydrogen and carbon dioxide.

After removal of the latter, the unchanged hydrocarbon is separated by further cooling and returned to the catalyst vessel; pure hydrogen is drawn off from the refrigerating plant.

R. H. GRIFFITH.

**Removal of ammonia and hydrogen sulphide from gases.** C. J. HANSEN, Assr. to H. KOPPERS A.-G. (B.P. 343,493, 19.11.29. Ger., 10.1.29).—Gases containing these impurities are cooled, and then scrubbed with a solution containing enough metal thiosulphate to react with all the ammonia to form ammonium thiosulphate and metal sulphide. The surplus hydrogen sulphide is next removed by a further quantity of the solution, precipitation of the dissolved metal as sulphide being completed by the addition of aqueous ammonia; the necessary ammonia is obtained by the primary cooling of the crude gases. The metal sulphide, e.g., iron sulphide, which is formed in the process is withdrawn and treated with sulphurous acid for the regeneration of a fresh scrubbing solution, and ammonium salts which remain in the spent liquor are converted into sulphate.

R. H. GRIFFITH.

**Production of a gas-purifying material.** E. J. MURPHY, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,771,136, 22.7.30. Appl., 6.12.23. Renewed 26.12.29).—The efficiency of iron oxide purifying materials (e.g., iron oxide ores) is increased by crushing the materials to pass 100- or 200-mesh and heating them at 200—400°. Hydrogen sulphide is removed from gas by passing the gas through the 100-mesh material or through an aqueous suspension of the 200-mesh material (e.g., containing 3% of the treated material).

H. E. BLAYDEN.

**Purification of gases.** E. C. UHLIG and E. J. MURPHY, Assrs. to BARTLETT HAYWARD Co. (U.S.P. 1,771,153, 22.7.30. Appl., 6.12.23).—Hydrogen sulphide is removed from gases by passing them upwards through a mechanical scrubbing tower against a countercurrent of a continuously agitated suspension of prepared iron oxide (cf. U.S.P. 1,771,136, preceding abstract). The used scrubbing liquid is revived by passage down a tower through which a current of air ascends. The temperature within the revivifying tower may be varied, but is approx. 71°. The revived liquid is returned to the scrubbing tower.

H. E. BLAYDEN.

**Apparatus for indicating the presence of inflammable vapours or gases.** H. T. RINGROSE (B.P. 344,287, 9.5.30).—The combustion of inflammable gases which diffuse through the porous walls of an apparatus containing an ignition filament can be observed through glass panels fitted to the side of the vessel. A visual check of a roughly quantitative kind depending on the intensity of the glow is thus provided for the movement of any indicator attached to the instrument.

R. H. GRIFFITH.

**Aqueous dispersions of bituminous material.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 343,427, 9.10.29).—Substances such as asphalt, pitch, etc. are mixed with a dispersing agent which is suspended in water to yield a stable emulsion of the bituminous material. For this purpose bentonite or similar clayey material is added in amounts less than 10% by wt. of the asphalt, at a temperature about

75° and preferably not much above the m.p. of the bituminous substance. The thick suspension which is formed is then beaten mechanically in order to reduce its viscosity. R. H. GRIFFITH.

**Pitch composition.** BARRETT CO., Assees. of C. R. ECKERT (B.P. 343,265, 13.1.30. U.S., 23.1.29).—Mica is ground to pass 60—100-mesh and added to pitch or asphalt for use in waterproofing compositions. The best results are given with 3—10 wt.-% of mica, and the product does not flow when raised considerably above the m.p. of the untreated pitch. It is also free from brittleness at low temperatures, and thus more resistant to extreme weather conditions. R. H. GRIFFITH.

**Pyrolysis of hydrocarbons.** IMPERIAL CHEM. INDUSTRIES, LTD., and D. BINNIE (B.P. 343,881, 12.11.29).—Gases which are to be used as a source of acetylene are diluted with a water-soluble gas or vapour before being rapidly passed through a furnace at 1400—1450°. A substance such as hydrogen chloride is useful for this purpose, and reacts only slightly with the other gases present; the yield of acetylene is appreciably increased by this method, and the diluent gas is easily removed from the products by washing with water. R. H. GRIFFITH.

**Decomposition, under the action of heat, of a mixture of methane or other hydrocarbons and water vapour.** UNION CHIM. BELGE, SOC. ANON. (B.P. 343,172, 3.9.29. Belg., 22.6.29. Cf. B.P. 341,393; B., 1931, 331).—A mixture of carbon monoxide and hydrogen is obtained by the interaction of methane, or a similar hydrocarbon, with steam in a hot chamber. The process is carried on intermittently, as the necessary heat is supplied by burning some of the hydrocarbon gas in the reaction vessel. Two recuperators are also provided, which are heated by escaping gases and in which air for combustion, or the mixture to be decomposed, can be preheated. R. H. GRIFFITH.

**Motor fuel.** M. MÜLLER-CUNRADI and W. WILKE, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,765,692, 24.6.30. Appl., 21.10.24. Ger., 14.1.24).—Knocking in engines using liquid fuel is prevented by additions of 0.1—1% of iron acetylacetonate to the fuel. H. E. BLAYDEN.

**Storage of [volatile] organic liquids.** R. LANT (B.P. 342,742, 9.8.29).—Such liquids from storage of which fire hazards may arise are kept in vessels containing a highly absorbent material which prevents accidental escape of the fuel. A higher fatty acid ester of cellulose, or a simple derivative of it, which is insoluble in the organic liquid, is best for this purpose. R. H. GRIFFITH.

**Vertical gas retorts and extractors therefor.** E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 344,860, 12.12.29).

**Gas producers [for gas engines].** R. F. CLAYTON and D. J. SMITH (B.P. 343,466, 18.11.29).

**Heating viscous liquids. Extracting liquid [from coal etc.]. Dryers. Crushers. Jigs. Petrol filter. Purification of liquids. Gas analysis. Testing gases.**—See I. Hydrogen-nitrogen.—See VII. Asphalt filler. Roads.—See IX. Fatty acids from wax.—See XII. Fireproof paste.—See XIII.

### III.—ORGANIC INTERMEDIATES.

#### Analysis of technical solvents. II. Colour tests for benzene, nitrobenzene, toluene, and xylene.

H. H. WEBER (Chem.-Ztg., 1931, 55, 201—203; cf. B., 1930, 232).—The colour reactions of polynitro-derivatives of benzene and its homologues with alkali and certain higher alcohols (cf. Hantzsch and Picton, A., 1909, i, 467; van Urk, A., 1924, ii, 356) are sufficiently characteristic to enable these hydrocarbons to be detected in presence of each other and of other solvents. The test suspension is prepared by shaking the solvent (0.5—5.0 c.c.) with nitric acid (1 c.c.) and pure sulphuric acid (2 c.c.) and adding 0.1 c.c. of the acid layer to 1 c.c. of water. The suspension (0.5 c.c.) with isoamyl alcohol (1 c.c.) is neutralised with 2*N*-sodium hydroxide and shaken with sodium hydroxide (1—2 g.). A blue or violet coloration on subsequent addition of acetone indicates benzene or nitrobenzene. When the suspension (0.5 c.c.) with benzyl alcohol (1 c.c.) is made alkaline with 2*N*-sodium hydroxide and shaken, toluene is detected by the appearance of a reddish-brown colour in the alcohol layer. Similarly, using cyclohexanol, *m*-xylene gives an intense green colour. The smallest proportion of each hydrocarbon which can be detected in presence of the others has been determined. Higher homologues do not interfere. II. E. F. NOTTON.

**Phenol.**—See II. Acetic acid.—See V. Honey acids.—See XIX.

#### PATENTS.

**Manufacture of organic halogen compounds [methylene dichloride].** H. DREYFUS (B.P. 341,878, 19.7.29).—Methyl alcohol is treated with sulphur chloride and chlorine at 60—100° in a closed vessel to give methylene dichloride. Other alcohols and other halides or oxyhalides of sulphur or phosphorus may be used, and the halogen may be omitted if desired. Variation in proportions or conditions leads to other di- and polychloro-compounds. C. HOLLINS.

**Manufacture of alkylene derivatives [glycols, glycol esters and ethers] from alkylene oxides.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,022, 11.11.29).—An alkylene oxide is led continuously with water, an alcohol, or a carboxylic acid through a hot tube, e.g., at 180°/30 atm., without a catalyst. The preparation of glycol monoethyl ether (from ethylene oxide and ethyl alcohol), propylene glycol monomethyl ether, ethylene glycol, glycol monoacetate, glycol monophenyl ether, and  $\gamma$ -chloropropylene glycol monomethyl ether (from epichlorohydrin) is described, together with a suitable apparatus. C. HOLLINS.

**Manufacture of lactic acid or its esters.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. R. H. HURTLEY (B.P. 341,961, 25.10.29 and 12.2.30).—The condensation of acetaldehyde with hydrogen cyanide is effected below 60° in presence of an alcohol and/or a lactic ester together with the necessary amount of water. Hydrogen chloride (or other mineral acid) is then added, and hydrolysis to lactic acid or hydrolysis and esterification to the lactic ester is carried out at 60—100°. C. HOLLINS.

**Production and application [as wetting agents] of sulphonated amides and the like.** H. T. BÖHME

A.-G. (B.P. 318,542, 19.7.29. Ger., 5.9.28).—The amides, anilides, or other *N*-substituted amides, of hydroxylated or unsaturated fatty acids above  $C_6$  are sulphonated, *e.g.*, with sulphuric acid, oleum, or chloro-sulphonic acid, in presence or absence of anhydrous organic acids, acid anhydrides, or acid chlorides, to give wetting or dispersing agents. The products are useful as addenda to liquids or plastics for treatment of textiles, leather, and paper, as softening agents for textiles and leather, as auxiliary agents in dyeing (enabling weak alkali to be used in vat dyeing or in padding for ice colours), in spinning, in ink manufacture, in fat splitting, as dust-binding agents, and as addenda to boring oils and to ceramic masses. C. HOLLINS.

**Manufacture of derivatives of 3-nitro-4-hydroxybenzamide.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 341,970, 25.10.29, 15.11.29, and 9.7.30).—A 3-nitro-4-acyloxybenzoyl halide is condensed with a primary or secondary amine carrying substituents suitable for azo dye formation, or with an aminoazo compound. The acyloxy-group is hydrolysed (usually during condensation); the 3-nitro-group may subsequently be reduced. 3-Nitro-4-acetoxybenzoyl chloride, m.p. 55°, with *m*-phenylenediamine-4-sulphonic acid gives 3-nitro- and, after reduction, 3-amino-4-hydroxybenzoic 3-amino-4-sulphoanilide. 3-Nitro-4-acetoxybenzoyl chloride is similarly condensed with 2:5-diamino-4'-hydroxy-3'-carboxydiphenylsulphone, benzidine-3-sulphonic acid, H-acid, J-acid,  $\gamma$ -acid, S-acid, *N*-ethyl-H-acid, *m*-aminobenzoyl-J-acid, 2-nitro-4-amino-phenol-6-sulphonic acid, 3-nitro-5-aminosalicylic acid, 2-nitro-*p*-toluidine, mono-oxalyl-*m*-phenylenediamine, 5-aminosalicylic acid, 5-*p*-amino benzamido-3-sulphosalicylic acid, and 4-amino-4'-hydroxyazo-benzene-3'-carboxylic acid. C. HOLLINS.

**Manufacture of [3:4:6-trisubstituted] aniline-o-sulphonic acids.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 341,612, 13.11.29).—Anilines carrying in positions 3, 4, and 6 alkyl, alkoxyl, aryloxy-, aralkyloxy-, aroyl, arylsulphonyl, nitro-, sulphonic, carboxylic, or halogen substituents are sulphonated with chlorosulphonic acid, preferably in a solvent (carbon tetrachloride, *o*-dichlorobenzene, nitrobenzene, tetrachloroethane), in absence of moisture. The sulphonation of a variety of such amines is described. C. HOLLINS.

**Manufacture of sulphonic acids of 2-halogeno-3-chloro-4-amino-1-methylbenzene [2-halogeno-3-chloro-*p*-toluidines] and separation into their constituents of mixtures of 2:3- and 2:5-halogenochloro-4-amino-1-methylbenzene [-halogenochloro-*p*-toluidines].** I. G. FARBENIND. A.-G. (B.P. 318,837, 9.9.29. Ger., 7.9.28).—2-Halogeno-3-chloro-*p*-toluidines are sulphonated with sulphuric acid at 180–200° or with weak oleum at 100–130° to give the 5-sulphonic acids, or with excess of high-strength oleum to give the 6-isomeride, separable from the small amount of 5-sulphonic acid also formed by dissolution in water containing sodium chloride. 2-Halogeno-5-chloro-*p*-toluidines are not sulphonated under these conditions and sulphonation may be used to remove the 2:3- from the 2:5-isomeride. The 5-sulphonic group is readily eliminated by heating with 80% sul-

phuric acid. The sulphonation of 2:3-dichloro- and 3-chloro-2-bromo-*p*-toluidines, and the separation of 2:3- and 2:5-dichloro-*p*-toluidines, are described. Pure 2:3-dichloro-*p*-toluidine has m.p. 55–58°.

C. HOLLINS.

**Manufacture of benzoic acid [from phthalic anhydride].** H. E. POTTS. From MONSANTO CHEM. WORKS (B.P. 341,902, 21.8.29).—Molten phthalic anhydride below 350°, preferably at 220–285°, is treated with steam in presence of a decarboxylating catalyst, *e.g.*, chromic and sodium phthalates. The benzoic acid may be distilled off in steam at intervals (cooling to 140–170°) or continuously. C. HOLLINS.

**Manufacture of derivatives of carbazole.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 341,905, 14.9.29).—The process of B.P. 320,641 (B., 1930, 233) is extended to the preparation of other carbazole derivatives by removal of sulphonic groups from substituted carbazolesulphonic acids by heating with water or dilute acid under pressure. 4:6-Dihydroxycarbazole (*N* = 5), m.p. 211–212°, is obtained from its 2:8-disulphonic acid; 4-amino-8-hydroxycarbazole, m.p. 215–216°, from its 2:8-disulphonic acid; 4-chlorocarbazole, m.p. 125°, 4-bromo- and 4-iodo-carbazoles, from their 2:6:8-trisulphonic acids; 2-aminocarbazole, m.p. 258°, from its 4:8:6-trisulphonic acid; 4:4'-dicarbazolyl sulphide, m.p. 228–230°, from its 2:2':6:6':8:8'-hexasulphonic acid (product gives a blue indophenol with *p*-nitrosophenol); 2-hydroxycarbazole from its 4:6:8-trisulphonic acid; 4-hydroxycarbazole-6-carboxylic acid, m.p. 284–285°, from its 2:8-disulphonic acid; 3-hydroxycarbazole, m.p. 275–276°, from its 8(?)sulphonic acid; and 4-hydroxycarbazole from its 2:7-disulphonic acid. C. HOLLINS.

**Hydrogenation of pyridine, quinoline, and homologues thereof.** SCHERING-KAHLBAUM A.-G. (B.P. 342,010, 5.11.29. Ger., 29.11.28).—Pyridine and quinolines are hydrogenated in presence of nickel by nascent hydrogen provided by cyclohexanol, tetrahydronaphthalene, or other suitable hydrogenated compound; the latter may be produced or regenerated *in situ* by introducing hydrogen. Pyridine and cyclohexanol (3 mols.) at 210–230° give piperidine and cyclohexanone. Piperidine is also produced by hydrogenating pyridine in presence of 10% of tetrahydronaphthalene or cyclohexanol at 160–180°/50 atm. A mixture of pyridine and phenol absorbs hydrogen at 120°, with production of cyclohexanol; on now raising the temperature to 160–180° piperidine and cyclohexanone are obtained.

C. HOLLINS.

**Manufacture of menthenes.** SCHERING-KAHLBAUM A.-G. (B.P. 341,997, 31.10.29. Ger., 15.11.28).—Menthadienes having one of the double linkings semicyclic or in a side-chain are reduced to menthenes by partial hydrogenation in presence of nickel at 40–80°. The preparation of  $\Delta^3$ -menthene from  $\beta$ -terpinene,  $\Delta^1$ -menthene from sylvestrene,  $\Delta^2$ -menthene from  $\beta$ -phellandrene, and carvomenthene from dipentene, is described.

C. HOLLINS.

**Manufacture of homogeneous halogen derivatives of 1-chloronaphthalene.** I. G. FARBENIND.

A.-G. (B.P. 341,926, 23.10.29. Ger., 24.10.28).—1-Chloronaphthalene-8-sulphonic acid is converted by chlorine at 25° into 1:4-dichloronaphthalene-8-sulphonic acid, from which 1:4-dichloronaphthalene, m.p. 71—72°, is obtained by hydrolysis with 50% sulphuric acid. At 90—100° 1:4:8-trichloronaphthalene, m.p. 130°, is produced. Bromination of 1-chloronaphthalene-8-sulphonic acid at 15° gives 1-chloro-8-bromonaphthalene, m.p. 87—88°, and 1-chloro-4-bromonaphthalene-8-sulphonic acid, from which 1-chloro-4-bromonaphthalene, m.p. 66—67°, is obtained by hydrolysis, or 1-chloro-4:8-dibromonaphthalene, m.p. 96—97°, and 1:8-dichloro-4-bromonaphthalene, m.p. 114—115°, by appropriate halogenation. 1:4-Dichloronaphthalene-8-sulphonic acid gives 1:4-dichloro-8-bromonaphthalene, m.p. 112—113°, on bromination. C. HOLLINS.

**Manufacture of homologues of anthraquinone.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,553, 19.8.29. Addn. to B.P. 327,128; B., 1930, 706).—The products from *p*-benzoquinone and two different  $\alpha\gamma$ -butadienes, or their isomerides, or the hexa- and tetra-hydroanthraquinones obtained from these, are converted into anthraquinones by ferric chloride (etc.) in acid medium. The preparation of 2-methyl- and 2:3-dimethyl-anthraquinones is described. (Cf. B.P. 340,509; B., 1931, 336.) C. HOLLINS.

**Volatile liquids.**—See II. **Heterocyclic compounds.**—See XX. **Products for destroying animals.**—See XXIII.

#### IV.—DYESTUFFS.

##### PATENTS.

**Manufacture of vat dyes of the benzanthrone series.** I. G. FARBENIND. A.-G. (B.P. 341,400, 4.10.29. Ger., 26.10.28. Addn. to B.P. 305,679; B., 1930, 755).—The process of the prior patent is applied to  $\alpha$ -1-benzanthronylaminoanthraquinones having in the anthraquinone residue an aliphatic acylamino-group and a free 2-position. 4-Acetamido-1-(2-benzanthronylamino)anthraquinone is converted by chlorosulphonic acid at 20—25° into a yellow-green vat dye. The 4-benzamido-compound gives a similar product, from which the benzoyl group may be removed by hydrolysis, leaving a dark green vat dye (grey after hypochlorite treatment); elimination of the amino-group from this product gives a dark green vat dye fast to chlorine. C. HOLLINS.

**Production and use of benzanthrone derivatives [vat dyes of the dibenzanthrone series].** I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 341,389, 5.7.29).—3:3'-Dibenzanthronyl is nitrated with mixed acid, and the resulting mono- or di-nitro-compound is reduced in acid medium, e.g., with zinc dust and aniline sulphate at 140—180°, to give mono- or di-aminodibenzanthrone. Nitrated 4:4'-dibenzanthronyl is similarly reduced to a vat dye. C. HOLLINS.

**Manufacture of a vat dye [from 2-thiol-1-methyl-anthraquinone].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,432, 15.7.29).—2-Thiol-1-methyl-anthraquinone gives an orange-yellow vat dye when

heated with sulphur at 250—260°. The same product is obtained by heating 2-chloro-1-methylantraquinone with sodium sulphide and sulphur at 250—260°. [Stat. ref.] C. HOLLINS.

**Manufacture of monoazo dyes [for lakes].** MAJOR & CO., LTD., H. H. HINCILIFFE, and W. J. DARBY (B.P. 341,969, 25.10.29).—An arylaminesulphonic acid, e.g., *p*-toluidine-3-sulphonic acid, is diazotised and coupled in alkaline solution with the product obtained from 2:3-hydroxynaphthoic acid and toluene-*p*-sulphonyl chloride. C. HOLLINS.

**Manufacture of monoazo [acid] dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 341,397 and 341,461, [A] 11.9.29, [B] 16.9.29).—(A) 4-Aminoacetanilide-3-sulphon-alkyl- or -dialkyl- or -arylalkyl-amides are diazotised and coupled with a  $\beta$ -naphthylaminesulphonic acid. The same dyes are obtained by diazotising the corresponding 4-nitroaniline-2-sulphonamides, coupling with a  $\beta$ -naphthylaminesulphonic acid, reducing the nitro-group, and acylating. Examples are: 4-nitroaniline-2-sulphonethylanilide  $\rightarrow$   $\gamma$ -acid, reduced, acetylated (bluish-red); 4-aminoacetanilide-3-sulphonethylanilide  $\rightarrow$   $\gamma$ -acid (bluish-red), *N*-methyl- $\gamma$ -acid (red-violet), or 2:7-naphthylaminesulphonic acid (red). (B) The *N*-chloroacetyl (or other halogeno-acyl) derivatives of aminonaphtholsulphonic acids are used as coupling components for monoazo acid dyes fast to fulling. Examples are: *N*-chloroacetyl-*H*- or -*K*-acid with diazotised aniline, *o*- and *p*-toluidines, *m*-aminobenzaldehyde, 2-amino-4-acetethylamidotoluene; *N*-chloroacetyl- $\gamma$ -acid or *N*- $\beta$ -chloropropionyl-*H*-acid with diazotised aniline. C. HOLLINS.

**Manufacture of azo-dye components [hydroxynaphthylguanidines].** KALLE & CO. A.-G. (B.P. 341,549, 19.10.29. Ger., 19.10.28).—Coupling components specially suitable for diazo-types are obtained by condensing an aminonaphthol or its sulphonic acid with cyanamide or dicyanodiamide. 1:7-Aminonaphthol and cyanamide yield 7-hydroxy-1-naphthylguanidine, m.p. 225—226° (decomp.) (hydrochloride, m.p. 113—114°). 5-Hydroxy-1-naphthylidiguanidine, m.p. 143—144° (decomp.) (hydrochloride, m.p. 216—217°), the 7:1-isomeride [hydrochloride, m.p. 235—237° (decomp.)], and the 7:2-isomeride (hydrochloride, m.p. 218—219°) are similarly prepared from dicyanodiamide. C. HOLLINS.

**Manufacture of [direct dis- and tris-]azo dyes.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & CO. (B.P. 341,898, 18.7.29).—A non-phenolic, sulphonated arylamine or aminoazo compound is diazotised and coupled with a non-phenolic arylamine; the product is condensed with a nitrobenzoyl chloride, reduced, diazotised, and coupled with a non-phenolic arylamine; the resulting dis- or tris-azo dye is acylated, e.g., with benzoyl or *p*-nitrobenzoyl chloride; in the latter case the nitro-group may subsequently be reduced. Examples are: 2-naphthylamine-6:8-disulphonic acid  $\rightarrow$  *m*-toluidine or Cleve acid, *p*-nitrobenzoylated, reduced,  $\rightarrow$  *m*-toluidine, benzoylated (bright yellow), or acetylated (redder), or *p*-aminobenzoylated (developed with  $\beta$ -naphthol, reddish-yellow); sulphanilic acid  $\rightarrow$  *m*-toluidine, *p*-nitrobenzoylated, reduced,  $\rightarrow$  Cleve acid,

*p*-aminobenzoylated (yellowish-orange; developed with  $\beta$ -naphthol, orange-brown). C. HOLLINS.

**Derivations of 3-nitro-4-hydroxybenzamide.**—See III.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Flax wax and its extraction.** W. H. GIBSON (Inst. Chem. Eng. Advance proof, Mar., 1931, 3—6).—The "pouce" or fine dust separated from flax fibre by combing and drawing may contain 6—10% of wax resembling beeswax. This dust may be pressed into blocks and extracted by solvents to give a residue suitable for insulating or building materials and a wax which may be used for polishes. This residue contains also about 1.4% N, and may have manurial value.

T. McLACHLAN.

**Denaturation of wool by carbamide.** W. RAMSDEN (Nature, 1931, 127, 403—404).—The apparently typical thiol reaction obtained with a carbamide extract of sheep's wool (A., 1930, 1604) is due to a reaction between carbamide and a derivative of sodium nitroprusside which is formed on keeping a solution of the latter. The reaction occurs only in solutions which are not too strongly alkaline and in the absence of ammonia, which changes the red colour to yellow. Saturated solutions of carbamide reveal a sulphide in wool when they are alkaline above a certain degree.

L. S. THEOBALD.

**Wood chemistry. VI. Proximate compositions of two new pulp woods.** Y. UYEDA and H. SHŌZI (J. Cellulose Inst., Tokyo, 1931, 7, 16—17; cf. B., 1929, 552).—The woods "*Doronoki*" (*Populus Maximowiczii*) and "*Ezoyanagi*" (*Salix rorido*), which may be of use in making paper pulp, have the following percentage compositions, respectively: loss on drying 16.33, 10.74; benzene extract 0.96, 1.48; alcohol extract 1.04, 1.78; cellulose 53.79, 55.07; lignin 21.05, 27.44; soluble pentosans 4.01, 2.62; galactans 0, 0.52; ash 1.48, 0.87; and pentosans 16.47, 13.74. Their cellulose and lignin contents are similar to those of other pulp woods used industrially, but they differ from the latter in their deficiency in mannans.

B. P. RIDGE.

**Change in lignin content of spruce wood according to climatic conditions.** P. KLASON (Cellulosechem., 1931, 12, 36—37).—Lignin determinations on 222 samples of spruce wood from different parts of Sweden show that the average lignin content is 28.1% (max. 28.4%, min. 27.8%), and that climatic conditions have no apparent effect. Wood from the trunk contains 28.2% of lignin, and from the branches 27.9%. A very tall tree examined throughout its length was found to contain 27.1—27.9% of lignin. The age of the tree has no influence on the lignin content of the wood.

T. T. POTTS.

**Purified wood fibre. Paper-making characteristics of wood fibre high in  $\alpha$ -cellulose.** G. A. RICHTER (Ind. Eng. Chem., 1931, 23, 266—272).—The relationship between the time of beating, the degree of hydration of the cellulose, and the strength of the paper produced is shown graphically for a number of types of wood pulp and various purified wood-fibre products.

Wood fibre containing a high percentage of  $\alpha$ -cellulose hydrates more slowly than ordinary sulphite pulp and forms a paper with increased tearing resistance and folding endurance, in this respect resembling that from the better grades of rag stock. Examination of commercial papers shows that purified wood fibre and rag papers are both superior to others in strength and toughness, but the presence of purified wood pulp imparts weakness to the paper. F. R. ENNOS.

**Solubility and physical strength of cellulose derivatives in relation to constitution.** M. HAGEDORN and P. MÖLLER (Cellulosechem., 1931, 12, 29—33).—Esters prepared by methods involving minimum effect on micelle size have been employed. It is confirmed that tri-esters are the highest form obtainable. The simple esters are all insoluble in water, but may exhibit slight swelling, or, in films, shrinkage or expansion. Water-resistance increases with carbon content. Excepting the formate, all esters are soluble in chlorinated hydrocarbons. The behaviour towards other solvents is shown to be largely dependent on the nature of the acid radical of the ester; also solubility of a given ester increases for solvents in the order alcohol, ether, ketone, ester. Cellulose esters are more soluble in chlorinated hydrocarbons than in the hydrocarbons themselves. The solubility of mixed esters is determined to some degree by the solubilities of the simple esters of the acids used. Strength of ester films decreases with increasing length of chain of the esterifying acid, stretch increasing. The cellulose esters of unsaturated acids do not conform to classification to the same degree as do those of the saturated acids. Ethers formed by the treatment of alkali-cellulose with alkyl chlorides all have solubilities, strengths, and elongations of the same order.

T. T. POTTS.

**Solubility of nitrocellulose.** M. UCHIDA (J. Cellulose Inst., Tokyo, 1930, 6, 253—263).—The solubility in ether-alcohol of 5 varieties of nitrocellulose has been determined for the temperature range  $-20^{\circ}$  to  $30^{\circ}$  and the nitrogen contents of the dissolved and undissolved parts have been found. The following conclusions are drawn. Sample A (13.23% N): The solubility increases with falling temperature and the maximum for the solvent containing ether-alcohol = 2:1 is twice as great at  $-20^{\circ}$  as at the ordinary temperature. Solvent of this composition gives maximum solubility at any temperature. Variation of solubility with temperature is greatest for the solvent giving maximum solubility. The nitrogen content of the dissolved part increases with falling temperature, and it is inferred that guncotton begins to dissolve at  $0^{\circ}$ . Sample B (12.69% N): Dissolution is the better at the lower temperature; its solubility in solvents containing 14—50% of alcohol varies from 60 to 100%, whilst the nitrogen content of the dissolved part is constant. Samples C and D (12.24 and 11.50% N, respectively) are very soluble and the solubility appears to increase as the temperature falls; the former is the more soluble in ether-rich, and the latter in alcohol-rich, solvents. The solubility of sample E (10.97% N) increases with rise in temperature, and a 1:1 mixture (by vol.) of ether and alcohol is a very good solvent.

B. P. RIDGE.



**Acetic acid recovery in the cellulose acetate industry.** H. R. S. CLOTWORTHY (Ind. Chemist, 1931, 7, 111—114).—The acid may be recovered in the form of glacial acetic acid (i) by conversion first into acetates, (ii) by extraction with an immiscible solvent. The first method is uneconomical, but the second gives successful results if a solvent having a b.p. above that of acetic acid is used. Two examples of this latter type of process are described, viz., the Suida process and that devised by Les Distilleries des Deux-Sèvres (abstracts of which have already appeared). These processes yield acid of 99·8—99·9% concentration. For reabsorption into the cycle of operations in the cellulose acetate factory, a part of the glacial acid is converted by known methods into acetic anhydride, any dilute acid formed being returned to the plant for concentration.

F. R. ENNOS.

**Determination of alkali-cellulose.** O. SCHWARZKOPF (Cellulosechem., 1931, 12, 33—36).—A mathematical analysis of the direct method of determination of alkali in the products of mercerisation.

T. T. POTTS.

**Factors during spinning which influence the physical properties of rayon.** II. P. C. SCHERER, JUN., and R. E. HUSSEY (Ind. Eng. Chem., 1931, 23, 297; cf. B., 1930, 812).—A maximum in the physical properties (tensile strength and elongation) of viscose rayon occurs at some definite temperature of the spinning bath for each time of contact with the bath. F. R. ENNOS.

**Detection of strained viscose silk.** A. J. HALL (Rayon Record, 1930, 4, 1347).—A new reagent for detecting strain in viscose threads, which is superior to that previously described (B., 1930, 1145) since it forms no precipitate when heated and can be used either neutral or alkaline, consists of glycerin 10 c.c., 20% ferric sulphate solution 5 c.c., 10% caustic soda solution 0—40 c.c., and water 85—45 c.c. Stretched viscose silk when immersed for a few minutes in the reagent at 100°, then rinsed with water, and immersed in dilute potassium ferricyanide acidified with sulphuric acid develops a blue colour which deepens as the alkalinity of the reagent is increased, but which is proportional to the degree of stretching. The reagent is equally suitable for detecting oxycellulose in overbleached cotton materials.

A. J. HALL.

**Paper mulch for soils.**—See XVI.

## PATENTS.

**Wet treatment [washing] of artificial silk [during manufacture].** GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 344,279, 29.4.30. Ger., 29.4.29).—Cakes of artificial silk yarn (produced by centrifugal spinning) are mounted on supports and arranged in vertical columns; water is led into the central space of each cake so that it rises and then flows over the top of each cake, thereby causing the cake to extend upwards and loosening the yarn so that the water can pass outwards through the cake itself.

A. J. HALL.

**Drum-type mill for paper and pulp manufacture.** U. KIRCHNER and G. STRECKER (B.P. 343,364, 27.3.30. Ger., 10.4.29).—In a mill comprising a drum rotating in one direction and internal mullers rotating at centri-

fugal speed in the opposite direction, the mullers are connected to each other and to the shaft by linkwork which ensures that when one muller is pressed inwards by a large or hard piece all the others follow suit. With such an arrangement the rotating system remains in balance and the centrifugal weight of all the mullers is available to crush a piece under one of them.

B. M. VENABLES.

**[Spinning funnels for] manufacture of artificial silk.** BRYSLKA, LTD., F. W. SCHUBERT, and W. A. ENTWISTLE (B.P. 344,560, 19.9.29. Addn. to B.P. 296,856; B., 1928, 852).

**Apparatus for manufacture of films and foils.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 344,485, 29.11.29).

**Suction chamber for machines for production of pulp sheets, and for removal of water from cellulose, or the like.** E. KRAMER (B.P. 345,023, 28.1.30. Czechoslov., 30.1.29).

**Heat insulation. Separating particles from water.**—See I.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Turkey-red dyeing.** L. R. PARKS (J. Physical Chem., 1931, 35, 488—510).—Data are recorded showing that sulphonated oils, and, to a slight extent, alumina, are adsorbed by cotton. Turkey-red oil and soap form colloidal solutions. Alumina adsorbs soap from solution and *vice versa*. In the absence of calcium the alizarin anion is adsorbed by alumina, whilst sodium sulphate, but not sodium chloride, tends to strip the dye from the alumina. Sodium alizarate and calcium acetate yield insoluble calcium alizarate, which is peptised by an excess of the former. Cotton adsorbs a soap-alumina colloid which is fixed on the fibre by boiling water. Alizarin "G" produces the fiery-red colours. The colour is brightened by boiling in distilled water, but the brilliancy of Turkey-red is produced by mordanting the dyed fibre with an alumina-soap mordant and heating. A quick method of mordanting with alumina is proposed. The results are discussed and interpreted in relation to the process of Turkey-red dyeing. L. S. THEOBALD.

**Alkali-cellulose. Detecting strain in silk.**—See V.

## PATENTS.

**Bleaching of cellulose products.** R. H. MCKEE, E. H. MORSE, and P. E. ROLLHAUS, Assrs. to PILOT LABORATORY, INC. (U.S.P. 1,767,543, 24.6.30. Appl., 15.2.28).—Cellulose artificial silk is bleached at slightly above ordinary temperatures without deterioration as regards strength, elasticity, and lustre by treatment with dilute aqueous solutions or soap emulsions of peroxidised organic acids or organic peroxides (e.g., benzoyl, phthalyl, acetyl, succinyl, maleic, and fumaric peracids or peroxides).

A. J. HALL.

**Coloration of textile and other materials [containing cellulose esters or ethers with leuco-vat dyes].** BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and R. C. STOREY (B.P. 341,408, 4.7.29).—Acetate silk etc. is dyed with the free leuco-compounds of vat or sulphide dyes or of aminoanthraquinones in

aqueous suspension, preferably from a bath having  $pH$  4–8, and the colour is developed by oxidation with air, black liquor, soap, and sodium perborate, and especially with soap and hydrogen peroxide. Examples are Ciba blue G, Caledon jade green, 1-benzamido-4-hydroxy-anthraquinone, indophenol from *p*-aminodimethylaniline and  $\alpha$ -naphthol.

C. HOLLINS.

**Printing of acetate silk or materials containing a cellulose ether or ester.** SOC. CHEM. IND. IN BASLE (B.P. 341,419, 14.10.29. Switz., 12.10.28).—Acetate silk etc. is printed with aminoanthraquinonesulphonic acids, *e.g.*, sulphonated 4-benzylamino-1-methylamino-anthraquinone (blue), 1-amino-4-*p*-acetamidoanilino-anthraquinone-2-sulphonic acid (blue), 1-amino-4-anilinoanthraquinone-3-sulphonic acid (blue), 4:8-diaminoanthrarufin-2-sulphonic acid (blue), 1:4-di-(2'-sulpho-*p*-toluidino)anthraquinone (green), 1-amino-4-hydroxyanthraquinone-5-sulphonic acid (red-violet), and 1-aminoanthraquinone-5-sulphonic acid (orange). These colours do not sublime during the process.

C. HOLLINS.

**Manufacture of [low-lustre] materials made of or containing organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 344,093, 12.12.29. U.S., 27.12.28. Addn. to B.P. 318,467; B., 1931, 346).—The lustre of cellulose acetate silk is reduced by precipitation of zinc sulphide therein; the zinc salt necessary is introduced into the silk in the presence of a swelling agent, or the solution of the zinc salt (*e.g.*, zinc chloride) is itself used also as a swelling agent.

A. J. HALL.

**Waterproofing of gut.** R. LANT and W. KORESKA (B.P. 343,117, 16.10.29).—Gut threads are impregnated with cellulose distearate dissolved in xylene.

A. J. HALL.

**Dyeing of regenerated cellulose materials.** E. I. DU PONT DE NEMOURS & Co. (B.P. 318,892, 27.8.29. U.S., 11.9.28).—See U.S.P. 1,755,119; B., 1930, 986.

**Dyeing and like machines.** C. S. BEDFORD (B.P. 344,587, 9.1.30).

**Wetting agents.**—See III. Dibenzanthrone vat. —See IV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Increasing the purity of common salt.** T. B. BRIGHTON and C. M. DICE (Ind. Eng. Chem., 1931, 23, 336–339).—The objectionable odour of some brands of crude salt is removable by heating to above  $210^{\circ}$ , but this treatment causes discoloration owing to oxidation of clay in the salt or charring of organic matter. By washing the salt with brine and then roasting at  $225^{\circ}$ , a pure white product is obtained. W. J. WRIGHT.

**Water-solubility of the phosphoric acid of calcium phosphates.** W. KLEBERGER (Z. Pflanz. Düng., 1931, 19A, 309–313).—The decomposition of monocalcium phosphate in concentrated superphosphate solutions (Stollenwerk, B., 1930, 710) was not confirmed.

A. G. POLLARD.

**Bauxite.** H. JORDT (Chem.-Ztg., 1931, 55, 211).—Alumina, relatively free from iron, may be obtained by heating certain types of bauxite with coal tar in a

graphite crucible and washing the ignited mass with dilute hydrochloric acid; contact with air or oxygen during the washing should be avoided. The residue is ignited in an oxidising atmosphere. The method of magnetic separation proposed by the U.S. Department of Commerce is considered useless.

H. F. GILLBE.

**Technical importance of hydrolytic decomposition of aluminium salts at higher temperatures.** J. Z. ZALESKI (Przemysl Chem., 1931, 15, 104–110).—Ammonium alum solution on being heated under pressure deposits  $(NH_4)_2SO_4 \cdot Al_2(SO_4)_3 \cdot 3Al_2O_3 \cdot 15H_2O$  as crystals which on further heating yield  $(NH_4)_2SO_4 \cdot 3Al_2(SO_4)_3 \cdot 10Al_2O_3 \cdot 56H_2O$ . Under optimal conditions of temperature and concentration 94% of the aluminium originally in solution is precipitated in 5 hrs. The addition of silica gel greatly accelerates the process, whilst precipitation is inhibited by sulphuric acid. The precipitate is readily filterable, and does not sinter on heating; it constitutes a convenient source of aluminium oxide for the manufacture of aluminium. Under analogous conditions aluminium chloride solution does not deposit basic salts, whilst aluminium sulphate gives 43% yields of a salt,  $2Al_2(SO_4)_3 \cdot 4Al_2O_3 \cdot 9H_2O$ . Ammonium iron alum is practically quantitatively converted into the salt  $(NH_4)_2SO_4 \cdot 2Fe_2(SO_4)_3 \cdot 3Fe_2O_3 \cdot 9H_2O$ , and potassium alum and ammonium chrome alum are similarly almost quantitatively precipitated, whilst potassium chrome alum does not under these conditions deposit basic salts.

R. TRUSZKOWSKI.

**Extraction of [pure] beryllium, caesium, and rubidium [oxides] from beryl.** C. JAMES, H. C. FOGG, and E. D. COUGHLIN (Ind. Eng. Chem., 1931, 23, 318–320).—The finely-ground raw material is fused with lime in a blast furnace. The resultant slag is ground, decomposed with sulphuric acid, heated to dehydrate the silica, treated with hot water, and the liquor filtered, the filtrate being concentrated so that, on cooling, potassium, caesium, and rubidium alums crystallise out. The mother-liquor is treated with ammonium sulphate and cooled to separate aluminium as ammonium alum, and iron is removed by oxidising the solution with, *e.g.*, potassium bromate or nitric acid, and carefully precipitating with dilute ammonia solution, the last traces of iron being eliminated by treatment with hydrogen sulphide under slight pressure. Beryllium is finally obtained as the basic carbonate by addition of ammonium hydroxide or carbonate, from which a pure white oxide is obtained on ignition. The mixed alums, as described above, on being fractionally crystallised accumulate the caesium and traces of rubidium in the soluble portion. These are separated by further fractionation and/or precipitation of caesium antimony chloride and crystallisation of rubidium acid tartrate.

W. J. WRIGHT.

**Storage of carbon monoxide.** O. H. WAGNER (Brennstoff-Chem., 1931, 12, 87–89).—The possibility of storing large quantities of carbon monoxide under pressure in steel cylinders containing a suitable absorbent has been investigated. No suitable liquid solvent could be found; for the best solvents, ether and pentane, Ostwald's coefficient (*i.e.*, the ratio of concentration of gas in liquid to that over liquid) amounted only to

0.5 at 100 atm. Under 40 atm. pressure a cylinder filled with activated charcoal took up 1.4 times the amount which could be compressed under the same pressure into the empty cylinder, but at 150 atm. and higher pressures the volume of the adsorbed gas was less than the volume of gas displaced by the charcoal. By the action of carbon monoxide under high pressure on solid cuprous chloride and bromide additive compounds ( $\text{Cu}_2\text{Hal}_2\cdot 2\text{CO}$ ) have been prepared. These can be utilised for the storage of the gas; thus, under 150 atm. pressure, a cylinder packed with solid cuprous chloride would take up 4 times the amount of carbon monoxide as would be compressed into the empty cylinder. To recover the gas at atmospheric pressure the compound must be heated to about  $50^\circ$ .

A. B. MANNING.

**Magnesia in minerals.**—See VIII. **Lead tungstate.**—See XIII.

## PATENTS.

**Manufacture of potassium nitrate.** LE NITRO-SEL SOC. ANON., and J. MARGOLES (B.P. 343,287, 23.1.30).—A potassium ore is caused to interact with saturated sodium nitrate solution, and the mixture oxidised by means of a peroxide, persulphate, or current of hot air. The solution, freed from its impurities, is evaporated at a high temperature, pure sodium nitrate being thus separated continuously; the solution, after crystallisation, yields potassium nitrate of 90% concentration.

W. J. WRIGHT.

**Apparatus for manufacture of acid phosphate.** T. J. STURTEVANT, Assr. to STURTEVANT MILL CO. (U.S.P. 1,773,287, 19.8.30. Appl., 18.5.27).—Devices for automatically moulding the mixture of sulphuric acid and phosphate rock into a solid porous block, for removing this block rapidly from the mould, and for feeding it into the disintegrator are claimed.

A. R. POWELL.

**Manufacture of barium carbonate and sodium sulphhydrate [hydrogen sulphide].** J. B. PIERCE, JUN., Assr. to BARIUM REDUCTION CORP. (U.S.P. 1,774,523, 2.9.30. Appl., 29.1.27).—Carbon dioxide is passed into a hot solution made by mixing equimolecular amounts of barium and sodium sulphides, each in 15–20% solution, until all the barium is precipitated as carbonate. The precipitate, after washing and drying, is a dense, white powder weighing 67 lb./ft.<sup>3</sup> (cf. U.S.P. 1,634,338; B., 1927, 777). The solution is evaporated for the recovery of sodium hydrogen sulphide.

A. R. POWELL.

**Production of thiocyanogen salts.** E. HENE (B.P. 343,178, 19.11.29. Ger., 19.11.28).—Metal sulphides, hydrosulphides, polysulphides, or thiocarbonates are heated above  $300^\circ$  with dicyanodiamide or thiourea, with or without sulphur.

W. J. WRIGHT.

**Utilisation of greensand.** W. VAUGHAN and W. M. BRUCE, Assrs. to PERMUTIT CO. (U.S.P. 1,774,533, 2.9.30. Appl., 3.6.27).—Waste greensand, finer than 50-mesh, is treated with 2 vols. of 20% hydrochloric acid and 1 vol. of concentrated sulphuric acid is added slowly with stirring until decomposition of the sand is complete. The gelatinous silica so obtained is separated from the acid solution, washed, and dissolved in sodium hydroxide solution to yield a solution of water-glass

(d 1.38), which may be used in the manufacture of water softeners. The acid solution is cooled to crystallise out potassium alum, and the mother-liquor is evaporated for a second crop of crystals. The alum is recrystallised for purification and the final mother-liquors are distilled for the recovery of the hydrochloric acid. The residual liquor deposits a third crop of alum, and the remaining solution is evaporated and heated to obtain a red pigment from its content of ferric sulphate.

A. R. POWELL.

**Manufacture of carrier bodies for catalytic agents.** VER. F. CHEM. U. MET. PROD. (B.P. 343,285, 22.1.30. Ger., 22.1.29).—Natural materials with a high content of silicic acid, e.g., kieselguhr, excluding base-exchange materials, are heated with silicate-forming metallic oxides or salts, the basic constituent being then removed by treatment with a mineral acid.

W. J. WRIGHT.

**Apparatus for subliming sulphur.** E. F. WHITE (U.S.P. 1,773,562, 19.8.30. Appl., 13.11.24).—A flat rectangular retort in which the sulphur is to be vaporised contains a pipe terminating in a nozzle just above the level of the liquid sulphur. A current of cold inert gas at high pressure is blown through this nozzle, so that the sulphur vapour is swept rapidly into a large, narrow, vertical condensing chamber in which flowers of sulphur are formed by the rapid cooling action of the large surface and the gas stream. The gas is compressed and returned to the nozzle continuously.

A. R. POWELL.

**Recovery of sulphur [from roaster gases].** R. C. BENNER and A. P. THOMPSON, Assrs. to GEN. CHEM. CO. (U.S.P. 1,771,480—1, 29.7.30. Appl., [A] 2.3.26, [B] 30.6.26).—(A) The roaster gases are introduced with coal into the upper part of a combustion chamber the temperature of which is maintained at  $700$ – $800^\circ$  by the combustion of part of the coal by the air in the gases; in this way the coal is suddenly converted into a light porous coke and the hydrocarbons evolved reduce the sulphur dioxide to sulphur, a process which is completed by passing the gas mixture through the bed of glowing coke at the lower part of this chamber. The issuing gases are mixed with a further quantity of roaster gas and passed over a bauxite catalyst at  $500^\circ$  to oxidise any unchanged hydrocarbons, hydrogen sulphide, etc. and thence to a condenser to recover sulphur. (B) The sulphur dioxide is reduced with gaseous hydrocarbons, carbon monoxide, or hydrogen in the presence of a bauxite catalyst heated at  $500^\circ$  (cf. U.S.P. 1,773,293—4; B., 1931, 295).

A. R. POWELL.

**Manufacture of phosphorus compounds.** VICTOR CHEM. WORKS, Asses. of H. F. NOYES, R. WEIGEL, and H. W. EASTERWOOD (B.P. 343,390, 22.4.30. U.S., 29.4.29).—The gases produced by the reduction of phosphorus ore in a blast furnace pass through a separator and dust remover into a rectangular combustion chamber. This chamber is connected to a number of equidistant regenerators, so that the conditions of distribution to these of the gases containing phosphorus pentoxide are the same. The gas flow is controlled so that as soon as one regenerator is heated to the desired temperature the gas is diverted to another.

W. J. WRIGHT.

**Production of mixtures comprising hydrogen and nitrogen.** H. RITTER (B.P. 343,054, 7.11.29. Ger., 9.11.28).—Steam and a gas containing hydrocarbons are passed over a nickel catalyst at 850°, the reaction taking place in an annular vessel of high-grade steel. The mixture is treated with preheated air and passed over a nickel catalyst at 950–1200°, reaction being effected without additional external heating. Complete conversion of methane into hydrogen and carbon monoxide is thus ensured, and the latter may be removed by any desired process. W. J. WRIGHT.

**Recovery of iodine [from natural brines].** C. W. GIRVIN, Assr. to GEN. SALT CO. (U.S.P. 1,774,882, 2.9.30. Appl., 8.2.27).—The brine is treated with a reagent which liberates iodine from the iodides in solution, the iodine is absorbed in charcoal, and the charcoal is treated with a solution of sodium hydrogen sulphite to convert its iodine content into hydriodic acid. Iodine is then regenerated from the acid solution by addition of oxidising agents and the purified charcoal is returned to the adsorption vessel. A. R. POWELL.

**Machines for handling superphosphate or like material.** A./S. DANSK SVOVLSYRE- & SUPERPHOSPHAT-FABRIK, Assees. of K. WARMING (B.P. 344,679, 24.3.30. Denm., 20.9.29).

**Crystallisation.** Flaker. Synthetic ammonia.—See I. Hydrogen chloride. Mixed gas. Hydrogen, sulphur, ammonia, and hydrogen sulphide from gases.—See II. [Acid-resisting] alloy.—See X. Zinc white.—See XIII. Manures.—See XVI.

## VIII.—GLASS; CERAMICS.

**Heat balances of some ceramic kilns.** H. H. MANSUR (J. Amer. Ceram. Soc., 1931, 14, 89–124).—Six kilns (round, downdraught, muffle, horizontal draught, semi-continuous, and continuous) were investigated. The details of instruments, methods, procedure, and results are recorded. J. A. SUGDEN.

**Mullite formations in ceramic bodies.** L. SOLODOVNIKOVA (Trans. Ceram. Res. Inst., Moscow, 1930, No. 25, 45–56).—The size and quantity of the mullite formations depend on the temperature, duration of firing, and porosity. Crystallographic data for mullite needles are given. CHEMICAL ABSTRACTS.

**Organic compounds as electrolytes and their effect on the properties of clay slip and on the life of plaster moulds.** C. W. PARMELEE and C. G. HARMAN (J. Amer. Ceram. Soc., 1931, 14, 139–147).—Piperidine, tetramethylammonium hydroxide, or ethylamine may replace the usual mixture of sodium silicate and carbonate as the casting-slip electrolyte, and do not possess the disadvantages of the latter, viz., adverse effect on the life of the moulds and possible formation of vitreous patches on the fired ware. The deflocculating power of the organic compounds decreases in the order given. Measurements of the viscosity and surface tension of several clay slips were made and also transverse strength tests of plaster. J. A. SUGDEN.

**Determination of the refractoriness of clay.** P. A. ZEMYATCHENSKI and F. A. ZENKOVICH (Trans. Ceram. Res. Inst., Moscow, 1930, No. 24, 1–29).—

When cones did not fuse, fluxes were added (ferric oxide and lime being the best) and the relative refractoriness was obtained by the use of tables.

## CHEMICAL ABSTRACTS.

**Kaolin as raw material for refractories.** E. KÖHLER (Trans. Ceram. Res. Inst., Moscow, 1930, No. 25, 1–44).—By firing at high temperatures, it is possible to prepare very resistant products which show no deformation at a pressure of 2 kg. per cm.<sup>2</sup> above 1550°.

## CHEMICAL ABSTRACTS.

**Volumetric determination of magnesia and alumina in minerals and refractory bricks.** H. ECKSTEIN (Chem.-Ztg., 1931, 55, 227).—For the determination of magnesia the filtrate from the ammonia precipitate in the ordinary course of analysis is boiled with 3–5 g. of ammonium chloride, 2 c.c. of ammonia, and 15 c.c. of 3% 8-hydroxyquinoline solution, the yellow precipitate is collected, washed with hot very dilute ammonia, redissolved in hydrochloric acid, and reprecipitated as before, the second precipitate is dissolved in dilute hydrochloric acid, and the solution titrated with 0.1N-potassium bromide-bromate, using a 1% solution of indigo-carmin as indicator (1 c.c. of 0.1N-KBr-KBrO<sub>3</sub> = 0.000504 g. MgO). Alumina may be determined in refractories by fusing 0.5 g. of the finely-ground material with 5 g. of fused borax, dissolving the fusion in dilute hydrochloric acid, diluting to 250 c.c., and treating 25 c.c. with ammonia to neutrality, a few drops of hydrochloric acid, 5 g. of sodium acetate, water to 100 c.c., and 3% 8-hydroxyquinoline solution in slight excess to precipitate the alumina. The washed precipitate is dissolved in hydrochloric acid and the solution titrated as for magnesia (1 c.c. = 0.000425 g. Al<sub>2</sub>O<sub>3</sub>). A. R. POWELL.

**Glass standards for varnish.**—See XIII.

## PATENTS.

**Lehrs.** L. MELLERSH-JACKSON. From BALL BROS. Co. (B.P. 343,445–6 and 343,457, 12.11.29).—Component parts of a continuously operated lehr are described: (A) deals with heat insulation, (B) with a feeding device, and (C) with a method of preventing the transport rollers from sagging. B. M. VENABLES.

**Ultra-violet [light] transmitting glass.** W. C. TAYLOR, Assr. to CORNING GLASS WORKS (U.S.P. 1,774,854, 2.9.30. Appl., 15.10.27).—Primary or vein quartz, after treatment to remove titaniferous and ferruginous impurities, is used as the main ingredient in making glasses of this type. A typical batch composition is: SiO<sub>2</sub> 75, Na<sub>2</sub>O 15, CaO 4, ZnO 5, and Al<sub>2</sub>O<sub>3</sub> 1%; such a glass will transmit approx. 87% of the radiation at 302 mm. in thicknesses of 2 mm.

C. B. MARSON.

**Furnace lining.** G. D. EVANS, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,775,161, 9.10.30. Appl., 14.3.28).—A lining for copper-refining furnaces consists of silica particles bonded in a matrix of oxides of copper and absorbed silica. The lining is built up by forcing finely-divided silica against the wall while the charge is being withdrawn. Treatment of the lining by this method increases considerably the working life of the furnace. C. B. MARSON.

**Lehr.** L. J. HARTLE (U.S.P. 1,775,281, 9.9.30. Appl., 10.12.26).—Flues are arranged in close heat-exchanging relation with the tunnel for the goods and a proportion of the used gases is returned to the flues at points distributed over the length of the kiln.

B. M. VENABLES.

**Refractory brick.** A. J. JACKMAN and C. L. JONES, Assrs. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,775,396, 9.9.30. Appl., 31.5.28).—Composite bricks comprising a body of relatively poor heat-conducting material such as fireclay or silica have embedded therein rods of relatively good, heat-conducting, non-metallic, refractory material, *e.g.*, a silicon carbide-graphite mixture. The rods are placed in a direction normal to the face of the brick which is to be exposed to furnace heating. Bricks so constructed are less liable to spalling.

C. B. MARSON.

**Furnace wall.**—See I. **Wetting agents.**—See III. **Mirrors.**—See X. **Adhesive.**—See XIII.

### IX.—BUILDING MATERIALS.

**Solubility of cement.** D. WERNER (Tekn. Tidskr., 1930, 60, No. 37, Kemi, 57—64, 68—72; Chem. Zentr., 1930, ii, 3835).—The removal of constituents of various cements by shaking with water was examined; the results are expressed graphically. The use of acid addenda to diminish the solubility of calcium is discussed.

A. A. ELDRIDGE.

**Hygroscopicity of wood after various drying processes.** C. G. SCHWALBE and K. BERNDT (Kolloid-Z., 1931, 54, 314—326).—The experiments were conducted with fir and pine wood. When air-dried wood is exposed to air which is 92% saturated with moisture, it takes up water rapidly at first and then more slowly, reaching a water content of 20%. Wood dried in a vacuum at the ordinary temperature takes up less water (18.4%), and only 17.6% of water is taken up by wood dried in air at 105°. In all cases the maximal absorption is reached after about 14 days. The hygroscopicity of wood is reduced by heating at 105° in oxygen or in nitrogen and the effect is therefore not due to an oxidation process. When wood is allowed to swell in water and then kept in moist air the rate of loss of water is slower than the regain of dried specimens, and at equilibrium the wood contains about 1.2% more water than do specimens which have previously been dried. The hygroscopicity is also increased by allowing the wood to swell previously in air saturated with water vapour. Swelling and shrinkage are reversible and exhibit hysteresis. Measurements of hygroscopicity can be taken as a measure of swelling, and a simple method for technical purposes is described. Extraction of wood with alcohol or benzene does not alter the hygroscopicity or swelling of the wood in water.

E. S. HEDGES.

**Flax residues.**—See V. **Plaster moulds.**—See VIII. **Waste pine wood.** **Kauri timber.**—See XIII.

### PATENTS.

**Asphalt filler [for pavings etc.].** H. L. MEAD, Assr. to AMER. CYANAMID Co. (U.S.P. 1,774,608, 2.9.30. Appl., 9.5.25).—The use of phosphatic slimes and finely-divided dusts and silts, obtained in hydraulic

mining operations and suitably pretreated, is claimed. The fineness of the added material is such that 90% passes 200-mesh, 75% 300-mesh, and 40% 400-mesh.

C. B. MARSON.

**Surface treatment of roads.** F. C. RANDS and T. F. N. ALEXANDER (B.P. 343,523, 20.11.29).—Road surfaces which contain bituminous substances, and especially those having an excess of this component, are prepared for re-surfacing by treatment with an oily solvent such as kerosene, creosote, etc.

R. H. GRIFFITH.

**Rotary kilns. Drying wood etc.**—See I. **Bituminous dispersions. Pitch composition.**—See II. **Refractory brick.**—See VIII. **Adhesive.**—See XIII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Explanation of the slag problem in the basic open-hearth furnace.** R. BACK (Stahl u. Eisen, 1931, 51, 317—324, 351—357).—The amounts of iron and manganese which pass by oxidation into the slag during the melting down of a charge in the basic open-hearth furnace and during the subsequent "boiling" are shown, by continuous analyses of metal and slag during 19 fusions, to increase with an increase in the CaO : SiO<sub>2</sub> ratio (*V*), but the manganese losses during melting are liable to be somewhat variable (*cf.* Schleicher, B., 1931, 250). In order to ensure the maximum return of manganese to the metal bath during the deoxidation stage, the slags produced should have *V* = 1.5—2. More acid slags with *V* = 1.2—1.4 also give a good return of manganese, whereas highly-basic slags with *V* = 2.5—2.8 give, contrary to expectation, only a poor return of manganese; this behaviour is probably due to the high content in highly basic slags of ferrosilicic oxide, which has a powerful oxidising action on manganese in the metal bath. The "slag test" is shown to be a useful method of determining the correctness of the operations; a good slag button should have a completely smooth, glistening, black surface and a beautiful matt-black fracture free from any trace of lustre.

A. R. POWELL.

**New cyanide furnace [for steel hardening].** R. F. JAMES and G. COLEY (J. Franklin Inst., 1931, 211, 327—334).—The chromium-nickel heating elements used for steel hardening from a bath of molten cyanide have a short and uncertain life owing to corrosion and finally to burning out. Experiments showed that this effect was associated with the presence of cyanide vapour, which cannot in practice be altogether excluded from the element chamber. The corrosion increases with the working temperature. It is concluded that it was probably due to the formation of complex cyanides, and that the provision of adequate air circulation, which oxidises cyanides to inert cyanates, would prevent it. A furnace was therefore designed with a baffle on the air intake which directed the air on to the heating elements during the heating period and against the cyanide pot during the cooling period. The size of the elements was increased in order to lower the element temperature without loss of heat capacity. This furnace has been run for 1800 working hours without appreciable

deterioration, which is a great improvement on usual practice.

C. IRWIN.

**Chemical reactions in the acid steel processes and in the deoxidation of steel with manganese and silicon.** H. SCHENCK (Arch. Eisenhüttenw., 1930—1, 4, 319—332; Stahl u. Eisen, 1931, 51, 292—294).—Curves have been constructed showing the relation between the ferrous oxide content of molten iron at various temperatures and the manganese and silicon contents, and from these curves ternary diagrams have been developed to show the equilibria conditions between the silicon and manganese contents of the metal and the composition of the ferrous manganous silicate slag and the amount of free ferrous oxide in the slag. High temperatures tend to increase the silicon and manganese contents of the metal, this tendency being also favoured by the greater solubility of the hearth silica in the slag. At 1527° for metal containing 0.3% Mn and 0.05% Si the composition of the slag in equilibrium is 8.6% FeO, 40% MnO, and 51.4% SiO<sub>2</sub>; the slag contains 0.8% of free ferrous oxide, and the metal 0.008% FeO ( $\equiv$  0.0017% O). In order to obtain a slag of sufficient fluidity for good deoxidation it is essential that the manganese content of the metal exceed a definite minimum value, dependent on the silicon content; thus for 0.05% Si, at least 0.13% Mn is necessary at 1527°; for 0.1% Si, 0.22% Mn; for 0.2% Si, 0.35% Mn; and for 0.5% Si, about 0.5% Mn. A. R. POWELL.

**Reduction of magnetite and limonite by methane.** O. MEYER and W. EILENDER (Arch. Eisenhüttenw., 1930—1, 4, 357—360; Stahl u. Eisen, 1931, 51, 294—295).—Reduction of limonite by methane commences at about 700°, and takes place more rapidly than that of magnetite at a somewhat higher temperature. At constant temperature the rate of reduction of both oxides increases with increasing gas velocity to a maximum and then decreases at higher velocities; at constant gas velocity the rate of reduction increases linearly with rise of temperature, the deposition of carbon on the iron particles preventing sintering of the mass. The shape of the reduction curves indicates that methane itself is not an active reducing agent, but that reduction is brought about by the carbon and hydrogen produced by its thermal dissociation; for this reason it is impossible to obtain an iron sponge sufficiently low in carbon and phosphorus to justify the use of methane as a reducing agent for iron ores in an externally heated furnace. On the other hand, quite good sponges may be produced by a regulated partial combustion of the methane inside the reducing chamber in such a way as to produce the heat necessary for the reaction and sufficient hydrogen and carbon monoxide to effect the reduction; the consumption of methane in this procedure may, under favourable conditions, be as low as 300 m.<sup>3</sup> per ton of iron produced.

A. R. POWELL.

**Effect of certain elements on the segregation of cementite, and its relation to the modification process [in steels].** J. H. ANDREW (J. Roy. Tech. Coll. Met. Club, 1929—30, No. 7, 16—17).—The influence of certain elements, notably manganese, has been studied. Specimens of steels, containing (a) 0.08% Mn, 0.22% C,

and (b) 0.60% Mn, 0.21% C, have been subjected to prolonged treatment at 1100—1300° followed by rapid cooling in air. The carbide in (a) was largely present as films in the grain boundaries, whereas in (b) carbide occurred only as a constituent of pearlite. The effect of manganese in this case is compared with that of sodium in producing the modification of aluminium-silicon alloys.

E. H. BUCKNALL.

**Scheme for research on steel-production methods on physico-chemical principles.** H. SCHENCK (Stahl u. Eisen, 1931, 51, 197—202).—The importance of a thorough knowledge of the chemical reactions which occur in the manufacture of steel is emphasised and several promising lines of research are indicated. Finally a scheme of co-operation between works, research laboratories, and colleges for further examination of the metallurgical processes in steel manufacture is outlined.

A. R. POWELL.

**Large-scale tests in pig-iron analysis.** K. DAEVES (Stahl u. Eisen, 1931, 51, 202—204).—The results of daily analyses of pig-iron from three blast furnaces over a period of three months' working are collated and frequency curves are constructed for each element present, showing the distribution of the results between the maximum and minimum values. A further series of curves showing the relation between the content of each pair of the elements silicon, manganese, sulphur, and phosphorus indicates that there is some connexion between the amounts of these elements in the pig iron irrespective of the conditions of operating the furnace.

A. R. POWELL.

**Hair-line cracks [in steel].** T. M. SERVICE (J. Roy. Tech. Coll. Met. Club, 1929—30, No. 7, 9—15).—Ingots of steel (C 0.24—0.29%, Ni 3.4—3.8%, Cr 0.52—0.82%, Mn 0.40—0.58%, S + P < 0.35%) have been cast and subjected to hot-working. Ingots forged hollow have yielded material free from cracks, but solid-forged ingots were markedly affected by cracking. This was especially the case when the temperature of final annealing was above the Ac<sub>3</sub> point. Similarly, forged material annealed below the Ac<sub>1</sub> point were sound, even though the original ingot was badly cracked. The defect arises from stresses set up in cooling after forging, and not from unsoundness of the ingot, as was previously believed.

E. H. BUCKNALL.

**Vibration fatigue strength [of steel].** W. SCHNEIDER (Stahl u. Eisen, 1931, 51, 285—292).—The importance of a knowledge of the fatigue strength of constructional steels is emphasised and modern ideas on the mechanism of fatigue are discussed. The effect of surface irregularities, notches, bends, and variations in the cross-section on the behaviour of metal subjected to high loads is considered, and it is shown that the fatigue strength of such metal is improved if it is first subjected to a high static load above the yield point.

A. R. POWELL.

**Sensitivity of mild ingot steel to overheating.** E. POHL, E. KRIEGER, and F. SAUERWALD (Stahl u. Eisen, 1931, 51, 324—326).—Specimens of electrolytic iron (0.06% C, 0.11% Mn, 0.10% Cu, 0.013% P, and 0.03% S), mild steel (0.07—0.14% C, 0.46—0.52% Mn, 0.2% Cu, 0.025—0.044% P, and 0.03—0.075% S),



and molybdenum steel (0.16% C, 0.48% Mn, 0.14% Cu, 0.3% Mo, 0.011% P, and 0.02% S) were annealed for 1 hr. and for 5 hrs. at 950–1400° and the increase in grain size and depth of penetration of the oxide layer measured. Grain growth in the electrolytic iron proceeded regularly with rise in annealing temperature from 900° to 1400°, but in the other steels grain growth was slow up to 1100°, above which it proceeded rapidly to 1300°, and then ceased; in two cases a slight decrease in grain size occurred at temperatures just above the A<sub>c</sub>3 point. Burning of the metal commenced at 1300° in the case of electrolytic iron and at 1350–1400° in the steels; above 1400° all the steels showed an overheated structure. In 5 hrs. oxidation had penetrated to a depth of 0.3 mm. into electrolytic iron at 1150° and of 0.8–0.9 mm. into the steels at 1300°. Apparently the variable sulphur and phosphorus contents of the steels had little effect on the grain growth or tendency to oxidise. Bending tests at 1200–1250° showed that normalised steel behaves satisfactorily owing to its fine-grained structure, whereas coarse-grained recrystallised steel rapidly develops intercrystalline cracks.

A. R. POWELL.

**Volume changes of steel under elastic and plastic stresses.** H. HANEMANN and R. YAMADA (*Arch. Eisenhüttenw.*, 1930—1, 4, 353–356; *Stahl u. Eisen*, 1931, 51, 225–226).—The increase in volume of annealed and worked steels during the application of the load in the tensile test has been measured. The load-increase in volume graph for any specimen is a straight line within the elastic range, but becomes curved in the plastic range; for all the samples tested the points at which this deviation occurred lay in a smooth curve bending slightly towards the volume axis, *i.e.*, plastic flow produces an abnormal increase in volume. Steel the state of which is represented by a point in the field below this curve is in a metastable condition and will revert to its normal volume after prolonged keeping or after gentle heating.

A. R. POWELL.

**Determination of the creep limit [of steel] by an accelerated test.** A. POMP and W. ENDERS (*Stahl u. Eisen*, 1931, 51, 157–162).—The apparatus comprises a furnace the temperature of which may be maintained practically constant for very long periods while the creep of the specimen is measured automatically on a photographic recorder. The creep of four steels with 0.06–0.61% C, three cast steels, and a 13% Cr steel at 300, 400, and 500° has been followed over periods up to 2400 hrs. From the results obtained it is suggested that the creep limit or endurance strength of a steel at any temperature should be taken as the highest load which the specimen will withstand without undergoing an elongation of more than 0.003%/hr. between the fifth and the tenth hours of the test. To determine the endurance strength at any temperature the elastic limits (0.01, 0.1, and 0.2%) at that temperature are determined in the usual tensile machine and 10-hr. tests under these loads are made in the apparatus mentioned above; from the rates of extension found in the period 5–10 hrs. the load corresponding with a 0.003%/hr. rate is obtained by interpolation.

A. R. POWELL.

**Tensile properties of alloy steels at elevated temperatures as determined by the "short-time" method.** W. KAHLBAUM, R. L. DOWDELL, and W. A. TUCKER (*Bur. Stand. J. Res.*, 1931, 6, 199–218).—The proportional limits of the following steels have been investigated by the "short-time" test, *i.e.*, by determining the stress-strain curve at the desired temperature during  $\frac{1}{2}$  hr.: plain carbon steel, and alloys of chromium-vanadium, chromium-vanadium-tungsten (also with additions of aluminium and silicon), chromium-tungsten, nickel-molybdenum, and austenitic steels containing nickel and chromium and in some cases tungsten. Addition of 1.5–2% of tungsten to a chromium-vanadium steel improves the tensile properties at high temperatures; further additions of aluminium or silicon produce no marked improvement. A marked increase in tensile strength of a steel brought about by heat treatment is maintained at high temperatures only if the temperature of testing is kept below that of tempering. "Short-time" tests cannot satisfactorily replace "flow" tests at high temperatures.

C. W. GIBBY.

**Iodometric determination of vanadium in special steels and in ferrovanadium.** W. WERZ (*Z. anal. Chem.*, 1931, 83, 161–164).—The method described previously (*B.*, 1930, 1156) is modified as follows: the steel (1–3 g.) is boiled with 25 c.c. of phosphoric acid ( $d$  1.7) and 200 c.c. of water and, when the metal is completely dissolved except for a small amount of chromium carbide, the solution is oxidised with nitric acid ( $d$  1.4), using an excess of 5 c.c. Red fumes are expelled by boiling, the solution is filtered through a porcelain filter crucible, and the filtrate boiled for 35 min. with 10 c.c. of 5% ammonium persulphate solution. A further 25 c.c. of phosphoric acid are added and, after cooling, 2–10 c.c. of 0.2*N*-potassium iodide. After 5 min. the iodine liberated is titrated with thiosulphate. For ferrovanadium, 1 g. is dissolved in 50 c.c. of 1:1 nitric acid and the solution diluted to 500 c.c.; 50 c.c. are then mixed with 50 c.c. of phosphoric acid, 150 c.c. of water, and 5 c.c. of nitric acid ( $d$  1.4), and the analysis is finished as above.

A. R. POWELL.

**Metallurgical considerations in autogenous welding [of steel].** F. RAPATZ (*Stahl u. Eisen*, 1931, 51, 245–253).—For mild steels electric welding with a clean welding rod connected to the negative pole gives the strongest joints, but the method is unsuitable for steels with a high content of carbon and alloy metals. With the welding rod connected to the positive pole it is difficult to get the welding metal to burn into the work, but this arrangement is the most suitable for spot-welding or for building up worn parts on high-carbon steels. Uncoated wires do not give satisfactory welds with alternating-current arcs, but cored wires can be used in many cases. Coated rods can always be used with alternating-current, but with direct-current arcs they should be connected to the positive pole. The effect of the composition of the welding rod and the welding conditions on the composition of the metal in the welded joints is discussed at some length with reference to many examples. The metal surrounding the weld usually shows several zones of different heat



treatment; immediately surrounding the weld is an overheated zone, then a normalised zone, a less well-annealed zone, and finally, in many cases, a zone of metal showing "blue brittleness." The extent of these zones is greatest with gas welds and least with electric welds made with uncoated rods. In the case of alloy steels there may be zones of air-hardened metal. The existence and extent of these various zones play a considerable part in determining the strength of the weld.

A. R. POWELL.

**Pickling and etching [of metal surfaces].** H. KURREIN (Chem.-Ztg., 1931, 55, 133—134, 150—151).—The composition, purpose, and method of action of pickling baths for iron and steel are discussed and the function of inhibitors is explained. The production of matt, coloured, and etched surfaces on copper alloys by treatment with concentrated acids etc., and on aluminium and light alloys by means of alkaline reagents is also described and details of the composition and method of using numerous etching baths are given.

A. R. POWELL.

**Importance of the crystal structure for the determination of the elastic limit and endurance strength of electron.** W. SCHMIDT (Z. Metallk., 1931, 23, 54—57).—From crystallographic considerations it is deduced that it is not permissible to compare the behaviour of hexagonal and cubic metals on the basis of their conventional elastic limits. The true elastic limit of a hexagonal metal can be deduced only from a consideration of the whole load-extension curve. Values for the static and dynamic endurance strength and for the hysteresis of electron are recorded.

A. R. POWELL.

**Corrosion of early Chinese bronzes.** W. F. COLLINS (Inst. Metals, Mar., 1930. Advance copy. 25 pp.).—Numerous Chinese bronzes ranging in age from the earliest known periods of the Chinese bronze age up to 906 A.D. have been analysed and the nature of the patina and patination or surrosion deposits thereon has been determined in many cases. As has been found to occur in the case of copper exposed to atmospheric corrosion, the corrosion products on Chinese bronzes which have been buried for many hundreds of years in the loess of Central China approximate in composition and characteristics to natural minerals. Many of the bronzes have a high content of lead which in some cases appears to replace the tin almost entirely.

A. R. POWELL.

**Debismuthising lead with calcium.** R. F. COHN (Trans. Amer. Electrochem. Soc., 1931, 59, Preprint 2, 13—18).—The bismuth content of lead may be reduced to below 0.05% by treating the molten metal with calcium at 480°, cooling to just above the m.p. of lead, and removing the dross, which resembles that produced in the Parkes process for desilverising lead, and consists of a ternary bismuth-calcium-lead compound. This treatment eliminates also the last traces of arsenic, antimony, copper, and silver; the excess calcium is removed by blowing air, steam, or chlorine through the molten metal. Calcium may be added to lead as a lead-calcium alloy produced by electrolysis of fused calcium chloride, using molten lead as the cathode, or by heating lead with calcium carbide at 870°.

A. R. POWELL.

**Influence of pressure and temperature on the extrusion of metals.** C. E. PEARSON and J. A. SMYTHE (Inst. Metals, Mar., 1931. Advance copy. 25 pp.).—Lead can be extruded into smooth rods below 325°, cadmium at 100—260°, bismuth at 30—250°, and tin at 16—223°. The rate of extrusion ( $R$ ) of all four metals is connected with the extrusion pressure  $P$  by the equation  $\log R = a \log P + \log b$ , where  $a$  and  $b$  are constants having distinct values for every temperature and metal. The value of  $a$  decreases in a smooth curve with increase of temperature in the cases of lead, bismuth, and cadmium, being greatest for bismuth and least for cadmium. For tin the value of  $a$  reaches a minimum at about 160°, but in all cases the curves are approximately hyperbolæ. The values of  $b$  at different temperatures are really the rates of extrusions produced by a load of 1 ton at these temperatures. Numerous photographs of the etched macrostructure of extruded rods of these metals are reproduced, showing the flow lines and zones of recrystallisation, and the causes of certain defects in extruded rods are briefly discussed.

A. R. POWELL.

**Electrothermic zinc.** A. WALTER (Trans. Amer. Electrochem. Soc., 1931, 59, Preprint 4, 27—31).—The development of electrothermal zinc during the last 30 years is reviewed. In modern practice the roasted ore is smelted with coke in a Héroult arc furnace, whereby most of the zinc distils, leaving in the furnace a bottom layer of lead covered with a thin layer of copper matte and a thicker layer of slag containing 2% Zn. The vapours from the furnace pass to a condenser, and thence to a rotating drum which causes the condensed particles to coalesce into liquid zinc. About 95% of the distilled zinc is collected as crude liquid metal and 5% as "blue powder." The crude zinc contains 93—95% Zn, 2% Fe, and 5—3% Pb, and is redistilled from a small closed retort heated by an indirect arc, whereby 99.7% Zn is obtained, leaving a pool of lead and an iron alloy in the refining furnace. Current consumption is about 2000 kw.-hrs. per ton of ore and 1250 kw.-hrs. per ton of crude zinc. In addition to the zinc, 94—96% of the lead, about 95% of the gold and silver, and about 80% of the copper in the ore may be recovered from ores containing more than 1% Pb, 1% Cu, and 0.5 g. Au per ton. The zinc dust obtained as a by-product contains 96% Zn and has a reduction efficiency of 20% above that of ordinary "blue powder."

A. R. POWELL.

**Influence of small quantities of foreign metals on the properties of refined zinc.** [O. BAUER and P. ZUNKER (Z. Metallk., 1931, 23, 37—47).—Addition of up to 3% of tin, cadmium, magnesium, or antimony to zinc reduces appreciably the pipe formed in cast ingots; copper, lead, and iron, however, have no action in this respect. Tin, antimony, lead, and cadmium have no effect on the hardness or impact strength, magnesium and iron make zinc hard and brittle, and copper renders it hard and increases the tensile strength. The variations in hardness, impact strength, and percentage contraction on solidification for zinc alloys with 0—3% of each of the above metals are shown in a series of graphs and tables.

A. R. POWELL.

**Hot-galvanising with zinc and cadmium.** ANON. (Chem.-Ztg., 1931, 55, 225—226).—An account is given of the methods used in galvanising iron and steel sheets by the hot-dipping process and in the preliminary cleaning of the metal in pickling baths. Cadmium or cadmium-zinc alloys are now replacing zinc in the galvanising bath when a higher resistance to corrosion is required; these baths can be operated at lower temperatures, *e.g.*, at 220° for the alloy with 29% Cd and 71% Zn, or at 300° for the alloy with 17% Zn and 83% Cd, do not take up nearly as much iron as does the ordinary zinc bath, and hence may be melted in iron pots.

A. R. POWELL.

**Some properties of metallic cadmium.** C. H. M. JENKINS. **X-Ray investigation of cold- and hot-rolled cadmium.** G. D. PRESTON (Inst. Metals, Mar., 1931. Advance copy. 32 pp. [with appendix]).—The physical properties of cadmium have been investigated by means of tensile and ball-hardness tests and by electrical conductivity measurements on "aged" and heat-treated specimens in the cast, forged, and rolled conditions. Hardness changes in rolled material which occur at room temperature are markedly affected by slight changes in the conditions of working, such as the temperature produced during rolling, or according as rod or sheet is the subject of examination. At the commencement of cold-working the metal hardens; on continued working it softens. By maintaining cadmium cold during rolling, the material produced possesses properties markedly different from those of metal which is allowed to become warm during the process. In the short-time tensile tests worked material is found to be stronger than cast material, but under prolonged stress the cast alloys are inferior to rolled samples. Microscopical examination of the various specimens shows that in the cold-rolled condition the material appears to be completely recrystallised and of fine grain size, but prolonged ageing over a period of years results in appreciable grain growth. After slight straining, the microstructure of cadmium is rendered complex, and full account must be taken of the effect of the methods of preparation for this work. Density determinations of variously-aged specimens have also been made.

The X-ray examination of samples of hot- and cold-worked cadmium does not indicate an allotropic modification at room temperature and ordinary pressures, but there are marked differences in properties which suggest a preferred orientation.

O. J. WALKER.

**Deformation of metals under prolonged loading.**

**I. Flow and fracture of aluminium.** D. HANSON and M. A. WHEELER (Inst. Metals, Mar., 1931. Advance copy. 29 pp.).—The microstructure of polished specimens of aluminium subjected to static stresses at various temperatures has been examined in order to follow the changes which occur during deformation by slow creep. Prolonged application of a load which will ultimately cause fracture causes a slow elongation at first, the rate of which slowly diminishes; this is followed by a stage in which movement is almost or quite at a standstill, and finally by a period in which flow commences again at a rate which continuously increases until fracture

ensues. Flow in the first period is due to slip within the crystals, and its retardation is caused by the accompanying hardening effect of this slip. Failure under creep may be caused by intercrystalline cracking, characterised by a low elongation and reduction in area, by resumption of slip within the original crystals leading to fracture of the normal tensile type, and by the removal of strain-hardening due to recrystallisation, leading to fracture with a high elongation and reduction in area at the break. Aluminium fails by creep at the ordinary temperature by shear along the slip planes, and polycrystalline aluminium by intercrystalline cracking at 250°. The behaviour of single-crystal specimens of aluminium under creep conditions has been examined at temperatures up to 400°: in the third stage of flow slip occurs only on some of the surfaces that had previously slipped during the primary flow, and when the rate of flow is slow slip is distributed over such a large number of slip surfaces that a polished surface of the metal may show only traces of slip bands. Lead fails under prolonged stress at the ordinary temperature with a large elongation and a point fracture, but at 200° it fails by the development of intercrystalline cracks.

A. R. POWELL.

**Dimensional stability of heat-treated aluminium alloys.** J. D. GROGAN and D. CLAYTON (Inst. Metals, Mar., 1931. Advance copy. 28 pp.).—Serious dimensional changes occur during the machining of many aluminium alloys after quenching in cold water; in the case of "Y"-alloy and duralumin these changes may be prevented by quenching in boiling water. No secular changes occur in these alloys after completion of the normal age-hardening process.

A. R. POWELL.

**Electrodeposition of gold from alkaline cyanide solutions.** E. T. DUNSTAN (J. Chem. Met. Min. Soc. S. Afr., 1930, 31, 118—127).—Attempts to recover the gold from pregnant cyanide solutions, with simultaneous regeneration of free cyanide, by electrolysis in a cell with a rotating cathode showed that the recovery of gold was much more difficult than by precipitation with zinc. The current efficiency with solutions containing less than 10 dwt. of gold per ton was much below 1%, and no regeneration of cyanide whatsoever was obtained.

A. R. POWELL.

**Microstructures of fifteen silver Greek coins (500—300 B.C.) and some forgeries.** C. F. ELAM (Inst. Metals, Mar., 1931. Advance copy. 10 pp.).—Photomicrographic investigation of some ancient Greek coins and of some forgeries shows that the structure varies considerably, although coins from the same mint and period resemble one another. All the genuine coins were struck between dies, whereas the forgeries, with one exception, were made by casting only. Analyses indicate that the earlier coins were made of nearly pure silver, and that in the others copper was included. The forgeries contained copper and, in two cases, zinc.

O. J. WALKER.

**Silver alloys resistant to tarnishing and capable of being age-hardened.** J. A. A. LEROUX and E. RAUB (Z. Metallk., 1931, 23, 58—63).—The solid-solubility of copper in  $\alpha$ -zinc-silver solid solution is approximately the same as that in pure silver, but the rate of diffusion

is much greater. Hence, although these alloys undergo age-hardening after appropriate heat treatment, the reproduction of definite mechanical properties by this treatment is difficult, and is rendered more so by the tendency which the ternary alloys show to develop a very coarse crystal structure. The solid-solubility of nickel in the ternary alloys is less than 0.1% at the ordinary temperature and somewhat above 0.3% at 700°, but alloys with satisfactory working properties may be obtained with up to 1.5% Ni. Such alloys containing 83.5% Ag and the remainder copper and zinc in the ratio 1:3 may be age-hardened at 280°, after quenching from 600–700°, without developing a coarse crystalline structure. The optimum mechanical properties are obtained after tempering for 3 hrs. at 280°; the hardness then exceeds 90 and the tensile strength 42 kg./mm.<sup>2</sup> with an elongation of 25%. Almost as good results are obtained under similar heat-treatment conditions with the plain copper-silver alloy with 83.5% Ag, but the replacement of the greater part of the copper by cadmium destroys the capacity for age-hardening, and it is not restored by addition of nickel.

A. R. POWELL.

**Nature and causes of efflorescences on electrolytic silver coatings.** H. MOSER, K. W. FRÖHLICH, and E. RAUB (*Z. angew. Chem.*, 1931, 44, 97–100).—Silver-plated brass or nickel-silver articles frequently become covered with small white to yellow spots sometimes shortly after plating and sometimes after the lapse of many weeks. The spots appear to spread outwards from small nuclei. Experiments indicate that these nuclei are small hollows in the plate filled with the solution used for plating. At the base of these hollows is usually either a speck of cuprous oxide derived from the annealing of the base metal or a particle of polishing material driven into the surface of the base metal by too heavy buffing before plating. In the early stages of plating silver fails to deposit on the impurities, but when the plate thickens it grows over the inclusions, thus forming a hollow which includes part of the plating solution. On subsequently burnishing the deposit the minute hole at the centre of this hollow may be closed up, imprisoning the solution contained therein, this subsequently works its way by chemical or electrolytic action through the relatively thin film of silver covering it and then spreads over the surrounding metal, causing the characteristic efflorescences.

A. R. POWELL.

**Electric melting of non-ferrous alloy.** K. OKIMORO (*J. Fuel Soc. Japan*, 1931, 10, 11–12).—The cost of operating an oscillating, direct-arc type of electric furnace in the melting of phosphor-bronze, brass, and nickel is compared with the cost of using a coke crucible furnace. Melting losses were 0.2 and 1.4%, respectively, and melting costs were in the ratio 35:60.

A. R. POWELL.

**Cathodic protection of metals in neutral solutions.** U. R. EVANS (*Metals & Alloys*, 1931, 2, 62–64).—A rapid method of determining the value of protectors is to apply an adjustable *E.M.F.* through a resistance (40,000 ohms) which is high compared with that of an experimental cell. A constant small current is allowed to pass between the zinc anodes and the iron specimen to be studied. The liquid, containing 0.1% of potassium

ferricyanide as indicator, is run in, 2 c.c. each 15 sec., thus gradually increasing the area covered and decreasing the current density (*C.D.*). When the *C.D.* passes below the protective value a blue discoloration appears around the specimen. Remarkable variation between individual specimens was observed without any reference either to the nature or concentration of the neutral solution. Sulphuric acid induced increased corrosion by destroying the primary skin, but carbon dioxide and calcium bicarbonate allow protection at *C.D.* lower than those needed with other salts. The present work shows that the low *C.D.* recommended by Bauer and Vogel only slightly reduced the attack, which is still serious even at 5 times that value if the solution is stirred.

C. A. KING.

**Adhesion of electroplated coatings.** W. BLUM (*Metals & Alloys*, 1931, 2, 57–59).—The best adhesion of plated coatings may be expected when the metal surface is absolutely clean of all foreign materials, including grease, oxide, alkali, or soap. All metals show a tendency to contract during deposition, and the adsorption of hydrogen produces strains not only during deposition, but later as the gas is escaping gradually. It may be predicted that relatively soft ductile coatings will be deposited under conditions of low cathode polarisation and high efficiency. Chromium for abrasion-resistance is usually applied directly to the steel. For cleaning the steel it is best to make it anodic in the chromic acid bath just before plating.

C. A. KING.

**Testing chromium plate for resistance to abrasion.** H. C. WOLFE (*Metals & Alloys*, 1931, 2, 60–61).—A plated specimen rests horizontally, under the influence of a determined load, on a Norton 120J alundum wheel (diam. 2 in., face  $\frac{1}{8}$  in.). The wheel is rotated at a linear speed of 10 ft./min. and is cleaned continuously. A dial gauge measures the depth of the cut directly to 0.0001 in. Increased accuracy was obtained by the use of a copper wheel rotating in a mixture of levigated alumina, but the operation is longer and the abrasive mixture soon becomes contaminated with particles of chromium. The results of comparisons of the quality of different chromium plate were quite satisfactory, but were not so when compared with the abrasive resistance of other metals.

C. A. KING.

**Resistance of chromium-plated plug gauges to wear.** H. K. HERSCHMAN (*Bur. Stand. J. Res.*, 1931, 6, 295–304).—Within the limits used in chromium deposition, no material differences in the hardness of chromium deposited directly on hardened steel (1% C) were found. Microscopic cracks increased in number and resistance to wear of unlapped chromium plate decreased with increased thickness of deposit. The wear-resistance of lapped chromium plate was increased by preheating to 300°. “Milky” and “frosty” deposits, subsequently lapped, were more resistant than bright deposits similarly lapped. Preliminary copper plating of the steel improved the wearing properties of “milky” and “frosty” deposits of chromium, but not of bright ones.

C. W. GIBBY.

**Steam boilers.**—See I. **Varnish for tins.**—See XIII. **Tinned insulations.**—See XIV.

## PATENTS.

**Blast furnace.** R. FRANCHOT and K. P. McELROY, Assrs. to FERRO CHEMICALS, INC. (U.S.P. 1,775,955, 16.9.30. Appl., 6.8.28).—An iron blast furnace is provided with wide, refractory, heat-insulated conduits for withdrawing gas containing cyanide vapour from the hot zone of the furnace, just above the tuyères. The gases in each conduit pass through a narrow jet-like constriction into a collecting chamber in such a way that a swirling stream of expanded gas, which is thereby cooled, is produced in the chamber and the saline content of the gas is deposited. Cooling of the gases is also produced by the injection of cold gas through the constriction, and the chambers are provided with refractory filtering material so as to clean the gases before they pass through a common flue into a heat exchanger in which they preheat the air feed to the tuyères.

A. R. POWELL.

**[Regenerative] open-hearth [steel] furnace.** T. T. McENTEE (U.S.P. 1,775,367, 9.9.30. Appl., 13.12.21).—The ends of the furnace are each provided with a throat opening into the hearth, an uptake communicating with the throat, a nozzle extending through the wall of the uptake and towards the throat, a second stationary uptake connecting with the nozzle which is supported by a carriage to admit of its being moved in the throat, and a water-seal between the nozzle and the stationary uptake flue to prevent escape of gases in any position of the nozzle.

A. R. POWELL.

**Open-hearth furnace construction.** (A—H, K—P) G. L. DANFORTH JUN., (I, J) E. J. McDONNELL, (Q) R. S. A. DOUGHERTY, (R, S) A. T. KELLER, (T, U) F. ORTH, (V) H. E. SMYTHE, and (W) R. H. STEVENS, Assrs. to OPEN HEARTH COMBUSTION CO. (U.S.P. 1,769,209—1,769,213, 1,769,366—8, 1,769,446—7, 1,769,492—6, 1,769,678, 1,769,826, 1,769,842—3, 1,769,853—4, 1,769,863, and 1,769,866, [A—W] 1.7.30. Appl., [A] 27.12.21, [B] 28.1.22, [C] 2.2.22, [D] 15.3.22, [E, H] 6.4.22, [F] 8.10.21, [G] 28.11.21, [I, J] 16.5.21, [K] 26.9.21, 9.10.22, [M—O] 4.4.30, [P] 10.7.22, [Q] 8.5.22, [R, S] 5.12.21, [T] 7.2.21, [U] 6.9.24, [V] 9.1.22, [W] 22.9.21. Renewed [L] 11.4.30).—Various arrangements of the ports, air uptakes, flues, valves, and dampers governing the admission of air and gas to regenerative open-hearth steel furnaces are claimed.

A. R. POWELL.

**[Metallurgical and like] furnaces.** MELDRUMS, LTD., and S. N. MELDRUM (B.P. 343,001, 2.8.29).—In a furnace provided with a mechanical stoking device liable to be damaged by radiated heat, such device is separated from the furnace proper by a water-cooled collar. [Stat. ref.]

B. M. VENABLES.

**Electrothermic zinc furnace.** E. C. GASKILL (U.S.P. 1,775,591, 9.9.30. Appl., 17.9.28).—The furnace comprises a shaft made up of a series of separately supported spaced sections to provide for expansion and contraction and for openings through which the zinc vapours can escape. The upper part of the furnace contains three electrodes spaced at angles of 120° to one another round the shaft and the lower part contains three corresponding electrodes which are displaced at 180° with respect to the corresponding electrodes at the top of the furnace so that the current passes down the

shaft diagonally. Each of the three pairs of electrodes has its own current supply from a single-phase alternating-current transformer. The shaft is supplied continuously with a mixture of zinc oxide and coke, which is heated to the required reducing temperature by the resistance of the column to the current; the issuing zinc vapours are condensed to metal or burned to zinc oxide.

A. R. POWELL.

**Sintering machine [for blast-roasting sulphide ores].** C. H. MACE (U.S.P. 1,773,916, 26.8.30. Appl., 25.5.28).—The apparatus comprises a laterally inclined hearth surmounted by a movable, refractory-lined hood into which is directed one or more oil-fired burners in such a way that the flame passes round the inside of the hood and downwards over the inclined bed of ore.

A. R. POWELL.

**Crucible for aluminothermic reactions.** H. SCHULTZ (U.S.P. 1,774,816, 2.9.30. Appl., 24.7.29. Ger., 31.8.28).—The crucible comprises a conical sheet iron casing with a thick refractory lining surmounted by a heavy cast iron annulus which is bolted to the lower casing by means of external lugs. The crucible is especially adapted for welding iron or steel by the aluminothermic process; the cast iron top resists the action of the slag formed and thus prolongs the life of the refractory-lined portion in which the metal formed by the reaction collects.

A. R. POWELL.

**Treating [oxidised copper] ores [preparatory to flotation].** J. M.<sup>2</sup> CALLOW, Assr. to UNION CARBIDE SALES Co. (U.S.P. 1,774,786, 2.9.30. Appl., 15.1.27).—Coarsely crushed, oxidised copper ores are made into a thick paste with water and the paste is fed by means of a screw conveyor into a wet ball mill with a short feed aperture in which the thick paste as it passes into the mill forms a continuous gastight plug. Calcium carbide is added to the mill so that on grinding the ore the particles of copper mineral are coated with copper acetylide. The discharge from the mill passes into a water-sealed classifier from which the coarser particles are returned for regrinding and the water passes to a vacuum apparatus in which the excess acetylene is recovered and from which it is returned to the mill.

A. R. POWELL.

**Reduction of ores of reducible oxides [e.g., iron ores].** T. F. BAILY (U.S.P. 1,775,713, 16.9.30. Appl., 3.1.24).—A mixture of finely-divided iron ore and an excess of carbon over that required for its reduction is allowed to fall in a regulated continuous stream through a closed shaft furnace provided with an annular gastight chamber containing granular carbon heated by electrical resistance and situated near the bottom of the furnace so as to form a constriction in the shaft. Through tuyères in the lower part of the furnace below the auxiliary resistance heater is passed a current of hot carbon monoxide which in passing up through the shaft effects reduction of the ore. The gases from the top of the shaft pass through a bed of hot coke to regenerate carbon monoxide for further use in the process. At the bottom of the furnace is a pool of molten iron containing an excess of carbon to form carbide.

A. R. POWELL.

**Production of sponge iron from its ores.** F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 343,618, 29.11.29. Ger., 20.9.29. Addn. to B.P. 340,641; B.,

1931, 255).—The gas withdrawn from the blast furnace as claimed in the prior patent is cooled, freed from dust, and reheated before being returned to the furnace.

A. R. POWELL.

#### Uniting of iron with other metals and elements.

W. H. SMITH, Assr. to GEN. REDUCTION CORP. (U.S.P. 1,775,358, 9.9.30. Appl., 23.9.29).—Finely-divided iron is mixed with carbon or other alloying element and the mixture is compressed into the desired shape and heated at a temperature below the m.p. of iron, but sufficiently high to cause the second element to alloy with the iron; further pressure is then applied to consolidate the article into a solid uniform mass. If desired, the proportion of alloying element may be varied in the different parts of the article when making the mixture, so as to confer special properties, e.g., great hardness, on certain parts.

A. R. POWELL.

**[Cast-]iron alloys.** W. E. DENNISON (B.P. 343,745, 19.2.30).—Cast iron containing 3–3.5 (3.35)% C, 1.5–3 (2.25)% Si, 0.5–2 (0.7)% Cu, 0.25–0.75 (0.43)% Cr, 0.7% Mn, and the usual amounts of sulphur and phosphorus is claimed to be highly resistant to corrosion by dilute sulphuric acid.

A. R. POWELL.

**Manufacture of alloy steels.** C. ARNOLD (B.P. 343,700, 21.1.30).—The fluorine-containing slag used in the removal of sulphur and phosphorus from the iron is replaced by an alkaline-earth and/or magnesia metasilicate slag before addition of the alloying elements in the form of ferro-alloys. As the temperature falls the slag is rendered more fluid by the gradual addition of alkali metasilicate.

A. R. POWELL.

**Steel alloys.** F. KRUPP A.-G. (B.P. 343,464, 18.11.29. Ger., 20.12.28).—Molybdenum or vanadium steels with 0.5–5% Ti or Be and/or 0.75–4.9% Si are claimed to maintain their high strength at temperatures above 600°.

A. R. POWELL.

**[Nickel-chromium] steels.** C. K. EVERITT, and E. ALLEN & Co., LTD. (B.P. 343,997, 30.10.29).—Steels containing up to 2% Cu, 0.5–2% W, and/or 0.25–1% Mo, together with 8–20% Ni and 12–24% Cr are claimed. The nickel and chromium contents in any case exceed 32%.

A. R. POWELL.

**Ferrous alloys [containing copper, silicon, and aluminium].** B. D. SAKLATWALLA (B.P. 344,167, 31.1.30).—The alloys comprise iron with 0.5–5% Cu, 0.5–6% Si, and 0.1–2.0% Al; they are claimed to resist scaling and corrosion at high temperatures.

A. R. POWELL.

**Cobalt high-speed tool steel.** OESTERREICHISCHE SCHMIDTSTAHLWERKE A.-G. (B.P. 343,508, 14.11.29. Austr., 14.11.28).—The steel contains 3.45–18.91% Co, 16.9–22.1% W, 0.13–1.06% Mo, 0.09–2.13% Ta, 0.51–1.74% V, and 0.88–1.21% C.

A. R. POWELL.

**Purification of zinc ore.** O. GERLACH, Assr. to MATTHIÉSSON & HEGELER ZINC Co. (U.S.P. 1,773,991, 26.8.30. Appl., 31.12.27).—Roasted blende containing 3–5% S is mixed with a small quantity of water and spread in a 2-in. layer on a  $\frac{1}{4}$ -in. layer of coarse sinter on the grate of a sintering machine in which it is heated at about 1500° by means of an oil flame containing excess of oil and steam introduced into a movable hood which travels over the grate. In this way the cadmium and

lead are partly volatilised and partly caused to enter the lower layers of the charge. The upper layers of the sintered material are scraped off and sent to the retort house, and the lower layers are returned to another grate for re-sintering.

A. R. POWELL.

**Refining of lead bullion.** G. K. WILLIAMS (U.S.P. 1,774,688, 2.9.30. Appl., 28.3.29. Austral., 11.4.28).—Molten lead containing gold and silver is fed, together with the requisite quantity of zinc for desilverising, into the top of a deep kettle the upper part of which is maintained at a temperature sufficient to cause alloying between the zinc and precious metals and the lower part of which is maintained just above the m.p. of the eutectic zinc-lead alloy. The feed to the kettle and the removal of desilverised lead from the bottom is continuous, whilst a steady temperature gradient is maintained from top to bottom of the kettle and the crusts are removed intermittently as they become rich in silver. The process may also be carried out in a series of kettles, the underflow from one passing into the top of the next and the temperature falling regularly throughout the series, that in each kettle, however, being maintained constant.

A. R. POWELL.

**Removal of a lead coating from articles.** DUDZEELE CORP. OF AMERICA, Assees. of C. L. MANTELL (B.P. 343,793, 29.3.30. U.S., 29.3.29).—The article is immersed in a hot 15% sodium chloride solution containing 5% of sulphuric acid and 0.001–0.5% of nickel or platinum chloride as a catalyst.

A. R. POWELL.

**Treatment of galvanised articles.** H. B. NEWHALL, H. W. PLEISTER, and C. A. GIBLIN, Assrs. to H. B. NEWHALL CORP. (U.S.P. 1,773,495, 19.8.30. Appl., 21.8.28).—The articles are transferred from the galvanising kettle to a rotary screen inside a closed chamber through which is passed a mixture of air and sulphur vapour or into which is blown sulphur dust while the temperature is maintained at 400–500°, the apparatus being rotated to remove excess zinc and to produce a bright non-oxidised surface on the articles.

A. R. POWELL.

**Production of metal layers.** B. LOEWE (B.P. 343,875, 11.10.29. Ger., 13.10.28).—Metallic layers of magnesium, aluminium, iron, calcium, mercury, or cobalt are produced on metallic or non-metallic articles by sublimation *in vacuo*, and a volatile compound of another metal, e.g., nickel, chromium, copper, arsenic, or tungsten, is caused to react with the first layer in order to effect an interchange of metals in the layer. The method is particularly applicable to the production of electric condensers, vacuum vessels, or electric resistances.

A. R. POWELL.

**Manufacture of optical metal mirrors.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 343,424, 12.8.29 and 23.1.30).—An alloy of 92.2% Ag and 7.8% Al is vaporised at a pressure of  $10^{-4}$ – $10^{-5}$  mm. on to a heated surface of quartz or glass with a low alkali content.

A. R. POWELL.

**Casting of metal.** E. BORNAND and H. A. SCHLAEPFER (B.P. 343,648, 14.12.29. Fr., 21.12.28).—The upper part of the metal in the mould is heated by an immersed high-frequency induction unit to prevent the formation of shrinkage pockets.

A. R. POWELL.

**Production of metal sponge.** E. G. T. GUSTAFSSON (B.P. 343,954, 19.11.29. Swed., 28.11.28).—Oxidised ores of iron, nickel, cobalt, or chromium and iron are mixed with a slight excess of finely-divided, carbonaceous reducing agent and/or a thermal reducing agent and the mixture is briquetted. The briquettes are then heated in a stream of gaseous, liquid, or solid (dust) fuel mixed with a slight excess of air to burn away the excess reducing agent in the briquettes. The reduction is effected in shaft or rotary furnaces in which the gas stream is applied countercurrent to the movement of the charge.

A. R. POWELL.

**Electrolytic cleaning of metal [iron or steel].** T. E. DUNN, ASSR. to BULLARD Co. (U.S.P. 1,775,671, 16.9.30. Appl., 1.11.26).—The article is made the cathode in an acid solution containing a lead salt and a high current is passed through the solution from a lead anode so that as the scale is removed from the iron or steel surface it is replaced by a protective coating of lead.

A. R. POWELL.

**Electroplating.** W. S. MURRAY, ASSR. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,772,840, 12.8.30. Appl., 8.3.24. Renewed 28.7.27).—The bath contains a number of anodes and means for automatically changing the current from one to another and altering its density for uniform, predetermined periods while keeping the same cathode in the circuit so as to vary the coating deposited on different parts thereof in a regular manner.

A. R. POWELL.

**Electroplating aluminium and its alloys [with zinc].** H. SUTTON and H. C. COCKS (B.P. 344,133, 10.1.30).—The surface of the article is cleaned in solvents which remove the oxide film, sand-blasted, and plated with zinc in a bath of  $p_H$  3.5—5.5, e.g., one containing 144 g. of zinc sulphate crystals, 34 g. of sodium acetate crystals, and 1 g. of gum arabic, per litre.

A. R. POWELL.

**Electrodeposition [of zinc] on aluminium and its alloys.** H. SUTTON and H. C. COCKS (B.P. 343,685, 10.1.30).—The bath used comprises a solution of sodium zinc cyanide in dilute ammonia and is operated at 8 volts at 15—25°, using zinc anodes.

A. R. POWELL.

**Casting of metals, ceramic masses, and other suitable materials [for dental plates etc.] by the centrifugal process.** P. C. FRITZSCHE (B.P. 342,861, 6.1.30).

**Heat-treatment furnaces. Regenerative furnaces. Heat insulation. Steel abrasives. Jigs.**—See I. Roaster gases.—See VII. Furnace lining [for copper].—See VIII.

## XI.—ELECTROTECHNICS.

**Tar removal. Methane. Transformer oils etc.**—See II. **Welding. Zinc. Gold. Electroplated coatings. Protection of metals. Non-ferrous alloy.**—See X. **Insulated conductors.**—See XIV. **Nutrient in soils.**—See XVI. **Conductivity.**—See XVII.

## PATENTS.

**Incandescence electric lamps [with reflecting bulbs].** J. VAN ECK and H. FEKKES (B.P. 344,611, 29.1.30. Belg., 9.8.29).

**Mercury-vapour rectifiers.** A.-G. BROWN, BOVERI & COE. (B.P. 344,901, 5.11.29. Ger., 8.11.28).

**[Partition walls for] electrical accumulators.** H. WEHRLIN (B.P. 344,709, 26.4.30).

**[Hard-paper] insulators.** INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 345,090, 18.3.30. Ger., 19.3.29).

**Scale removal. Gas cleaning.**—See I. **Mixed gas.**—See II. **Casting metals. Cleaning metals. Zinc furnace. Zinc on aluminium. Metal layers. Electroplating.**—See X.

## XII.—FATS; OILS; WAXES.

**Detection of foreign fats in cacao butter.** B. PASCHKE (Z. Unters. Lebensm., 1930, 60, 327—331).—Since, according to Amberger and Bauch (B., 1925, 105), cacao butter consists entirely of the glycerides of palmitic, stearic, and oleic acids, any addition of fat containing lower fatty acids may therefore be detected by isolation of the latter. Thus 21 g. of fat, 150 c.c. of 96% alcohol, and 3 c.c. of concentrated sulphuric acid are boiled briskly under reflux for 5—6 hrs. The solution is poured into 1 litre of water and the esters are extracted by means of light petroleum. The petroleum solution is washed several times with its own volume of water to remove sulphuric acid and alcohol, and dried over sodium sulphate. After removal of most of the solvent by distillation, the remainder is removed from the esters completely by heating on the water-bath for 2 hrs. The esters are then fractionated *in vacuo*, 50% being distilled over. The distillate is then redistilled, 3—3.3 g. being distilled over. The quantity of distillate is controlled by weighing the receiver. The saponification values of the residue and of the two fractions distilled are next determined, using 2.5 g. of esters and saponifying with 30 c.c. of 0.5*N*-alcoholic potash for 10 min. under reflux on the water-bath. If  $S_1$  be the saponif. value of the first distillate,  $S_2$  that of the second distillate, and  $S_3$  that of the residue, then the weight of foreign fat, expressed as coconut oil, present in 100 g. of cacao butter is  $(S_2 - S_1 - 5.5)/2$  or  $(S_2 - S_3 - 9.5)/2$ , the latter value being less accurate than the former. The value  $n_D^{20}$  of the fractions (Zeiss butyrefractometer) is also useful. W. J. BOYD.

**"Scheiber" oil.** G. G. SWARD (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 126—127).—The constants of 3 samples—unprocessed, blown, and heat-thickened—of "Scheiber" oil (the glyceride of dehydrated ricinoleic acid [cf. B.P. 306,452—3; B., 1930, 726, 871]) are reported. The unprocessed oil dries overnight to a frosty film. If 0.03% of cobalt be added in the form of drier, it dries with a clear film in 3—4 hrs. Their properties indicate that these oils could give satisfactory varnishes. S. S. WOOLF.

**Oiticica oil.** G. G. SWARD (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 120—125).—A review of the properties of oiticica oil and a bibliography are given, and the constants of two recent samples of the oil are reported. Spar varnishes made from these oils compare favourably with tung oil varnishes, both in appearance and character of film. Exposure tests are in progress. S. S. WOOLF.



**Bodied pilchard oils.** S. A. LEVY (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 139).—The constants of 4 samples of bodied pilchard oils, ranging in viscosity from 1.5 to 25 poises are tabulated. The oils were pale and had no pronounced odour. On refrigeration (8 hrs. at  $-15^{\circ}$ ), they showed opalescence, but did not deposit crystalline salts. S. S. WOOLF.

**Physical constants of oil from seeds of *Ribes rubrum*, L.** A. JERMSTAD (J. Pharm. Chim., 1931, 13, [viii], 243—244).—The seeds of Norwegian red currants (*Ribes rubrum*, L.) contain 20.4% of an oil having  $d_{20}^{20}$  0.9311,  $n_D^{20}$  1.4801, iodine value 176.3, saponif. value 193.3, and containing 1.8—2.3% of unsaponifiable matter of which 1% is phytosterol. F. O. HOWITT.

**Flax wax.**—See V. Linseed.—See XVI. Coconut oil.—See XIX.

#### PATENTS.

**Refining of fatty acids obtained by oxidation of paraffin wax or other high-molecular organic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,670, 23.10.29).—The products of the destructive oxidation of paraffin wax (etc.) with nitrogen oxides are heated at temperatures not above  $300^{\circ}$  with gaseous reducing agents, e.g., carbon monoxide, hydrogen, water-gas, under pressure if desired. If hydrogen be used, e.g., with a catalyst, or nascent hydrogen or electrolytic reduction, lower temperatures ( $50$ — $180^{\circ}$ ) may be employed. The reduced products are worked up by distillation etc. in the usual manner.

E. LEWKOWITSCH.

**Modification of the physical properties of thickened fatty oils.** L. AUER (B.P. 341,453, 8.7.29).—When thickened fatty oils (in which the disperse phase is in relatively high concentration) are used, simple dispersion of the modifying agent in the starting material is sufficient without prolonged heating. The modifying agents are those of the prior patents (for which see B., 1930, 652), particularly lithium carbonate or sulphite, *o*- and *p*-nitrophenols, benzoic acid, benzene-sulphonyl chloride, barium thiocyanate, chlorosulphonic acid, zinc bromide, naphthalene tetrachloride, 2:5-dichlorobenzenesulphonic acid. Dispersion is facilitated by using a solution of the modifying agent, e.g., in benzene, methyl ethyl ketone, etc. [Stat. ref.]

C. HOLLINS.

**Manufacture of sulpho-derivatives of the higher fatty acids.** I. G. FARBENIND. A.-G. (B.P. 342,761, 6.11.29. Ger., 6.11.28).—Lime- and acid-resistant derivatives of this type are prepared by refluxing the halogenated derivatives of fatty acids (or hydroxylated fatty acids), such as those described in B.P. 336,623 (B., 1931, 149), with aqueous sodium sulphite, preferably under pressure, and at temperatures of about  $100$ — $150^{\circ}$ . The products are water-soluble and liquid or solid according to the conditions.

E. LEWKOWITSCH.

**Flaker. Refining of oils etc.**—See I. Wetting agents.—See III. Varnishes.—See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Extraction of terpene chemicals from waste pine wood.** I. W. HUMPHREY (Inst. Chem. Eng., Mar., 1931. Advance proof. 11—16).—The stumps are

milled and extracted by the steam and solvent process to yield turpentine, pine oils, and resin. Known methods by which these products may be fractionated, purified, or converted into their derivatives are outlined.

T. McLACHLAN.

**Turpentine in varnishes and paints.** A. G. VAUDIN (Bull. Inst. Pin, 1931, 4).—The use of white spirit as a turpentine substitute is considered disadvantageous on the grounds of differences of distillation range, viscosity, solvent power, ease of working, drying power, surface tension, yield value, etc.

S. S. WOOLF.

**[Measurement of] hiding power [of paint].** S. A. LEVY (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 135—136).—The linoleum used in the "brushout" test previously described (cf. Circ. 362; B., 1930, 569) has been modified to conform to the following definition (A.S.T.M.): "A contrasting background of black and white, the black to be less than 8% in brightness, and the white to be  $80\% \pm 2$  in brightness." Using such a background, results of good concordance were obtained.

S. S. WOOLF.

**Effect of wood grain on paint durability.** H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 128—130).—Exposure tests indicate that flat-grain heart-side, or flat- and angle-grain sap-side, woods are not satisfactory woods to paint compared with vertical-grain wood. When other than vertical-grain woods are employed, aluminium, chromate, or graphite primers give better results than do white primers.

S. S. WOOLF.

**Red lead.** A. V. BLUM (Farben-Ztg., 1931, 36, 1045—1046).—The behaviour of red lead with linseed oil is not controlled entirely by its content of free litharge and by its degree of dispersion. The actual constitution of the particles themselves is a variable to be taken into account, as is indicated by microscopical examination. The existence of 6 types of particle of different activities is suggested, and these are illustrated by diagrams.

S. S. WOOLF.

**Lead tungate.** H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 133—134).—The preparation of lead tungate, by adding litharge and lead carbonate to heated tung oil fatty acids, is detailed and its solubility in various solvents and miscibility with oil, rosin, etc. are summarised.

S. S. WOOLF.

**"Non-fastness to oil" of mineral umbers.** A. EIBNER, R. SCHWARZ, and E. ROSSMANN (Farben-Ztg., 1931, 36, 962—964, 1006—1007, 1046—1049).—The question of saponification of fatty oils by basic pigments during the drying of oil paints is discussed at considerable length, the historical development of the conception of "soap-formation" in paints being traced, with special reference to lead pigments of different basicity. Experiments on the preparation of lead and zinc "soaps" at normal and raised temperatures are described, and the stoichiometry of the reactions is dealt with. Umbers are much less basic than litharge and zinc oxide, and their "non-fastness" is due to the formation of oleosols rather than to saponification. The formation of crystalline peroxides as intermediate products in the film formation of linseed and poppyseed



oils is deduced from a study of variously treated pastes of umbers in these oils. S. S. WOOLF.

**Varnishes for preserve tins.** G. HINARD (Peint. Fig. Ver., 1931, 8, 1394—1397).—The requirements of lacquers for tins intended to contain fruit, jam, vegetables, fish, etc. are described. Resistance to "tooling," "retorting," and the action of acids, and freedom from residual odour are important attributes. Tables of the approximate average content of malic and citric acid in fruits, and of oxalic acid in vegetables, are quoted. Fish, which is generally alkaline, is acidified with acetic or citric acid before tinning. Three proprietary preserve-tin lacquers were tested at 120° in 0.5*N*-solutions of lactic, tartaric, and the above-mentioned acids, and the results are described. Oxalic acid appears to be the most aggressive of the acids tested. Additions of common salt and sugar were tried without appreciable effect. It is pointed out that whilst any of the requirements of these lacquers can readily be met individually, a lacquer combining all the desiderata is necessarily expensive. S. S. WOOLF.

**Organic reagents for rapid detection of metals used in the varnish industry.** H. SCHEIBER (Farbe u. Lack, 1931, 36, 111—112).—Details are given of tests according to which iron, aluminium, manganese, cobalt, zinc, and magnesium may be detected by means of pyrogallol, alizarin, benzidine, 1-nitroso- $\beta$ -naphthol, resorcinol, and tetrahydroxyanthraquinone, respectively. These tests are then incorporated in a suggested systematic group analysis. S. S. WOOLF.

**Glass colour standards for varnish.** S. A. LEVY (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 140).—The original 9 glass colour standards described in Circ. 367 have been increased to 18 for closer approximations. The equivalents of the whole range in terms of potassium dichromate-sulphuric acid solutions and iodine-nickel sulphate solutions are tabulated. S. S. WOOLF.

**Alcohol-soluble nitrocellulose.** II. WOLFF [with B. ROSEN] (Farben-Ztg., 1931, 36, 964—966).—A range of samples of "alcohol-soluble" nitrocellulose of German and American origin was examined. For solutions above 10% concentration, a solvent mixture such as alcohol 50, benzol 40, diacetone alcohol 10 pts. is necessary. Butyl phthalate and shellac were incorporated (to the extent of  $\frac{2}{3}$  and  $\frac{1}{3}$  of the weight of nitrocellulose, respectively). The hardness, abrasion-resistance, elasticity, and tensile strength of films obtained from the various solutions were determined. S. S. WOOLF.

**[Nitrocellulose lacquer] solvents.** T. H. DURRANS (J. Oil & Colour Chem. Assoc., 1931, 14, 65—76).—The behaviour of solvents of low-, medium-, and high-boiling types in nitrocellulose lacquers is discussed generally, attention being given to evaporation rates, formation and effects of azeotropic mixtures, significance of mol. wt. of solvent in conjunction with its vapour pressure from the viewpoint of retention of solvent in film, effect of diluents on viscosity, water as a diluent, etc. A method for producing a lacquer of predetermined properties (involving the use of triangular co-ordinates) is briefly described. S. S. WOOLF.

**Lacquer diluents of the petroleum type.** J. R. STEWART (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 378, 143—153).—The requirements of aliphatic lacquer diluents are: non-objectionable odour, high flash point, satisfactory solvent power, and evaporation rate similar to that of coal-tar diluents. The kauri-butanol, aniline-point, methyl sulphate, and dilution-ratio methods of determining solvent power are detailed and the results obtained with them on a comprehensive range of commercial "toluol substitutes" are tabulated, it being shown that the four methods give results that may be correlated. The boiling ranges and evaporation rates of some of these diluents are also given. S. S. WOOLF.

**Recovery of gum from fossil kauri timber.** R. G. ISRAEL (Inst. Chem. Eng., Mar., 1931, Advance proof. 7—10).—In northern New Zealand exist immense tracts of land covered by the kauri tree (*Agathis australis*) which produces the fossilised resin. Portions of the wood contain about 20% of gum, to remove which the wood, after milling, is extracted, first with benzene and then with alcohol, thereby splitting the gum into a brownish fraction (resin acids, soluble only in alcohol and efficient as a substitute for shellac) and a pale gum. The latter, which is the more valuable, consists of resenes soluble in benzene or in alcohol-benzene and is suitable for nitrocellulose lacquers, in which a large percentage may be used owing to its low viscosity. T. McLACHLAN.

**Cativa resin.** L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1931, Circ. 377, 131—132).—A sample of Cativa resin (of the balsam type, from Panama—"Prioria copaiifera Griseb.") when dehydrated had acid value 113 and  $n_D^{20}$  1.5098. A tung-oil varnish made therefrom gave soft films, but the resin has possibilities for varnish manufacture, either in conjunction with other resins or after esterification. S. S. WOOLF.

**Nitrocellulose.**—See V. "Scheiber" oil. Oiticica oil.—See XII.

#### PATENTS.

**Manufacture of zinc white.** J. M. H. CORNILLAT (B.P. 344,087, 10.12.29).—The furnace flame is caused to develop in a U shape, so as to obtain the maximum use of the heat produced, and the zinc vapours are carried along at a high velocity. Means are provided for causing carbon dioxide to react with the zinciferous vapours as they are evolved. When impure zinc is used, it may be melted in an auxiliary furnace and the molten metal then led through a channel into the main furnace. W. J. WRIGHT.

**Manufacture of varnishes.** L. AUER (B.P. 341,431, 8.7.29).—Linseed oil, or other fatty oil and/or resin used in varnishes, is heated with less than 10% of an amine soap formed by interaction of a primary aromatic diamine and/or high-molecular monoamine with an equivalent amount of an organic isocolloid containing high-molecular acids or derivatives; there is produced an improved varnish base. Linseed oil, e.g., is heated at 250° with the benzidine soap of tung oil acids, and the product, after melting with Albertol resin, is made up into a water-resistant varnish with turpentine or white spirit. C. HOLLINS.

**Water-resistant varnishes.** H. WADE. From BAKELITE CORP. (B.P. 342,737, 6.11.29).—The water-resisting properties of oleoresin varnishes, *e.g.*, spar varnishes from tung oil, ester gum, or synthetic resins, are improved by incorporating solid chlorinated naphthalenes (*e.g.*, "Halowax," a mixture of tri- and tetrachloronaphthalenes) at any stage during manufacture of the varnish before the diluting solvents have been added.  
E. LEWKOWITSCH.

**Adhesives.** BRIT. CELANESE, LTD. (B.P. 342,674, 28.10.29. U.S., 27.10.28).—The adhesive comprises a mixture of cellulose derivative (ester or ether) and a soluble phenol-ketone resin (*e.g.*, the condensation product of diphenylolpropane and acetone), with or without additions of other compatible natural or synthetic resins; suitable low-boiling solvents, with or without admixed higher-boiling solvents and plasticisers are employed. The adhesive is suitable for all materials, including especially glass, stone, and asbestos.

E. LEWKOWITSCH.

**Means [adhesive paste or plaster] for preventing fires and rendering substances incombustible.** W. E. EVANS. From "S.A.I.G." (SOC. ANON. INVENZIONI GUADAGNIN) (B.P. 344,094, 13.12.29).—A fire-resisting composition for coating combustible materials or applying to metallic network consists of potassium or sodium silicate 50, asbestos 40, refractory earth 5, black lead 1, magnesium carbonate 1, and alum 3 pts.

W. J. WRIGHT.

**Production of condensation products [plastics] from polybasic acids and polyhydric alcohols.** H. A. BRUNSON, and CHEM. FABR. DR. K. ALBERT G.M.B.H. (B.P. 341,477, 11.10.29).—Soft, rubbery, plastic masses, suitable as plasticisers for cellulose esters (*e.g.*, nitrate), are obtained by heating glycerol (or other polyhydric alcohol) with an aliphatic dibasic acid having a normal chain of at least C<sub>7</sub>, namely pimelic, suberic, azelaic, and especially sebacic acids. Glycerol and sebacic acid at 165–170° give in 4½ hrs. a thin syrup which after a further 15 min. changes to a soft, rubbery mass, still soluble in acetone, and finally after another ½ hr. to a soft, tough, flexible solid, insoluble in common organic solvents. Cellulose nitrate plasticised with this product adheres well to rubber. Ethylene glycol condenses with sebacic acid in 30 hrs. at 240°. A harder product is obtained by adding colophony to the melt. The soluble products may be incorporated with phenol-aldehyde and other synthetic resins in a common solvent.

C. HOLLINS.

**Paint mills etc.**—See I. **Wetting agents.**—See III. **Azo lakes.**—See IV. **Greensand.**—See VII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**[Rubber] latex chemistry.** **Surface tension measurements with fresh latex and their relation to the non-crêpe constituents.** P. SCHOLZ (Kautschuk, 1931, 7, 42).—Examination of latex from various trees within 4 hrs. of tapping shows that the difference between the proportion of total solids present and of crêped rubber obtainable by coagulation falls in the inverse order to the surface tension (*cf.* Hauser and Scholz, B., 1927, 947; 1928, 61).  
D. F. TWISS.

**Investigation of factice [rubber substitute].** LABORATORIUM DER FIRMA DEUTSCHE OELFABRIK DR. GRANDEL & Co. (Kautschuk, 1931, 7, 48–54).—A detailed description is given of the procedure adopted for tests as to the physical characteristics and chemical composition of rubber substitute of the vulcanised-oil type.

D. F. TWISS.

**Reinforcing action of "pigment" mixtures on rubber compounds.** D. J. BEAVER and J. W. MACKAY (Ind. Eng. Chem., 1931, 23, 294–296).—Mixtures of channel black or a "soft" carbon black with whiting, lithopone, or clay show additive physical properties. Mixtures of "soft" carbon black with zinc oxide also show additive properties, but mixtures of channel black and zinc oxide exhibit poorer resistance to abrasion, higher modulus, and higher tensile strength than would be expected from a purely additive effect. This is due probably to reaction between the zinc oxide and the acidic compounds in the rubber or on the black, the medium thereby becomes more alkaline with consequent decrease in the dispersion of the channel black. In mixtures containing a high proportion of zinc oxide with a smaller proportion of channel black it is consequently possible with advantage to replace the channel black and also even part of the zinc oxide by an equal volume of soft black.

D. F. TWISS.

**Action of lead selenide in rubber mixtures.** R. DITMAR and K. H. PREUSSE (Caoutchouc et Gutta-Percha, 1931, 28, 15,448–15,450).—In unaccelerated rubber mixtures lead selenide assists softening, but otherwise acts as an inert filler. In accelerated mixtures it is a powerful activator and effective reinforcing agent and causes a marked increase in the abrasion-resisting qualities. Lead selenide is resistant to vulcanisation with sulphur chloride.

D. F. TWISS.

**Hard spots in vulcanised rubber compounds.** J. H. HOWEY (Ind. Eng. Chem., 1931, 23, 287–290).—The hardness of different portions of a small vulcanised slab of motor-tread rubber shows greater variation than does a mixture of the same composition after vulcanisation in a motor tyre. Regions where maximum flow or displacement occur during moulding and vulcanisation coincide with areas of excessive hardness. This is attributed to alteration in the condition of dispersion of the reinforcing agents or to a local grain effect induced by the flow.

D. F. TWISS.

**Testing of the tinning of rubber-insulated conductors.** A. VAN ROSSEM and P. DEKKER (Kautschuk, 1931, 7, 42–47).—The effectiveness of the tinning on rubber-coated copper wire may be assessed by determining the amount of copper in a definite area of the stripped rubber coating. A more direct method of assessment is by exposing the tinned wire, after dipping it in a 3:1 mixture of oleic and stearic acids, to the air at the ordinary temperature for 24 hrs. and then determining the amount of copper which has passed into the fatty acid. Other known methods are described, but the two preceding tests are the most satisfactory.

D. F. TWISS.

#### PATENTS.

**Manufacture of rubber-like products by modifying the physical properties of organic isocolloid**

materials containing unsaturated carbon compounds. L. AUER (B.P. 341,490, 8.7.29. Addn. to B.P. 318,562; B., 1930, 8).—To the modified fatty oil emulsion of the prior patent are added non-rubber constituents of natural latex, *e.g.*, stearic acid, casein, peptone, starch, etc., which are then precipitated upon the globules of emulsified oil by addition of sodium sulphate, cadmium iodide, or other precipitant. The whole is then further modified by gas treatment, *e.g.*, with air, hydrogen sulphide, or substances producing gas, *e.g.*, barium peroxide. Emulsions of rubber-like products are obtained. C. HOLLINS.

Perforated rubber sheet.—See I. [Rubbery] plastics.—See XIII.

### XV.—LEATHER; GLUE.

Hydration of goat skin. E. R. THEIS and A. GRYNKRAUT (J. Amer. Leather Chem. Assoc., 1931, 26, 134—143).—A distinction is drawn between the terms swelling and hydration used in tanning terminology. Hydration is measured by the contraction in volume of the skin and solution and is attributed to residual valencies. The hydration of dry-cured goatskin was measured in different solutions. It was shown that in water alone the hydration attained a maximum in 14 hrs., and with acids or alkalis in 20—24 hrs. The hydration was diminished by acids or alkalis. The hydration in formic acid solutions of different concentrations was a maximum at the isoelectric point and diminished as the concentration increased. The hydration was diminished and the swelling increased by rise in temperature. Two stages were noted in the hydration of dry goatskin: an initial slow period followed by a rapid period. Degeneration of the skin substance was practically stopped by formic acid solutions  $> 0.0075N$ , but the hydration was low in such solutions.

D. WOODROFFE.

Nature of the water-soluble [matter] in leather tanned with wattle bark extract. II. R. O. PAGE and H. C. HOLLAND (J. Amer. Leather Chem. Assoc., 1931, 26, 143—156; cf. B., 1929, 66).—Less water-soluble matter than tannin was combined with the hide in the early stages of tanning until the combined tannin amounted to 30 pts. per 100 pts. of hide substance, after which 30—60 pts. of combined water-soluble matter were found. This amount depended on the pretreatment of the hide, but was not increased by prolonged tannage, although the combined tannin could be increased to 60 pts. per 100 pts. of hide substance. The amount of combined water-soluble matter was diminished slightly and that of the combined tannin greatly increased by raising the temperature. The amount of combined water-soluble matter was independent of *pH* values between 3 and 8. The combined water-soluble matter was partly converted into combined tannin by treating the tanned leather with 0.2*N*-sulphuric acid at temperatures above 34°, and such converted water-soluble matter was replaceable by further tanning. It is suggested that combined water-soluble contents of 30 and 60, and amounts of combined tannin of 30, 60, and 90 pts. per 100 pts. of hide substance are definite compounds of collagen with tannin. D. WOODROFFE.

De-tanning and utilisation of chrome-leather waste. E. SAUER and W. ESCHMANN (Kolloid-Z., 1931, 54, 326—334).—For the manufacture of glue from chrome-leather waste the chromium content of the leather should be reduced as far as possible: complete removal is not possible and the resulting glue is always dark-coloured, through traces of chromium compounds. The chromium compounds in the tanned leather can be removed by treatment with acid or by oxidation to chromate. Alternate treatment with sodium hydroxide solution and dilute sulphuric acid is more effective than with acid alone. Treatment with a mixture of sodium hydroxide and hydrogen peroxide bleaches the leather and removes the chromium in the form of chromate.

E. S. HEDGES.

Wetting agents.—See III.

### XVI.—AGRICULTURE.

Soil structure. P. EHRENBURG (Z. Pflanz. Düng., 1931, 19A, 44—77).—A discussion of the formation and structure of aggregate particles of soil. An extensive bibliography is included. A. G. POLLARD.

Physical chemistry of cultivated soils. V. Determination of the nutrient content of soils by direct-current electrolysis. R. A. HERZNER (Z. Pflanz. Düng., 1931, 19A, 93—110; cf. B., 1931, 132).—Simple apparatus and technique is described for the electrolysis of soils. Comparison is made of the base content of soils, so determined, with that obtained by purely chemical methods. Relationships between the electrically removable bases, soil acidity, and adsorption potential are discussed. A. G. POLLARD.

Preparation of soil profiles for exhibition and soil study. R. C. COLLISON and J. D. HARLAN (N.Y. State Agric. Exp. Sta. Tech. Bull., 1930, No. 173, 8 pp.).—Methods and equipment are described for obtaining profile samples as long narrow strips or as cylindrical cores for storage in celluloid cases.

A. G. POLLARD.

Mechanical analysis [of soils]. G. W. ROBINSON (Imp. Bur. Soil Sci., 1930, Tech. Comm. No. 17).—Preliminary work on the comparative examination of methods is discussed. The International method is inadequate for soils rich in gypsum. The Puri method fails in highly unsaturated organic soils. The value of soda for rapid and efficient dispersion is confirmed, and the substitution of soda for ammonia in the International method yields excellent results.

A. G. POLLARD.

Mechanical analysis of soils by the pipette method. P. KÖTTGEN and H. HEUSER (Z. Pflanz. Düng., 1931, 19A, 257—281; cf. B., 1929, 369).—The sampling device of the apparatus described has two opposite lateral inlets and withdraws a sample from a substantially uniform and thin layer, with less disturbance of the sedimentation of the surrounding particles than the pipette of Köhn (B., 1928, 583; 1929, 787). Analysis of the heavier particles may be made by the use of highly viscous liquids, sugar solutions being particularly suitable for the purpose. A. G. POLLARD.

Effect of drying soil on its adsorption capacity. A. I. ACHROMEIKO (Z. Pflanz. Düng., 1931, 19A, 36—43).

—Drying did not markedly affect the adsorptive capacity of soils except when they contained appreciable quantities of calcium carbonate. This phenomenon is examined. The state of aggregation of dried soils does not retard the leaching of adsorbed bases. Base exchange occurs both on the surface of and within the soil granules.

A. G. POLLARD.

**Effect of burning on moorland soils.** E. G. SVINUFVUD (Wiss. Veröff. finn. Moorkulturvereins, 1929, No. 10, 95; Bied. Zentr., 1931, 60, 56–57).—During the burning of moorland, ammonia is produced both from the actually burning layer of peat and from that immediately beneath it. Most of this ammonia is reabsorbed by the unburnt peat. A mobilisation of mineral matter and also a reduction of soil acidity result from the burning. Soil bacteria generally are not injured, and in some cases are stimulated to increased activity. On land so treated the use of fertilisers is largely unnecessary.

A. G. POLLARD.

**Action of frost on soils.** E. JUNG (Z. Pflanz. Düng., 1931, 19A, 326–333).—Aggregation of soil particles by frost is not merely a mechanical effect of the expansion of water in freezing, but is controlled by the nature and course of the crystallisation of the soil solution. The phenomena therefore depend on the freezing temperature and the rate at which the soil solution freezes. With slow cooling there is a deposition of ice crystals in the tension-free pore spaces, and the possibility of the growth of these crystals at the expense of "bound" water in the soil is discussed. The effect of electrolytes on the freezing process is considered.

A. G. POLLARD.

**Heat of wetting of soils.** H. JANERT (Z. Pflanz. Düng., 1931, 19A, 281–309).—Apparatus and technique are described for determining the heat of wetting of soils. The heat of wetting decreases slightly with falling temperature of measurement, but does not become negative below 4°. Values obtained are influenced by the proportion of soluble matter in soils and by the degree of saturation with calcium. A hyperbolic relationship exists between the heat of wetting and the maximum saturation capacity of the soil for calcium. Deformation of soil blocks during drying was apparent only in soils with high heats of wetting. Relationships exist between the heat of wetting of soils and their clay contents, hygroscopicity, and other physical properties. The value of determinations of heats of wetting in the general examination of soil is indicated.

A. G. POLLARD.

**Buffer power of moor soils.** T. ARND [with W. SIEMERS and W. HOFFMANN] (Z. Pflanz. Düng., 1931, 19A, 334–342).—Types of moor soils are sharply differentiated by their buffer powers (Jensen). High-moor soils are strongly buffered towards bases and weakly towards acids, whereas low-moor soils are well buffered to acid and poorly to bases. Transitional types occupy an intermediate position. In moor soils the buffer power is closely related to acidity, base content (notably calcium), and the degree of saturation with bases.

A. G. POLLARD.

**Titanium in some New Zealand soils and pastures.** H. O. ASKEW (New Zealand J. Sci. Tech.,

1930, 12, 173–179).—The titanium contents of a number of soils are recorded. Soils reputed to be "bush sick" were characterised by low proportions of titanium. Heavy loams and clays contained more titanium than did sands. The titanium contents of pastures varied from 1.0 to 8.5 mg. per 100 g. of dry matter, and in no case were they sufficiently high to interfere with the phosphate assimilation of animals by precipitation of the insoluble phosphate. Variations in the iron and titanium contents of soils showed a general similarity. No relationship existed between titanium and manganese contents.

A. G. POLLARD.

**Mineral soil acidity.** M. TRÉNEL (Z. Pflanz. Düng., 1931, 19A, 238–241).—A discussion of current views of exchange acidity in soils in the light of the author's recent researches (cf. Trénel and Wunschik, B., 1930, 832).

A. G. POLLARD.

**Determination of the acid point of a soil, at which easily-soluble or exchange acidity appears.** S. GOY [with O. ROOS] (Z. Pflanz. Düng., 1931, 19A, 31–36; cf. B., 1929, 335).—The soil sample is shaken with N-potassium chloride and a measured amount of acid or alkali. After settling, the clear liquor is decanted and methyl-red added. By varying the proportion of acid or alkali used, a value is obtained at which the extract just causes the requisite colour change in methyl-red. This point marks the appearance of exchange acidity, and lies in the  $p_H$  range 4.7–5.5.

A. G. POLLARD.

**Soil acidity in relation to electrolyte content of soil suspensions.** A. TERÄSVUORI (Valiion Maat. Julkaisuja, 1930, No. 29, 7–200).—Except in a few cases, where the change seldom exceeded  $p_H \pm 0.2$ , keeping soil in dry air did not affect the acidity or the soluble electrolyte content. When much ferrous iron or other reducing substance was present, however, appreciable changes were observed. Heating soil samples at 100–110° for 3 hrs. generally increased the electrolyte content and lowered the  $p_H$ . The effect of various degrees of humidity are recorded. Changes in the  $p_H$  of fertilised soil do not indicate change in acidity.

CHEMICAL ABSTRACTS.

**Source of error in Trénel's quinhydrone acidimeter [for soil suspensions etc.] and its removal.** P. PFEFFER (Z. Pflanz. Düng., 1931, 10B, 57–63).—A new type of electrode is described by which the errors recorded by Laufer (B., 1928, 27) are obviated.

A. G. POLLARD.

**Changes in the colloid clay content of soils under the influence of cation exchange; centrifugal determination of colloidal clay.** E. UNGERER (Z. Pflanz. Düng., 1931, 19A, 111–118).—Colloidal clays of mean diam.  $< 0.2 \mu$  are separated centrifugally and examined. The dispersion of clay by univalent ions was in the order of the lyotropic series  $K < NH_4 < Na < Li$ .

A. G. POLLARD.

**Neutral salt decomposition by humus soils.** T. ARND (Z. Pflanz. Düng., 1931, 19A, 90–93).—Acidity developed in humus soils by treatment with neutral salts is not an adsorption interchange (cf. Behrens, B., 1930, 999), but is the result of direct double

decomposition of the neutral salt by organic acid, yielding insoluble organic salts and free mineral acid.

A. G. POLLARD.

**Distribution of fertilisers in soil by cultivation.** E. VOLZ (Z. Pflanz. Düng., 1931, 10B, 73—80).—Prior to its application the fertiliser is treated with a solution of anthracene in benzene, or other solvent in which the fertiliser is insoluble, and dried. The distribution of fertiliser after any cultural operation is observed by photographing a soil sample by ultra-violet light, the fluorescent anthracene particles being readily visible. Suitable sampling boxes are described.

A. G. POLLARD.

**Lime requirement of soils.** A. GEHRING (Z. Pflanz. Düng., 1931, 10B, 40—57).—Loams and clay soils of which the degree of saturation with calcium was less than 70 (Hissink) responded to liming. Results with light sandy soils were less definite, but in nearly all soils the maximum yields corresponded with applications of lime calculated to bring the calcium saturation to 70°. Larger applications reduced yields. Heavy soils of low organic content and with calcium saturation > 70 sometimes produced greater crops after liming, but the effect was due, not to any specific action of lime, but to improved mechanical condition which could be attained equally well by admixture with sand. Highly organic soils with calcium saturation > 70 produced smaller crops after liming. In general, marl, quick and slaked lime, and defecation lime were equally effective, but the fineness of grinding of the material was of prime importance. Potato scab appeared in soils with calcium saturation of 40 approx., and increased in intensity with added lime.

A. G. POLLARD.

**Soil reaction and lime requirement.** P. E. TURNER (Imp. Bur. Soil Sci., 1930, Tech. Comm. No. 17).—A summary and discussion are given of a paper already abstracted (B., 1931, 82).

A. G. POLLARD.

**Determination of exchangeable bases and lime requirement [in soils].** ANON. (Imp. Bur. Soil Sci., 1930, Tech. Comm. No. 12).—Details of existing methods and recent modifications are given together with an extensive bibliography.

A. G. POLLARD.

**Solubility of the adsorptively bound bases in soils in relation to their lime condition and degree of saturation.** A. GEHRING, O. WEHRMANN, and A. WOLTER (Z. Pflanz. Düng., 1931, 19A, 77—90).—The solubility of adsorptively bound potash in soil increases with the degree of saturation of the soil with potassium, with calcium, or with ammonium, the effect, in the case of the last two bases, being greatest where the proportion of adsorbed potassium is high. Neubauer tests confirm these results in soils in which the degree of saturation with calcium reaches 60—70%. Above this point unsatisfactory factors intervene to injure the seedlings. Increasing saturation of soil with calcium tends to increase the solubility of adsorbed magnesium, but the degree of saturation with ammonium or potassium does not appreciably influence magnesium solubility. The solubility of adsorbed potassium is not markedly affected by changes in the degree of saturation with magnesium or sodium. The effect of calcium in increasing the solubility of potash is almost eliminated by pre-

treatment of the soil with 0.05N-hydrochloric acid to remove adsorbed basis.

A. G. POLLARD.

**Phosphate status of soils.** L. MEYER (Landw. Jahrb., 1931, 73, 119—138).—Comparison of the phosphate requirement of numerous soils as determined by (a) the seedling method, (b) field trials, and (c) von Wrangell's water-extraction method (B., 1930, 833, 1166) confirmed the satisfactory nature of method (c). This method not only indicates the immediate phosphate requirement of soils, but gives information as to its general phosphate status. Changes in the phosphate contents of soils following cropping or fertilisation are accurately reflected in the water extracts.

A. G. POLLARD.

**Causes of the action of basic slag [in soils].** A. WILHELMJ (Z. Pflanz. Düng., 1931, 19A, 129—238).—The active phosphatic constituent of basic slag is soluble in water at  $pH$  7.0. Its solubility is somewhat depressed by weak acids and considerably reduced in water containing appreciable quantities of lime. In successive, neutral, aqueous extracts of slag, phosphate appears conjointly with calcium and silica. Slags containing 74% of soluble silicic acid show a relatively high phosphate solubility in initial extracts. Differences in the soluble silica contents of slags are reflected in marked differences in phosphate assimilability in Neubauer tests. In culture tests on acid soils slags were superior to superphosphate or Rhenania phosphate, the best results being produced by slags having the highest proportions of soluble silica. On loams containing chalk, slags with high soluble silica contents were as effective as was superphosphate or Rhenania phosphate, but low-silica slags were definitely inferior. The percentage utilisation of phosphate was greatest in slags of high soluble silica content, especially when the after effects on succeeding crops were included. Only the water-soluble phosphate of slags is utilised by plants, but the rate of dissolution is sufficient to satisfy the plants' needs. Slag forms a satisfactory top-dressing, provided sufficient moisture is present to dissolve its phosphate contents. Tetracalcium phosphate is as effective a source of phosphorus for plants as monocalcium phosphate. The former is decomposed by water to give dicalcium phosphate and calcium hydroxide, hence its retarded solubility in successive water extracts. The active phosphatic constituent of slag is the double salt of tetracalcium phosphate and calcium metasilicate, which is decomposed into its constituent salts by water, and plants utilise the aqueous solution of dicalcium phosphate finally produced. The rate of action of slag depends primarily on the rate of decomposition of the double salt, and this is conveniently measured by extraction with 2% citric acid solution.

A. G. POLLARD.

**New fertiliser "Nolitt."** E. BLANCK, F. GIESECKE, and F. KLANDER (J. Landw., 1931, 79, 55—68).—Chemical analysis of and culture trials with "Nolitt" are recorded. Increased crop growth produced by the fertiliser does not exceed that attributable to its nutrient contents. It has no other specific action. A mixture of powdered stone, ammonium sulphate, crude potash salts, and basic slag has similar properties.

A. G. POLLARD.

**Evaluation [as fertiliser] of the phosphoric acid of Rhenania phosphate.** H. NIKLAS, A. STROBEL, W. SCHROPP, and K. SCHARRER (Agric. Chem. Inst. Hochsch. Landw. u. Brauerei, 1930; Bied. Zentr., 1931, 60, 59—61).—Pot-culture experiments with numerous crops and varied soil types showed that the citrate-soluble phosphate of Rhenania phosphate is practically as efficient as the water-soluble phosphate of superphosphate and superior to the citric acid-soluble phosphate of basic slag. Distinct and superior qualities are attributed to the citrate-soluble phosphate of Rhenania phosphate as compared with the citric acid-soluble portion.

A. G. POLLARD.

**Comparative pot experiments with "Nitrophoska I.G. III."** K. ENTRES (Z. Pflanz. Düng., 1931, 10B, 103—108).—In pot trials with oats on both weakly acid and weakly alkaline soils, Nitrophoska proved as efficient as other complete fertiliser mixtures in which the nitrogen was supplied as ammonium sulphate, calcium cyanamide, urea, or any of the commonly used nitrates.

A. G. POLLARD.

**Fertilising action of phosphorites and their direct use in agriculture.** U. PRATOLONGO (Giorn. Chim. Ind. Appl., 1931, 13, 1—6).—The results of five years' field and pot experiments show that acidity of the soil, richness in organic constituents, and a plentiful supply of water, either separately or in conjunction, are not sufficient to ensure that phosphorites may exert fertilising action comparable with that of the soluble phosphates, either technically or economically. In soils of high acidity, the phosphorites exert an appreciable fertilising action, but this is always surpassed by that of soluble phosphates, such as superphosphate and basic slag. In neutral and especially in alkaline soils, phosphorites exhibit scarcely any direct fertilising action (cf. Tommasi and di Delupis, B., 1931, 215).

T. H. POPE.

**Measuring [soil]-absorbed phosphates and nitrogen.** W. J. SPILLMAN (Science, 1931, 73, 215—216).—A method for determining the amounts of phosphate apparently absorbed by soil and held in a condition unavailable to a growing crop is described. In accurate work, an amount of fertiliser equal to this quantity should be applied to the control plot.

L. S. THEOBALD.

**Influence of the C : N ratio of various organic substances on the nitrogen cycle in soil.** H. ENGEL (Z. Pflanz. Düng., 1931, 19A, 314—325).—The mineralisation of the nitrogen of fresh yard manure proceeds slowly, no nitrate formation being apparent in 4 months. With green manures the change is more rapid and the amount of nitrate formed exceeds that corresponding to the ammoniacal nitrogen content of the original material. Nitrogen losses during mineralisation are greater in the case of green manures than for cattle manure. Delayed nitrification of cattle manures is attributed to the difficulty of decomposition of some of the organic nitrogenous constituents and not to any particular C : N ratio in the manure tending to inhibit the activity of the organisms. The part played by denitrifying organisms in the process is discussed.

A. G. POLLARD.

**The potash reserve of field soils after unbalanced manuring, as determined by the *Aspergillus***

**method of Niklas, Poschenrieder, and Trischler.** E. KIESSLING (Z. Pflanz. Düng., 1931, 19A, 241—250).—Results of the *Aspergillus* method for determining the available potash in soil (B., 1931, 37) were generally as anticipated. Applications of nitrogenous and phosphatic fertilisers affect the yields of mycelium obtained. Seasonal variations in the potash reserve of soils indicated by this method are greater in soils rich in potash than in those of moderate or small potash contents.

A. G. POLLARD.

**Dependence of the physiological reaction of potash salts on hydrogen-ion concentration.** M. GÓRSKI and O. DĄBROWSKA (Poln. Jahrb. Land- u. Forstwirtschafts., 1930, 24, 46; Bied. Zentr., 1931, 60, 64—65).—Potassium chloride solutions, the  $p_H$  values of which were adjusted by means of potassium carbonate or hydrochloric acid, were physiologically acid to plants in the range  $p_H$  7.1—5.7 and physiologically alkaline at  $p_H$  4.4. Hydrogen-ion concentration affects the physiological reaction of salt solutions directly, and also indirectly through its effect on the intake of anions by plants.

A. G. POLLARD.

**Influence of potash sources and chlorine content of fertilisers on yield of cotton.** J. J. SKINNER (J. Amer. Soc. Agron., 1931, 23, 13—21).—Kainite is not so good a source of potassium as potassium chloride or sulphate; the chlorine in the quantity used is not unfavourable. The chlorine endurance limit for cotton is about 118 lb. Cl per acre. CHEMICAL ABSTRACTS.

**Potash fertiliser experiments [on tobacco].** ANON. (Conn. Agric. Exp. Sta., Bull. 311, 1930, 207—215).—In experiments using different potassium salts at rates to give up to 300 lb. of potash per acre, it was found that, even on a soil containing an enormous reserve of potash, the percentage of potassium in tobacco leaf is appreciably affected by the quantity applied to the soil in the fertiliser. Potassium deficiency in the leaf was very pronounced on the no-potash plots, especially after two years; the application of 200 lb. of potash per acre remedied the deficiency. Decrease in the leaf potassium is accompanied by an increase in both calcium and magnesium. The fire-holding capacity of leaf, as measured by the strip test, increases as the ratio of potassium to calcium and magnesium increases. The source of potash had little effect on the quality of the tobacco raised, except that the use of the sulphate resulted in an increase of sulphate in the leaves, which is injurious to burn.

E. HOLMES.

**Curing of yellow tobaccos. II. Changes in the nitrogenous compounds during starvation.** A. I. SMIRNOV and V. P. IZVOSCHIKOV (U.S.S.R. State Inst. Tobacco Invest., 1930, Bull. 71, 21 pp.).—During the starving period (barn ripening), which is the first stage in the curing of tobacco, the protein content of the leaves diminishes by 23% (old leaves) or by 35% (young leaves), the proteins of the young leaves becoming richer in basic constituents and thus approaching in composition the proteins of the old leaves. At the same time the polypeptides soluble in hot water rapidly become poorer in bases and richer in amide nitrogen, the total amount of polypeptides increasing somewhat. Hydrolysis of the water-insoluble part of tobacco on prolonga-



tion of the starving period is accompanied by decrease, and that of the aqueous extract by increase, in the formation of melanin. Part of the amino-acids and bases liberated from the polypeptide complexes undergoes decomposition with formation of ammonia, but a proportion of this is used in the formation of amide nitrogen and, during the early stages of the starvation period, of bases. The nicotine content of the leaves diminishes.

T. H. POPE.

**Boron as a regulator of the growth of tobacco, in relation to the reaction of the nutrient solution and the source of nitrogen.** A. I. SMIRNOV (U.S.S.R. State Inst. Tobacco Invest., 1930, Bull. 70, 29 pp.).—Experiments with *Platana arcadia* show that cultivation of tobacco in solutions of common nutrient salts free from boron results in a characteristic depression of the normal development. The function of the boron in regulating the growth concerns mainly the stem and only slightly, if at all, the roots or leaves. The storage of nitrogen compounds by the plants is not influenced by the presence of boron in the solution and depends solely on the reaction of the nutrient solution and on the nature of the source of nitrogen.

T. H. POPE.

**Effect of fertilisers on the growth and oil content of a variety of linseed (*Linum usitatissimum*, L.).** A. MOHAMMAD (Agric. J. India, 1930, 25, 471—486).—The effects of sodium nitrate (82 lb. per acre), potassium sulphate (164 lb.), superphosphate (164 lb., grade not stated), and a mixture of these three, on a variety of linseed (T. 124) grown for 1 year in lysimeter pots are described in detail. Sodium nitrate and the complete fertiliser gave increase in height, number of basal branches, and yield of seed, whilst the oil content of the seeds, which in the same variety varies from year to year, was not appreciably affected by any treatment.

E. HOLMES.

**Utilisation of coals as fertilisers.** R. LIESKE (Brennstoff-Chem., 1931, 12, 81—85. Cf. Bottomley, B., 1920, 200 A; Olsen, B., 1930, 434; Kissel, B., 1930, 877).—The addition of small quantities of brown coal to a pure culture of duckweed (*Lemna major* and *L. minor*), and to the soil in which maize, oats, tobacco, and tomatoes were growing, greatly stimulated the growth of these plants. The optimum results were obtained with an addition of about 1% of brown coal to the soil. A further improvement was brought about by first treating the brown coal with ammonia. The effect of adding a bituminous coal was very much less marked. The cause of the fertilising action of brown coal is still obscure. Olsen attributed it to the presence of complex iron salts, but the results of his experiments cannot be regarded as conclusive. "Humunit" and "Kalkhumat," commercial fertilisers prepared from brown coal, have been shown to be very active in stimulating plant growth.

A. B. MANNING.

**Paper mulch for the vegetable garden and its effect on plant growth, soil moisture, nitrates, and temperature.** R. MAGRUDER (Ohio Agric. Exp. Sta. Bul., 1930, No. 447, 60 pp.).—Effects of mulching a number of crops with various papers are described. Crop yields were increased in many cases, and, in general, the quality of the crop was improved. Paper mulching

increased the average moisture content and temperature (by 3—4°) of the soil, but no consistent differences in nitrate content between cultivated and paper-mulched soils were observed.

A. G. POLLARD.

**Residual effect of fertilisers.** F. A. WYATT (Sci. Agric., 1931, 11, 387—396).—Cereal crops were not appreciably affected by the residual effect of fertilisers other than farmyard manure, but clover crops showed definite gains both in yield and in the percentage of phosphorus in the crop. When the clover was ploughed in, the beneficial residual effect was passed on to the subsequent crop.

A. G. POLLARD.

**Establishment of grasses on very acid moorland.** R. B. DAWSON and T. W. EVANS (J. Min. Agric., 1931, 37, 1188—1191).—A series of seed mixtures failed, with the exception of wavy hair grass (*Aira flexuosa*), to germinate on moorland soils of  $p_H$  about 4.0, whereas all mixtures germinated and became established almost as well on plots receiving lime at the rate of 1 ton per acre as when 8½ tons per acre were applied.

E. HOLMES.

**Basis of chemical stimulation effects in higher plants.** A. NIETHAMMER (Z. Pflanz. Düng., 1931, 13A, 343—352; cf. B., 1929, 694).—Freezing of seeds increased their germinative power and mechanically increased the permeability of the seed coat to chrysoidin. Exposure of seed to an atmosphere containing chloroform also improved germinative power and increased permeability. Prolonged exposure to light increased the percentage germination of old seed, but on new seed (1 and 2 yrs.) the effect was reversed, and the period of germination lengthened. Chemical stimulation depends on the nature and condition of the seed.

A. G. POLLARD.

**Effect of various treatments on micro-organisms in the soil under a five-year rotation.** P. E. BROWN and B. A. TOMLIN (Proc. Iowa Acad. Sci., 1929, 36, 79—85).—Manuring, liming, and addition of rock phosphate, superphosphate, and crop residues stimulate bacterial development, nitrification, and non-symbiotic nitrogen fixation; the increase in crop yield may therefore be partly due to this stimulation.

CHEMICAL ABSTRACTS.

**Bacterial activity of soils variously treated.** P. E. BROWN and D. I. AQUINO (Proc. Iowa Acad. Sci., 1929, 36, 87—91).—A direct relation between activity and numbers of bacteria and the crop-producing power of soil, as affected by manuring, is indicated.

CHEMICAL ABSTRACTS.

**Chemical and microbiological processes in the decomposition of plant residues in soils.** S. A. WAKSMAN (Z. Pflanz. Düng., 1931, 19A, 1—31).—A general review and discussion of recent work.

A. G. POLLARD.

**Biological oxidation of sulphur. IV. Influence on ammonification and nitrification in activated sludge.** C. V. R. ARYAR (J. Indian Inst. Sci., 1930, 13A, 165—171; cf. B., 1930, 295).—Suspensions of sulphur in activated sludge inhibit the production of nitrates, but after an initial lag period ammonification proceeds vigorously because of an increase in fungoid growths. The acidity gradually increases to  $p_H$  2.4 and produces a floc which settled readily on keeping. The



effluent obtained after moderate aeration in the presence of sulphur contains less nitrogen than one from activated sludge alone, but the latter is not so effective in conserving plant nutrients in the sludge. C. JEPSON.

**Straw manuring.** W. JESSEN and E. GERDUM (Z. Pflanz. Düng., 1931, 10B, 97—102).—Reduction of crop yields following the ploughing-in of straw may be satisfactorily counteracted by applications of nitrogenous fertilisers. The requisite amount of nitrogen for the purpose cannot, however, be determined in advance.

A. G. POLLARD.

**Effect of calcium cyanamide manuring on the yield, reaction, buffer capacity, and degree of saturation with bases of strongly acid soils.** L. SCHMITT (Z. Pflanz. Düng., 1931, 10B, 1—40).—Contrary to general opinion, calcium cyanamide gave satisfactory results on some very acid soils with a number of crops. When used in conjunction with superphosphate and kainite on both limed and unlimed soils, higher crop yields were obtained than with ammonium sulphate. The favourable effects of cyanamide are attributed to its ability to improve reaction conditions,  $pH$ , buffer capacity, and degree of saturation with bases of acid soils. In this respect it is superior to sodium nitrate or basic slag.

A. G. POLLARD.

**Influence of the permanent level of farmyard manure applications on crop yield and fertiliser action.** EICHINGER (Z. Pflanz. Düng., 1931, 10B, 81—97).—Collected data from numerous farms show that the average yield of oats increases with the level of permanent applications of farmyard manure, the latter being judged by the number of fully-grown cattle carried by the farm per unit area of arable land. The relationship is much more definite and regular on soils of good adsorptive capacity than on light sands. Crop yields from sandy soils, even when heavily manured, are consistently lower than those from good loams. In general, the efficiency of fertilisers decreases with a rising level of farmyard manuring.

A. G. POLLARD.

**Rate of decomposition of some artificial manures.** F. B. SMITH (Proc. Iowa Acad. Sci., 1929, 36, 69—73).—Adco reagent at first stimulates the production of carbon dioxide, which then falls to the normal value. With Iowa reagent there was a slight increase in carbon dioxide production and a considerable loss of nitrogen as ammonia.

CHEMICAL ABSTRACTS.

**Factors influencing the effectiveness of organic mercury dusts in pea-seed treatment.** L. K. JONES (J. Agric. Res., 1931, 42, 25—33).—Organic mercury dusts containing not less than 12% of mercury phenoxide were the most successful of those examined. Watering seed beds immediately after sowing considerably reduced the germination of untreated seed. Dusting improved germination at all soil-moisture contents.

A. G. POLLARD.

**Removal of [arsenical] spray residues from apples. Wax-solvent method.** J. R. NELLER (Ind. Eng. Chem., 1931, 23, 323—325).—The removal of arsenical residues from sprayed fruit by washing with dilute hydrochloric acid at moderate temperatures is not satisfactory when the fruit has also been oil-sprayed or has become waxy in storage. Pretreatment of the

fruit with a wax solvent reduced the residue by about 90%. Thus unwashed fruit having an arsenic residue of 0.035 grain/lb. (as  $As_2O_3$ ) after ordinary treatment with dilute acid still retained 0.019 grain/lb. (tolerance limit 0.010 grain/lb.), but when dipped in acetone for 1½ min. and then treated with 0.1% hydrochloric acid at 20°, retained only 0.0018 grain/lb. Petroleum was unsatisfactory as a wax solvent, whilst under the same conditions diacetone alcohol and synthetic methyl alcohol gave residues of 0.0054 and 0.0041 grain/lb., respectively. A 75% solution of diacetone alcohol was not satisfactory. Another variety of waxy fruit, having in the unwashed state an arsenical load of 0.039 grain/lb., when dipped in methyl alcohol for 1 min. and then washed with 0.7% hydrochloric acid at 32° retained only 0.0034 grain/lb. Dilution of the methyl alcohol greatly reduced the efficiency of the treatment. The practical applications of the pre-dip treatment are discussed.

H. J. DOWDEN.

**Methods of field experimentation.** J. WISHART (Imp. Bur. Soil Sci., 1930, Tech. Comm. No. 17).

**Flax residues.**—See V. **Solubility of calcium phosphates.**—See VII.

PATENT.

**Manufacture of phosphatic manures.** B. BODRERO (B.P. 343,125, 8.11.29. Fr., 13.11.28).—If finely-powdered tribasic calcium phosphate is incorporated with a solution of colloidal sulphur, a product is obtained in which sulphur and calcium phosphate are uniformly distributed, acid monocalcium phosphate being absent, and in which the proportion of sulphate is insufficient to injure the soil.

W. J. WRIGHT.

## XVII.—SUGARS; STARCHES; GUMS.

**Pectin and "plus-sugar."** COLIN, BILLON, and MIOSSEC (Bull. Assoc. Chim. Sucr., 1931, 48, 16—20).—Values for "plus-sugar" (the amount by which the sugar in the extracted juice exceeds that in the sugar-beet pulp before extraction) depend only on polarimetric measurements, and are obtainable when the extraction is carried out at temperatures up to 90—96°. It is suggested that under these conditions the pectic materials of the sugar beet are soluble and affect the rotations.

B. W. TOWN.

**Causes of error in the determination of sugar in the beetroot.** J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 455—459).—The author reviews the history of this subject and concludes that the methods of alcoholic extraction and hot aqueous digestion, with the limited amounts of basic lead acetate usually prescribed and not too prolonged heating, give the most trustworthy results at present obtainable.

J. H. LANE.

**Determination of reducing sugars in presence of sucrose by potassium ferricyanide.** R. HAMY (Bull. Assoc. Chim. Sucr., 1930, 47, 385—387).—In the method described by Jonescu and Vargolici (B., 1920, 829 A), the volume of 0.5% dextrose solution required by 10 c.c. of the reagent was found to be quite unaffected by the presence of sucrose at concentrations up to 30%. Applied to raw beet juices rich in invert sugar and to diluted beet molasses, after clarification with basic

lead acetate and removal of excess of lead, the method indicated percentages of reducing sugar before and after Clerget hydrolysis in close agreement with those found by Bertrand's method. According to Jonescu and Vargolici, 10 c.c. of their reagent correspond to 0.050 g. of dextrose, 0.0491 g. of invert sugar, 0.0637 g. of maltose, or 0.0676 g. of lactose. J. H. LANE.

**Electrical conductivity and ash content of sugar factory products.** K. ŠANDERA (Bull. Assoc. Chim. Sucr., 1930, 47, 446–149).—A description, with diagrams, of the author's conductivity apparatus in which the balancing of resistances is effected by comparison of the illumination from two electric lamps (cf. B., 1927, 312, 920; 1929, 695). J. H. LANE.

**Adsorption from sugar solutions. III. Adsorption in the carbon layer: acetic acid.** J. VÁCHA and J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1931, 55, 334–338).—The influence of temperature, pressure, and the nature of the solution on the velocity of flow,  $v$ , of water through activated charcoal has been determined for a number of different specimens. With increase of particle size  $v$  increases, whilst  $1/v$  is related exponentially to the applied pressure; the influence of temperature is relatively greater at the lower pressures. Data are given of the relative rates of flow of water and aqueous solutions of sucrose, molasses, and crude sugar. Measurements of the adsorption of acetic acid by active carbon confirm Dèdek's theoretical views on the mechanism of the adsorption (cf. B., 1927, 920). With a constant ratio of adsorbent to solution the maximum adsorption is the same whether determined by the suspension or the layer method. H. F. GILLBE.

**Mill sanitation and [sugar-]juice deterioration.** R. H. KING (Sugar News, 1930, 11, 640–655).—The acidity of sugar-cane juice increases rapidly after 2 hrs., the  $p_H$  decreasing less rapidly. The changes are related to the quality of the cane and the maceration water. Decomposition is minimal when hot water is used at 3-hr. intervals, and bagasse and sediment are periodically removed. The use of deep, sloping pans and troughs, but not of chemicals, is recommended.

#### CHEMICAL ABSTRACTS.

**Hydrolysis of inulin under pressure.** E. C. KLEIDERER and D. T. ENGLIS (Ind. Eng. Chem., 1931, 23, 332–334).—Attempts have been made to hydrolyse pure inulin by heating with water alone, so as to avoid contaminating the syrup with the products of the hydrolytic catalysts. When heated at 100° for 40 hrs. in a sealed tube the extent of the hydrolysis, as indicated by the copper-reduction method, was about 12%, and on a steam-bath under reflux for a like period there was a 15% conversion. In order to improve the yield, carbon dioxide under pressure was then used to catalyse the hydrolysis. At 150° and 70–80 atm., hydrolysis was complete in 1 hr. and about 92% at 130° in 2½ hrs. The reaction is influenced more by temperature than by pressure, since changing the pressure from 70 to 20 atm. at 130° reduced the apparent conversion in 1 hr. from 65 to only 55%. When nitrogen was substituted for carbon dioxide the percentage hydrolysis was slightly reduced, being 86% in 1 hr. at 160° and 70 atm. Varying the pressure of nitrogen from 46 to 93 atm. had practically

no influence on the yield, with constant temperature. Sulphur dioxide at 3 atm. gave complete conversion in 30 min. at 145°, but there was serious corrosion of the reaction vessel. The syrups obtained had a satisfactory appearance and taste, but no attempt was made to produce crystalline levulose. H. J. DOWDEN.

**Measurement of foam. Molasses separator.**—See I. Cane sugar in wine.—See XVIII. Water in foods.—See XIX.

### XVIII.—FERMENTATION INDUSTRIES.

**[Alcohol from] distillation of bananas.** E. L. DE LAPERSONNE (Bull. Assoc. Chim. Sucr., 1931, 48, 28–32).—Details are given for the preparation of fermented liquors both from green and ripe bananas. The must from the ripe banana, after sterilisation, is fermented with barm from fermented cocoa. The green fruit, which consists mainly of starch, is fermented with a *Mucor* in symbiosis with a yeast; fermentation is rapid and a 97.5% yield of alcohol is obtained. B. W. TOWN.

**Nitrogenous substances in beer.** I. S. AYAI (J. Soc. Chem. Ind. Japan, 1931, 34, 72–74 B).—Japanese lager beer kept in bottle for two months remained bright, but became hazy when the time of storage was extended to 17 and 29 months. The contents of total nitrogen and protein nitrogen decreased during storage, whilst that of non-protein nitrogen remained almost constant. Expressing the contents of nitrogenous compounds as percentages of the total nitrogen, the coagulative protein nitrogen decreased during storage from 16.8 to 15.0%, the albumoses decreased from 27.0 to 23.7%, peptones increased from 39.8 to 42.6%, free amino-acid nitrogen increased from 4.5 to 10.0%, and amide nitrogen decreased from 11.6 to 8.5%. C. RANKEN.

**Rôle of the iron in wine and other fruit products.** A. HANAK (Z. Unters. Lebensm., 1930, 60, 291–297).—The deepening of the colour of wine on exposure to ultra-violet light is caused by the alternate reduction and oxidation of the iron present. Total iron in wine may be determined on the ash, or directly in the case of light-coloured wines. In the former case the wine residue is gently incinerated, the ash dissolved in hydrochloric acid, and iron determined in the filtrate by the usual colorimetric method. In the direct method 25 c.c. of wine are treated with 0.7–1.5 c.c. of 0.5% potassium ferrocyanide solution and acidified with dilute nitric acid, gum arabic solution being added to prevent the formation of a precipitate. To a second 25-c.c. portion of the same wine 0.7 c.c. of 0.5% potassium ferrocyanide solution, 1 drop of 1% ferric chloride, and a little mineral acid are added in the order given. If this second solution is not deeper coloured than the first it should be discarded and another prepared using 1 c.c. (or more if necessary) of the ferrocyanide solution. When the required depth of colour is attained, gum arabic is added to the solution and the whole is diluted to the same intensity of colour as that of the first solution, a dilution colorimeter being used for making comparisons. If necessary a solution of a suitable yellow dye may be added to the diluted solution to compensate for the coloration due to the wine itself. The volumes of the

two solutions are measured. The iron equivalent of the potassium ferrocyanide added to the second solution is calculated and from the volumes of the solutions the iron present in the first solution is found. In determining ferrous iron, 25 c.c. of wine are treated with 2 g. of potassium or ammonium thiocyanate and 1 c.c. of dilute (1:10) hydrochloric acid. To another solution similarly prepared a few drops of nitric acid are added. The second solution is diluted until the colour intensity is equal to that of the first. Then, if  $V_1$  and  $V_2$  be the volumes of the two solutions,  $V_1/V_2$  = ferric iron/total iron. The method cannot be used for deeply coloured wines.

W. J. BOYD.

**Rapid determination of volatile acidity in wine.** M. MARTIN (Ann. Falsif., 1931, 265, 36—37).—A new apparatus for steam-distillation is described consisting of a device for bubbling steam through 10 c.c. of the sample in such a way that the volume is kept constant, the supply being cut off automatically after 20 min. The fixed acidity is determined on the residue, the volatile acidity being obtained by difference from the total acidity.

E. B. HUGHES.

**Determination of sucrose in sweet wines.** H. JAHR (Z. anal. Chem., 1931, 83, 321—338).—The official method for the determination of sucrose in wine gives correct results only if the lead acetate is added after the wine has been made slightly acid with acetic acid. The following simpler method of analysis is recommended: a quantity of wine equal to 3000/ $\epsilon$  c.c., where  $\epsilon$  is the content of solids in g./litre, is weighed and diluted to 100 c.c. with water in a graduated flask. The liquid is transferred to a larger dry flask and shaken for 10 min. with 1.5 g. of freshly-burned bone charcoal; 25 c.c. of the filtered solution are neutralised with *N*-sodium hydroxide and the reducing sugars determined with Fehling's solution. A further 50 c.c. of the filtrate are diluted to 75 c.c. and the sucrose is inverted in the usual way; after dilution to 100 c.c., 50 c.c. are removed and diluted to 150 c.c., and 50 c.c. of this solution are neutralised with *N*-sodium hydroxide and the total sugar content is determined as before. The difference between the two results is calculated to sucrose.

A. R. POWELL.

**Enzymic clarification of unfermented apple juice.** Z. I. KERTESZ (N.Y. State Agric. Exp. Sta., Bull., 1930, No. 589, 10 pp.).—A pectin-destroying enzyme obtainable from *Penicillium glaucum* and other moulds is used to clarify apple juice. Coagulated colloidal matter is removable by filtration.

A. G. POLLARD.

**Determination of the quality of spirit by the Barbet test.** B. LAMPE (Z. Spiritusind., 1931, 54, 69—70).—For the test 50 c.c. of spirit are mixed with 2 c.c. of a solution of potassium permanganate containing 0.2 g. per litre, and the time required for the colour to change to pale salmon is determined at 18°. For a spirit of "good quality" 20 min. is chosen as the standard. The value of the test is limited, as the time required for the colour change is prolonged with any increase in the storage time of the spirit. An example is given of a spirit which showed no lessening of undesirable flavour and odour at the end of a year, i.e.,

showed no improvement of quality, but with which the original time of 11 min. required for the colour change increased to 30 min.

C. RANKEN.

**Distillation of [fermented juice from] apples.** J. VIDAL and P. VIDAL (Bull. Assoc. Chim. Sucr., 1930, 47, 393—404).—In distilleries operating on apples the yields of alcohol commonly correspond to less than 80% of the sugar in the freshly-pulped fruit, as determined by copper reduction, and only a small proportion of the deficit is accounted for by the reducers remaining in the exhausted pulp and the vinasses. Careful control of well-conducted distilleries showed 1—3% of the original reducers in the exhausted pulp and 87—89% in the juice before fermentation, i.e., a loss of about 10% not accounted for in the extraction process. Analysis of the fermented juice revealed a further loss of 6—8% not accounted for as alcohol (on the basis of 61.1 litres of absolute alcohol per 100 kg. of hexoses) or as residual reducers. That these undetermined losses are mainly due to destruction of fermentable sugar, possibly in a labile form, is indicated by experimental fermentations of mashies of the fresh pulp, which gave yields of alcohol representing up to 96% of the reducers in the pulp. This method of working has not yet been adapted to large-scale operations. Rapid fermentation with specially-selected yeasts, as soon as possible after the pulping of the fruit, offers the best means of reducing the losses of sugar.

J. H. LANE.

**Distillation.**—See I. Water in foods. Pineapple [wine].—See XIX.

## PATENTS.

**Nutrient gelatin for production of a stable preparation of lees of wine cultures.** A. KUBICEK (B.P. 343,122, 7.11.29).—The nutrient gelatin is produced by mixing disintegrated edible mushrooms and fruit juices with gelatin so that the mixture, which is solid at normal temperature, becomes liquid at approx. 35°.

C. RANKEN.

**Digestible milk.**—See XIX.

## XIX.—FOODS.

**The  $p_H$  values of New Zealand wheats and flours.** J. K. DIXON (New Zealand J. Sci. Tech., 1930, 12, 146—153).—The quality of flour cannot be predicted from the  $p_H$  and buffer action of laboratory-milled samples. Values obtained are largely determined by the treatment received by the wheat in the mill, and although they may be utilised to indicate the most suitable treatment in milling, they offer no criterion of any inherent properties of the wheat.

A. G. POLLARD.

**March of acidity in stored flour.** II. M. C. MARKLEY and C. H. BAILEY (Cereal Chem., 1931, 8, 29—38; cf. Fifield and Bailey, B., 1930, 213).—The initial acidity of flour determined by the A.O.A.C. method is greater than that by the Greek official method, but the latter shows that a greater rate of increase in acidity is produced on storage.

E. B. HUGHES.

**Importance of degree of acidity in bread-making.** E. and L. ELION (Chem. Weekblad, 1931, 28, 103—105).—The work of earlier authors is discussed, and it is shown experimentally that the amount of diastase present in solution when flour is extracted is increased

when acid is added. It does not appear that formation of sugar is otherwise influenced by the degree of acidity.

S. I. LEVY.

**Methods of examining flour, with special reference to the effects of heat. I. Effects of heat on flour proteins.** C. W. HERD (Cereal Chem., 1931, 8, 1—23).—Heat-treatment was found to increase the viscosity of flour suspensions, interference by bacterial action being prevented by addition of ammonium fluoride or lactic acid. Changes in acidity of dough during normal fermentation do not exceed  $p_H$  0.2. The effects of heat treatment are greatly retarded on reducing the moisture content of the flour. The work of previous authors bearing on this subject is reviewed and an extensive bibliography presented.

E. B. HUGHES.

**Relation of peptisation of wheat-flour protein to loaf volume.** R. H. HARRIS (Cereal Chem., 1931, 8, 47—63).—A comparison is made of three methods of determining peptisation of flour protein. Using 44 samples of Marquis wheat from the 1929 crop and 12 samples from the 1928 crop, a 75% patent flour from each was submitted to baking test with and without bromate. The correlation between protein content and loaf volume, also between peptisable protein and loaf volume, was worked out and it was concluded that total protein was of greater value than peptisable protein in predicting loaf volume. E. B. HUGHES.

**Wheat and flour studies. XVII. Factors influencing the viscosity of flour-water suspensions. IV. Effect of extraction with solutions of the potassium and sodium halides.** A. H. JOHNSON (Cereal Chem., 1931, 8, 39—44; cf. B., 1930, 683).—Flours were extracted with solutions of potassium halides followed by extraction with distilled water to remove the halide. The viscosity of the residue, suspended in lactic acid, was greater where the halides had been used for extraction than in the case of water alone. It is concluded that the protein removed by extraction with halide solution consists mainly of the gliadin fraction. E. B. HUGHES.

**Stability of leavening in self-rising flour.** P. LOGUE and E. McKIM (Cereal Chem., 1931, 8, 24—29).—Keeping tests on self-raising flours containing varying percentages of water show that with a flour of moisture content less than 12.5% deterioration is slight over a period of 1 year. The experiments were carried out on phosphate-type baking-powder. E. B. HUGHES.

**Effect of overgrinding of flour on the keeping quality of bread.** L. P. KARACSONYI and C. H. BAILEY (Cereal Chem., 1931, 8, 44—46).—Tests by the viscosimetric method of Karacsonyi (B., 1929, 760) showed no difference in keeping quality between bread made with normal and with overground flour.

E. B. HUGHES.

**Preliminary report on experimental baking test.** P. P. MERRITT (Cereal Chem., 1931, 8, 64—68).—Preliminary experiments giving the results of comparison of hand and machine mixing, also of hand and machine moulding, are recorded. E. B. HUGHES.

**Biometric analysis of cereal-chemical data. I. Variation.** A. E. TRELOAR (Cereal Chem., 1931, 8, 69—88).—A discussion of the meaning and use of

statistical constants and of their application to the results of analysis of cereal products.

E. B. HUGHES.

**Determination of the ash of bread.** F. BERNARDINI and E. A. GAUTHIER (Giorn. Chim. Ind. Appl., 1931, 13, 7—9).—To determine the ash of bread free from any sodium chloride added during the manufacture, 5 g. of the finely-powdered, dry bread is swollen with 3.2 c.c. of water in a beaker and then treated for 1 hr., with occasional stirring, with 50 c.c. of 99.5% methyl alcohol. The liquid is decanted off on to a filter, and the residue twice treated in the same way with one half of the above amounts of water and methyl alcohol. The final residue is dried, incinerated with the filter, and weighed.

T. H. POPE.

**Hardening of moti, a Japanese rice foodstuff.** T. MAEDA (Nature, 1931, 127, 442—443).—After pounding, steamed glutinous rice hardens in air to a stone-like mass. Hardening is due to loss of water and to internal changes. The material which has been cooled once in a sealed vessel shows a transition temperature at 63.5°.

L. S. THEOBALD.

**Solubility of milk powder.** J. C. MARQUARDT (Creamery and Milk Plant Mo., 1930, 19, No. 12, 32—33).—The quality of ice cream and cream cheese is not affected by the solubility of skim milk powder, which is preferably stored in sealed containers. Slight acidity is developed during storage. The insoluble portion of highly soluble powders consists of protein, whilst that of highly insoluble powders may consist of sugar and protein in various proportions. CHEMICAL ABSTRACTS.

**Application to Italian butters of Hoton's method for detecting coconut oil.** C. MANICARDI (Giorn. Chim. Ind. Appl., 1931, 13, 9—11).—Hoton's method (B., 1929, 109) is satisfactory for the purpose of the test.

T. H. POPE.

**Coloration of frozen meat.** R. HEISS (Z. ges. Kälte-Ind., 1930, 37, 189—194; Chem. Zentr., 1930, ii, 3660).—Coloration is due to the formation of methemoglobin, which is favoured by increase in acidity. Freezing in envelopes or treatment with carbon monoxide preserved the red colour.

A. A. ELDRIDGE.

**Liver extract as a source of vitamins-B and -G [B<sub>2</sub>].** W. D. SALMON and N. B. GUERRANT (Science, 1931, 73, 243—244).—A commercial liver extract was found to be a good source of vitamin-B<sub>2</sub> but a comparatively poor source of vitamin-B. L. S. THEOBALD.

**Effect of drying and sulphuring on the vitamin-C content of prunes and apricots.** A. F. MORGAN, A. FIELD, and P. F. NICHOLS (J. Agric. Res., 1931, 42, 35—45).—Frozen, fresh prunes retained their vitamin-C content. Apricots similarly treated entirely lost this property, but retained it if packed in cases evacuated and subsequently filled with nitrogen prior to freezing. Sulphured dehydrated and sun-dried prunes retained their vitamin-C only when dipped in lye, in the customary manner, prior to treatment with sulphur dioxide. Dehydrated products of both fruits retained more vitamin-C than did corresponding sun-dried samples. Dehydrated or sun-dried apricots retained their full vitamin-C content if treated to contain > 450—500 p.p.m. of sulphur dioxide. A. G. POLLARD.

**The pineapple industry.** J. GUILLAUME (Bull. Assoc. Chim. Sucr., 1931, 48, 25—28).—A short survey is made of the pineapple industry, indicating its uses and giving details for the preparation of a wine from the waste and over-ripe fruit. B. W. TOWN.

**Some properties of honey colloids and the removal of colloids from honey with bentonite.** R. E. LOTHROP and H. S. PAINE (Ind. Eng. Chem., 1931, 23, 328—332).—The colloidal constituents of certain low-grade honeys are largely responsible for their low caramelisation point, and they have in all cases considerable influence on colour, flavour, and crystallisation. The amounts as determined by ultra-filtration through collodion membranes ranged from 0.1 to 1.0%. Cataphoresis tests showed that the colloidal particles of most honeys are positively charged, but the charge can be reversed by increasing the  $p_H$ , and an isoelectric point, which is practically the same for all types of honey, occurs at  $p_H$  4.3. At this value spontaneous flocculation occurs after several hours. The removal of the positively-charged colloids can be effected by adding the negatively-charged colloidal clay, bentonite. Successive portions of an aqueous suspension of bentonite are added to the honey, diluted to 40° Brix, until cataphoresis tests show no migration of the aggregates. After sedimentation and filtration, the filtrate was evaporated under reduced pressure to the original honey density. When applied to three typical honey samples, viz., clover, lucerne, and buckwheat, the treatment gave products almost identical with those obtained by ultra-filtration. In all cases colour, flavour, clarity, and brilliance were improved.

H. J. DOWDEN.

**Some organic acids in honey.** E. K. NELSON and H. H. MOTTERN (Ind. Eng. Chem., 1931, 23, 335—336).—The volatile and non-volatile acids in 15 samples of honey of different origin have been determined. The non-volatile acids were precipitated by adding lead subacetate to 5 kg. of honey diluted with 3 litres of water and 6 litres of alcohol. After settling and removal of the supernatant liquor, the precipitate was washed with 50% alcohol. A check analysis, using an 80% sucrose solution containing known amounts of malic and citric acids, showed that not more than 75% of the acids present were recovered. The malic acid contents varied from a mere trace (in the majority of the samples) to 0.056% for cotton-honey, whilst the citric acid contents varied from 0.001 to 0.008%. Succinic acid was detected only in samples of high acidity (sourwood, cotton). Tests for tartaric acid were negative in all cases. Volatile acids were determined by steam-distillation, the acids in the distillate being liberated by sulphuric acid and the formic acid then destroyed by boiling with mercuric oxide. The total volatile acids ranged from 0.011 to 0.051% and consisted mainly of formic and acetic acids. Sage-honey had the largest amount of acetic acid (0.046%) and tulip-honey the largest amount of formic acid (0.024%).

H. J. DOWDEN.

**Coffee.** K. LENDRICH (Volksernähr., 1930, 5, 310—312; Chem. Zentr., 1930, ii, 3660).—Roasting scarcely affects coffee oil. The high rotation (—19°) is due to the presence of an unsaponifiable substance having  $[\alpha]_D$  —155° as well as phytosterol. The astringent

taste of certain varieties of coffee is due to the presence of an excess of chlorogenic acid. A. A. ELDRIDGE.

**Determination of water in foods and food adjuncts etc.** B. BLEYER and W. BRAUN [with P. FUSCH] (Z. anal. Chem., 1931, 83, 241—268).—The theory, apparatus, technique, reproducibility, sensitivity, range, and rapidity of the following methods are discussed: (1) Loss of weight on heating. (2) Distillation with a solvent of high b.p. and immiscible with water (e.g., benzene, toluene, xylene, or, preferably, tetrachloroethane), the volume of which is measured. This is rapid, but requires 20—50 g. of sample, whilst the solvent must be recovered. (3) Volumetric determination of the acetylene evolved by the action of powdered calcium carbide for 15 min. at 130°. (4) Determination of  $\epsilon$ , water having a much higher value than most of the other constituents of foods. The weighed sample is inserted between the plates of a condenser, the change in capacity of which is measured by means of a valve (induction) circuit, and a calibration curve for the particular type of substance used then obtained by plotting the ratios, scale readings/weight, against the corresponding (known) water contents. Method (1) is recommended for barley (4 hrs. at 105°), malt, and hops; (2) gave high results; (3) gave results which were low by a small constant amount; (4) gave curves characteristic of each type of sample used (caramel malts differing from pale malts), and results having a maximum error compared with (1) of about 0.3%. Lactose, after 3 hrs. at 130°, gave reliable results by method (1), methods (2) and (3) being unsatisfactory, whilst with (4) the curves showed a change in  $\epsilon$  with increase in water content only in the presence of more than 5% of water. For dried milk and marmalade (1) was unsatisfactory owing to retention of small traces of water, (2) and (4) gave excellent results, and (3) may be used for the former if the results are multiplied by 1.05, but is useless for the latter owing to difficulty in producing an intimate mixture. J. GRANT.

**Cacao butter.**—See XII. **Varnished tins.**—See XIII. **Alcohol from bananas.**—See XVIII.

#### PATENTS.

**Increasing the digestibility and pharmacological action of animal milk.** A. HINDELANG, J. VON DALL'ARMY, J. FORSTER, F. RUHLAND, A. FORSTER, R. WEHSARG, and L. WILL ("SAYA" GES. ZUR HERSTELLUNG VON HEIMMILCH) (B.P. 343,127, 8.11.29. Ger., 10.11.28).—Filtered milk, or skim milk freed from cream by centrifuging, is cooled and inoculated with a culture of lactic acid bacilli, and, if desired, with a small amount of *Micrococcus acidilavolactici* and yeast. After the removal of air from the containers by means of an inert gas, the latter are hermetically sealed and the milk is allowed to ferment for about 6 weeks at 8—10°.

C. RANKEN.

**Preparation of dough.** BRIT. ARKADY CO., LTD., and H. HEWITT (B.P. 343,193, 23.11.29).—In the sponge system of breadmaking, the amount of water incorporated may be increased by beating the sponge, either before or after fermentation, by means of a high-speed mixer. By adding a proportion of leguminous flour before beating, a bleaching effect is obtained at the same time.

E. B. HUGHES.

**Apparatus for kneading of margarine and other edible fatty substances.** A. GERSTENBERG and H. J. J. BIGUM (B.P. 345,095, 19.3.30. Ger., 2.4.29).

[Cereal] crushers. Canned goods.—See I. Vegetable waste.—See II.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Determination of morphine in opium and its preparations.** L. DÁVID and S. NOVÁK (Pharm. Ztg., 1931, 76, 289).—Extractum opii is treated in aqueous suspension with calcium hydroxide and lead acetate and the filtrate evaporated to dryness with ammonium carbonate. Morphine is extracted from the residue (charcoal) and titrated with 0.1*N*-hydrochloric acid (lactoid). Anhydrous methyl alcohol is a better solvent than ethyl alcohol for this purpose. H. E. F. NOTTON.

**Chemical investigations of tobacco.** H. B. VICKERY and G. W. PUCHER (Conn. Agric. Exp. Sta., Bull. 311, 1930, 234—246).—Methods of determining the amounts of nitrogenous constituents in tobacco are discussed and a permutit method of determining free ammonia in the presence of nicotine and trimethylamine is indicated. A base similar to the nicotimine (dipicrate, m.p. 163°) of Pictet and Rotschy (B, 1901, 501) was isolated (dipicrate, decomp. 179.5—180.5°). Several varieties of mature tobacco seed contained no nicotine, and it is suggested that conflicting statements in the literature are due to the use of seed at various stages of maturity. Nicotine was found to be present in tobacco seed germinated for 9—11 days on blotting paper in the dark and was presumably derived from the nitrogenous reserves in the seed. Data are also given for the nitrogen distribution in germinated and ungerminated seed. The nitrate nitrogen of tobacco leaf may vary between 0 and 23% of the total nitrogen, or from 0 to 50% of the total soluble nitrogen, these figures being considerably influenced by the fertiliser application. E. HOLMES.

**Formation of carbon dioxide during the fermentation of tobacco.** I. A. I. SMIRNOV and S. M. PETRIK (U.S.S.R. State Inst. Tobacco Invest., 1930, Bull. 72, 18 pp.).—The effects of adding various compounds to different oriental tobaccos undergoing autolysis in moist air have been studied. Some tobaccos show increased formation of carbon dioxide in presence of organic acids, including hydroxy-acids, but with others decreases occur. Nicotine has a variable effect in this direction and carbohydrates are without influence, but amino-acids constantly increase the amount of the dioxide formed. It is concluded that amino-acids are permanent sources of carbon dioxide in tobacco and that, with some tobaccos, organic acids are still more fertile sources. T. H. POPE.

**Essential oil of Queensland sandalwood (*Santalum lanceolatum*).** T. G. H. JONES and F. B. SMITH (Perf. Ess. Oil Rec., 1931, 22, 47—48).—Queensland-grown sandalwood, on extraction, gave 4% of comparatively inodorous oil and the seven samples examined had constants within the following limits:  $d^{15.5}$  0.9522—0.9756,  $[\alpha]_D$  —47.1° to +12°,  $n_D^{20}$  1.5050—1.5100, acetyl value 180—191.9, alcohols 82—88%. Two alcohols have been isolated: (a)  $C_{15}H_{24}O$ ,  $d^{15.5}$  0.9908,  $[\alpha]_D$  +28°,  $n_D^{20}$  1.5127, b.p. 142°/5 mm.; (b)  $C_{15}H_{24}O$ ,  $d^{15.5}$  0.9510,  $[\alpha]_D$  +63°,  $n_D^{20}$  1.5062, b.p. 148°/5 mm.

A small quantity of another alcohol is present, together with a sesquiterpene having  $d^{15.5}$  0.8954,  $[\alpha]_D$  —30°,  $n_D^{20}$  1.4960. E. H. SHARPLES.

**Tobacco.**—See XVI.

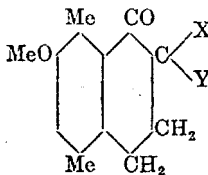
## PATENTS.

**Medicinal product for intravenous use.** A. W. LARSON and A. K. EPSTEIN (U.S.P. 1,775,133, 9.9.30. Appl., 22.12.25).—Hexamethylenetetramine is dissolved in a neutralised solution of phenyleinchoninic acid or its derivatives, with or without the addition of sodium salicylate. E. H. SHARPLES.

**Manufacture of new heterocyclic [azo] compounds [bactericides].** A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 341,598, 6.11.29).—A heterocyclic diazo compound is coupled with a mono- or di-aminopyridine to give azo compounds which are bactericides. Examples are: 2:6-diaminopyridine coupled with diazotised 6-chloro-3-aminopyridine (product, m.p. 242°), 3-amino-6-acetamidopyridine [sulphate, m.p. 253° (decomp.)]; hydrolysis product, m.p. 260°, 5-amino-6-ethoxyquinoline (m.p. 239°), 3-amino-6-hydroxypyridine (decomp. 290°), 3-amino-6-ethoxypyridine (m.p. 181°), 3-amino-2-ethoxypyridine (m.p. 154°), 3-amino-6-butoxypyridine (m.p. 129°); 3-amino-6-acetamidopyridine with diazotised 6-chloro-3-aminopyridine (decomp. 160°); 3-bromo-2:6-diaminopyridine, m.p. 176°, with diazotised 6-chloro-3-aminopyridine [m.p. 255° (decomp.)]; 6-hydroxy-3-aminopyridine with diazotised 6-chloro-3-aminopyridine (decomp. 195°); 3-iodo-2:6-diaminopyridine, m.p. 130°, with diazotised 6-iodo-3-aminopyridine (m.p. 224—225°). The following intermediates are described: 3-nitro-6-ethoxypyridine, m.p. 96°; 3-nitro-2-aminopyridine, m.p. 163—164°; 2-chloro-3-nitropyridine, m.p. 103°; 3-nitro-2-ethoxypyridine, m.p. 20°; 3-amino-2-ethoxypyridine hydrochloride, m.p. 230° (decomp.). C. HOLLINS.

**Manufacture of easily soluble sodium salts of acylaminophenolarsinic acids.** I. G. FARBERIND. A.-G. (B.P. 341,405, 9.10.29. Ger., 9.10.28).—The disodium hydrogen salts of acylaminohydroxybenzene-arsinic acids are sufficiently soluble for injection and are non-irritant. They are prepared by dissolving the arsinic acid (e.g., 3-acetamido-4-hydroxybenzene-arsinic acid or its 5-chloro-compound) in the calculated quantity of sodium hydroxide or carbonate solution, and precipitating the salt with alcohol, acetone, or brine. C. HOLLINS.

**Manufacture of alicyclic lactones of [*ar*-hydroxy-] tetrahydronaphthalenes [anthelmintics, *desmotroposantonin*].** I. G. FARBERIND. A.-G. (B.P. 341,402, 7.10.29. Ger., 5.10.28).—*r*-*desmotropo*Santonin and its analogues are synthesised from 2-alkoxy-1:4-dialkyl-8-keto-5:6:7:8-tetrahydronaphthalenes. 2-Methoxy-1:4-dimethyl-8-tetralone (in formula, X = Y = H), m.p. 63° (from *p*-xyleneol methyl ether and succinic anhydride, the product being reduced to a hydroxyxylylbutyric acid, which is cyclised and re-alkylated), is condensed with ethyl oxalate to give the naphthoylformic ester (X = H, Y = CO·CO<sub>2</sub>Et), m.p. 73—75°, which at



150—190°/15 mm. loses carbon monoxide and yields ethyl 2-methoxy-1:4-dimethyl-8-tetralone-7-carboxylate ( $X = H$ ,  $Y = CO_2Et$ ), m.p. 62—64°, b.p. 170—186°/1.5 mm. The potassium compound of this is condensed with ethyl  $\alpha$ -bromopropionate to give the di-ester ( $X = CO_2Et$ ,  $Y = CHMe \cdot CO_2Et$ ), b.p. 196—210°/2 mm., which when hydrolysed loses one carboxyl group and yields the acid ( $X = H$ ,  $Y = CHMe \cdot CO_2H$ ), m.p. 144°, and the corresponding enol-lactone, m.p. 160—162°. This last acid is demethylated by heating with aluminium chloride in chlorobenzene, and the resulting hydroxyketo-acid is reduced with sodium amalgam and carbonic acid to give the lactone, *r*-desmotroposantonin (cf. A., 1930, 919), m.p. 197—199°.

C. HOLLINS.

Digestible milk.—See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Depression of density produced by the presence of bromide in the developer of photographic emulsions exposed to light and X-rays. A. P. H. TRIVELLI and E. C. JENSEN (J. Franklin Inst., 1931, 211, 335—347).—The depressions of density caused by the addition of potassium bromide (5 mg./litre) to pyrogallol and quinol developers have been determined for varying exposures of kinema positive and negative films to light and X-rays. For equal original densities the depressions were greater (1) for X-rays than for light, except at low exposures (when they might be the reverse), or with longer development, when they tend to equalise; (2) for positive than for negative film; (3) for quinol than for pyrogallol developer. The results are discussed in relation to the distribution and size of developable specks in the emulsion and to current theories of the restraining action of bromide. Abegg's theory is favoured.

J. LEWKOWITSCH.

Double toning of motion-picture film. J. I. CRABTREE and W. MARSH (Brit. J. Phot., 1931, 78, 150—151).—The film is treated with (1) a toning bath, consisting essentially of a solution of ferric ferricyanide in oxalic acid; (2) thiosulphate solution (and then washed); (3) re-immersion in the toning bath; (4) a solution of a basic dye. Blue half-tones and purple (with safranine A), or green (with auramine), or dark green (with chrysoidine 3R) shadows are obtained. The toning has no effect on the quality of sound records.

J. LEWKOWITSCH.

PATENTS.

[Optical system for] colour cinematography. W. W. TRIGGS. From CINEMACOLOR CORP. (B.P. 344,878, 9.11.29).

[Automatic exposure control in] photographic printing. CAMBRIDGE INSTRUMENT CO., LTD., and E. B. MOSS (B.P. 344,900, 10.9.29).

Azo-dye components.—See IV.

## XXII.—EXPLOSIVES; MATCHES.

Sensitiveness to detonation and detonation velocity of gelatine dynamite. S. NAUCKHOFF (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 45—47).—Samples of Extra Dynamite (20% nitroglycerin) were submitted to

vacuum for periods of 10—120 sec., and determinations made of their final air content, sp. gr., loading density, detonation velocity in an aluminium tube, and transmission of detonation. Under high vacuum, the air content is rapidly reduced, with consequent increase in sp. gr., and the detonation velocity falls. No definite relationship between sp. gr. and velocity could be traced, however. After vacuum treatments of 1 min. and 15 min., the sp. gr. increased from 1.376 to 1.56 and to 1.58, respectively, and in both cases the explosive failed to detonate. Brisance, as determined in Hess' apparatus, is also decreased by vacuum treatment. Investigations were made to determine the relationship between the loss of air content, i.e., the ageing, of dynamite and the time of storage. It was established that high temperature has a marked influence, and that, in addition, the gelatinising power of the nitrocellulose is of great importance. To determine the effect of confinement, as it occurs in practice, detonation velocity tests were carried out on 50% ammonium dynamite in a borehole, one sample being untreated and another having been submitted to vacuum and having  $d$  1.56. No appreciable difference in the velocities was indicated, the figures being 6100 and 6200 m./sec., respectively.

W. J. WRIGHT.

Nitrocellulose.—See V.

PATENTS.

### Manufacture of smokeless powder propellants.

A. HOUGH (B.P. 343,107, 11.9.29).—Nitrocellulose (12.4—12.7 N) is dissolved in 20—25% of diethylene glycol dinitrate, with the addition of another solvent, which is subsequently recovered. The powder has a high stability.

W. J. WRIGHT.

Aluminium ophorite. D. B. BRADNER (U.S.P. 1,775,063, 2.9.30. Appl., 21.2.25).—The magnesium in ophorite may be replaced by aluminium foil, ground in a mineral oil; the oil content of the mixture should not exceed 2%. Such treatment increases the efficiency and stability of the explosive.

W. J. WRIGHT.

## XXIII.—SANITATION; WATER PURIFICATION.

Nomograms.—See I. Sulphur-oxidation of activated sludge.—See XVI.

PATENTS.

Manufacture of products for destroying animals. A. HOUSE-MORTON. From F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 342,237, 30.5.30).—The methyl sulphate quaternary salts of dialkylaminophenyl alkyl- or aralkyl-carbamates are stable, non-hygroscopic, easily soluble, tasteless poisons, suitable for mixing with bait for destroying rats etc. Examples are quaternary salts from methyl sulphate and *m*-dimethylaminophenyl methylcarbamate (m.p. 150—153°), ethylcarbamate (m.p. 126—131°), dimethylcarbamate (m.p. 129—132°), benzylcarbamate (m.p. 155—160°), diethylcarbamate (m.p. 136°), or pentamethylenecarbamate (m.p. 120—124°); and from methyl sulphate and *m*-diethylaminophenyl methylcarbamate. The first-mentioned product has the formula  $NHMe \cdot CO \cdot O \cdot C_6H_4 \cdot NMe_2 \cdot SO_4Me$ .

C. HOLLINS.

Greensand.—See VII.



# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAY 22 and 29, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Unfired pressure vessels.** W. SPRARAGEN (Ind. Eng. Chem., 1931, 23, 220—226).—The design and materials for pressure vessels which are welded are dealt with. When properly applied fusion-welding may withstand pressures that would cause rupture of the material itself. Steel plate of not more than 0.2% C is recommended on account of the injury to higher-carbon steels by any method of heating or by hot- and cold-working. Longitudinal seams are welded best from both sides in a double V-groove, although a single V is satisfactory if improperly fused material is chipped out from the back and the place filled in. C. A. KING.

**Kämpf viscosimeter.** E. LANDT (Z. Ver. deut. Zucker-Ind., 1930, 80, 949—955; cf. Kämpf and Schrenk, A., 1930, 1014; B., 1930, 844).—This instrument, in which the rotation of a solid body, suspended in the experimental liquid from almost frictionless bearings, is actuated by a falling weight and measured by the rate of fall of the weight, was found satisfactory with molasses and sugar syrups too viscous for capillary viscosimeters. It requires about 200 c.c. of liquid. At present no means are provided for maintaining constant temperature. The instrument can be used with liquids containing suspended particles, although these are not without influence on the results. With molasses the relation between actuating weight and rate of rotation was found to be strictly linear. J. H. LANE.

**Surface factor [of powders].** J. W. MELLOR (Trans. Ceram. Soc., 1930, 29, [Wedgwood Bicent. Comm. Vol., Pt. I], 251—257).—Mathematical. R. J. CARTLIDGE.

**Making a three-component liquid-vapour chart.** J. HAPPEL and J. GRISWOLD (Chem. and Met. Eng., 1931, 38, 92—93).—A liquid-vapour diagram for the ternary mixture of benzene, toluene, and xylene, under atmospheric pressure, is given as an example. D. K. MOORE.

**Flow of gases at high pressures through metal pipes.** D. M. NEWITT and S. K. SIKKAR (Inst. Chem. Eng., Mar., 1931, 17—26).—The frictional resistance to the flow of air, carbon dioxide, argon, nitrogen, hydrogen, and a 3:1 mixture of hydrogen and nitrogen through pipes of  $\frac{1}{8}$  in. and  $\frac{1}{4}$  in. diam. has been determined. Reynolds' index law has been verified and there is evidence of three distinct types of flow, viz., stream-line flow, that with eddies due to initial disturbance but not sustained, and turbulent flow in the latter size of pipe. D. K. MOORE.

**Control of gas supply at definite temperatures.** K. F. TROMP (Chem. Weekblad, 1931, 28, 163—164).—An apparatus constituting an automatic valve, which is

closed by the melting of a fusible alloy when a predetermined temperature is exceeded in a chosen locality, is described. S. I. LEVY.

See A., April, 449, **Radioactivity method for examination of powders.** Analysis of mixed volatile liquids. 455—6, **Density determinations with solids.** 456, **Colour measurement.** 457, **Analysis of gas mixtures.** **Electrodialysis apparatus.** 538, **Extraction apparatus for liquids.**

### PATENTS.

**Apparatus for drying filter-cake.** G. W. JOHNSON. From PROCTOR & SCHWARTZ, INC. (B.P. 345,247, 18.12.29).—See U.S.P. 1,755,005; B., 1930, 887.

**Preventing the formation of films and scums in water or watery liquids and on surfaces in contact therewith.** A. E. WHITE. From WALLACE & TIERNAN PRODUCTS, INC. (B.P. 345,637, 18.12.29).—See U.S.P. 1,745,141; B., 1930, 968.

**[Combined manually and automatically operated] chemical fire extinguishers.** J. H. COLLIE and G. F. RIMMER (B.P. 345,744, 30.1.30).

**Apparatus for optically investigating the dust content of air and other gases.** H. WITTEMEIER (B.P. 345,601, 23.7.30).

**Furnaces [internally corrugated].**—See II. **Fire-extinguishing agents.**—See III. **Furnace lining. Refractory brick.**—See VIII. **Passing currents through gases.**—See XI.

### II.—FUEL; GAS; TAR; MINERAL OILS.

**Origin of coal.** W. FUCHS and O. HORN (Z. angew. Chem., 1931, 44, 180—184).—A number of vegetable materials, such as cellulose, wood, cotton linters, grape sugar, cheese, and linoleum, carbonised in aqueous alkali under pressure by the method of Berl, Schmidt, and Koch (B., 1931, 50), yielded dark carbonaceous products which were non-coking; these, after hydraulic pressing, all furnished briquettes from which firm, caking cokes were obtainable. Briquettes so prepared from cellulose, lignin, and wood differed from coal in that they gave a brown streak, dark filtrates on treatment with alkali, and a strong reaction and orange colour with dilute nitric acid. A firm, though swollen, coke could thus be obtained from lignin, and no difference was observed between the coke from ordinary and deresinified pine woods. Coking is thus a property common to the carbonisation products of a diversity of materials and bears no special relation to natural coal-formation. Attention is drawn to the rôle of bacterial action, with reference to the work of Taylor

\* The remainder of this set of Abstracts will appear in next week's issue.

(B., 1927, 691; 1928, 509). Caking coles could not be prepared from brown coal (cf. Berl, *loc. cit.*).

E. LEWKOWITSCH.

**Composition of coal: its rational analysis.** W. FRANCIS and R. V. WHEELER (J.C.S., 1931, 586—593. Cf. B., 1929, 5).—Rational analysis of a coal involves the determination of the proportions of (a) the free hydrocarbons and resins, by extraction with pyridine; (b) the ulmins, *i.e.*, the compounds which are converted into alkali-soluble products by oxidation; and (c) the resistant residue, previously termed the "organised plant entities." The "reactivity index" of the ulmins, *i.e.*, the relative ease with which they are oxidised, is also determined. Oxidation is carried out by heating the coal with a solution of nitric acid, to which potassium chlorate may be added if necessary; the correct concentration to use varies with the carbon content of the coal. Durains contain material, absent from clarains and vitrains, which is more resistant towards oxidation than the bulk of the ulmins and less resistant than are the plant entities, and causes some difficulty in the accurate separation of these two constituents. For such coals the analysis is modified by carrying out a series of oxidations with increasing concentrations of nitric acid, plotting the weight of residue against the concentration of acid, and extrapolating the straight branch of the curve back to the concentration appropriate to the corresponding clarain. Examples of rational analyses of British coals are given. A. B. MANNING.

**Aluminium apparatus for determining the tendency of coal and other materials to self-ignition.** D. J. W. KREULEN (Brennstoff-Chem., 1931, 12, 107—111. Cf. B., 1930, 847).—As a result of some preliminary experiments the following conditions have been chosen as standard: the coal sample (5 c.c.) graded to 10—20-mesh (per cm.) as previously described, is introduced into the apparatus, which is heated to and maintained at 120° until the temperature of the coal, up through which a current of carbon dioxide is being passed, reaches 116°. Oxygen (10 litres/hr.) is then substituted for the carbon dioxide and the heating of the apparatus is resumed at a uniform rate of 1—3°/min.; if the temperature curve of the coal is abnormal at a low rate of heating, *i.e.*, if it passes through a maximum and again falls, a higher rate is used and the heating continued until ignition takes place. The temperature at which the heating curve of the coal cuts that of the aluminium block (the "Schnittpunkt") is recorded. Observations are also made of the temperature changes in the coal when the temperature of the aluminium block is maintained constant at successively higher values; the temperature curves so obtained pass through maxima and subsequently fall until a critical initial temperature is reached at which the temperature of the coal rises rapidly until ignition occurs. The "Schnittpunkt" is lower when a more finely-divided sample is used: with brown or bituminous coal it rises with an increase in the rate of heating, but with wood charcoal it is independent thereof. A. B. MANNING.

**Mechanism of combustion of individual particles of solid fuels.** D. F. SMITH and A. GUDMUNDSEN (Ind. Eng. Chem., 1931, 23, 277—285).—Small carbon

spheres were heated in a vertical tubular electric furnace through which nitrogen was passed and the temperature of the spheres was measured optically. At a specified temperature the nitrogen was replaced by a uniform current of air, the sphere burned for a definite time, allowed to cool in nitrogen, and the loss in weight determined. The results show that the "specific surface reaction rate" (*i.e.*, rate of loss of carbon per unit area of surface) increases with rise in temperature of the surface and with increase of air velocity. The rate of reaction and its temperature coefficient are much greater for small particles of carbon than for large. Carbon spheres with a surface area of 10—70 sq. mm. give a higher surface temperature, and burn faster, in moist air than in dry. The explanation of the phenomena is discussed. H. E. BLAYDEN.

**Adsorptive properties of commercial lamp-black.** M. M. DUBININ and S. A. TOROPOV (J. Appl. Chem., Russia, 1930, 3, 843—855).—Ignition or activation with steam or nitric acid partly, or with potash and hydrochloric acid thoroughly, cleans the surface. Methylene-blue and iodine are readily adsorbed from aqueous solution; the adsorption of acids is slight and complex.

CHEMICAL ABSTRACTS.

**Apparatus for sampling powdered fuel.** D. J. W. KREULEN (Chem. Weekblad, 1931, 28, 66).—A small scoop, by the aid of which rapid and uniform sampling may be effected, even when the moisture content varies from place to place in the material, is described. S. I. LEVY.

**Effect of sodium carbonate on gasification of carbon and production of producer gas.** D. A. FOX and A. H. WHITE (Ind. Eng. Chem., 1931, 23, 259—266).—The reaction products of sodium carbonate and carbon at 1000° are shown, by experiment, to be carbon monoxide and sodium. In the gasification of carbon by carbon dioxide, the sodium vaporises and reacts with carbon dioxide to form sodium oxide and carbon monoxide, the sodium oxide further reacts with carbon monoxide to form sodium carbonate, and the sodium carbonate is drawn back to the surface of the carbon and again enters the reaction; the net effect is similar to that which would obtain if the vapour pressure of the carbon were increased to 10<sup>15</sup> times its actual value. The gasification of carbon by water vapour in the presence of sodium carbonate can be similarly explained. The use of coke impregnated with sodium carbonate in the gas producer will cause a more rapid approach to equilibrium and also a change in the gas composition, due to the presence of sodium vapour, the gas having higher concentrations of carbon monoxide and hydrogen than those usually found; the use of basic refractories in the lining is suggested. The use, however, of sodium carbonate for water-gas manufacture is not recommended, as although high steam decompositions and small amounts of carbon monoxide would be obtained in the "run" period, large amounts of carbon monoxide would also be produced during the "blow" period. C. B. MARSON.

**Naphthalene removal [from gas] by means of tetralin.** P. DEUTSCH (Gas- u. Wasserfach, 1931, 74, 245—247).—A consideration of the vapour pressure

of tetralin and its solvent power for naphthalene shows it to be highly suitable for spraying into town's gas to prevent deposition of naphthalene, being as effective as 14 times the amount of xylene. Experiences with the use of tetralin are described, the average amount used being 0.03 lb., costing 0.1d. per 1000 cub. ft. of gas. The adoption of the process should effect considerable economy. A. KEY.

**Testing of gas mains with ethyl mercaptan.** A. THAU (Gas-u. Wasserfach, 1931, 74, 247—250).—It is concluded that ethyl mercaptan, because of its penetrating odour, volatility, harmlessness, and insolubility in water, is a very suitable substance to add to coal gas or natural gas for the detection of leaks in the mains. An account is given of experiences at Franklin and Middletown, U.S.A. For the testing of consumers' installations, about 4 g. per 1000 cub. ft. are introduced into the gas, using an apparatus which is described, followed by a larger amount, 16—20 g. per 1000 cub. ft., for the testing of underground mains. A. KEY.

**Materials for purifying acetylene.** I, II. K. SUMIYA and S. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 527—529 B, 530 B).—I. The efficiencies of various commercial preparations for the purification of acetylene (containing hydrogen phosphide, but not sulphide) were measured by a comparative method, using a mixture of an acid clay with 10% of hydrochloric acid containing 5% of mercuric chloride as standard. Of the non-recoverable purifiers, "Heratol" (cf. G.P. 116,058) is recommended; purifiers of this class, based on bleaching powder, absorb impurities well, but contaminate the gas with chlorine. Of the recoverable materials, Booser's preparation (B.P. 181,571; B., 1922, 579 A) is the most efficient, and its regeneration by air appears to be complete.

II. The addition of a minute amount of mercuric chloride to recoverable purifying materials (consisting chiefly of infusorial earth and ferric chloride) enhances the purifying action; a small amount of cupric chloride, sulphate, or acetate, or copper powder accelerated regeneration, which was also assisted by the addition of ferric acetate. The following proportions are recommended: ferric chloride 9 pts., copper acetate 0.6 pt., mercuric chloride 0.15 pt., ferric acetate 1.68 pts., infusorial earth as required. E. LEWKOWITSCH.

**Hydrogenation of bitumen from the bituminous sands of Alberta.** E. H. BOOMER and A. W. SADDINGTON (Canad. J. Res., 1930, 2, 376—383).—The sands were intimately mixed with sodium silicate, heated, and added to a large volume of hot salt water. The crude bitumen separated thereby had  $d_{25}^{20}$  0.9984 and contained sulphur 4.16%, water 1.0%, and carbon residue 14.3%. Hydrogenation of the bitumen at high temperatures and pressures, with and without catalysts, resulted in the formation under the most favourable conditions of 80% of a light crude oil. The optimum temperature was about 380°. The best catalysts used were ammonium molybdate and aluminium chloride, the latter producing little gas and coke, but having the disadvantage of being corrosive and yielding an oil containing hydrogen chloride. The other catalysts had little effect on the time of reaction, but caused a marked

decrease in the formation of coke and gas. An absorption of hydrogen equal to about 3 wt.-% of the bitumen was obtained. The resultant oil had a sulphur content about one half that of the bitumen and was easily refined to produce a stable white gasoline.

H. S. GARLICK.

**Soluble tars from maritime pine wood.** G. DU PONT and J. L. LUSSAUD (Bull. Inst. Pin, 1929, 301—311. Cf. Hawley and Calderwood, B., 1925, 198).—The dissolved tarry matter in crude pyroligneous liquors from maritime pine gives on evaporation in a vacuum at 100°/20 mm. a viscous, brown residue almost completely soluble in water. This complex mixture yields no useful results by extraction with organic solvents or by neutralisation and extraction. Distillation up to 100° at 1—3 mm. resinifies about 50%. In the distillate there are present furfuraldehyde, methylfurfuraldehyde, acetic and propionic acids, butyrolactone, 3:4-dihydro-pyrocatechol, m.p. 104°, maltol [3-hydroxy-2-methyl-1:4-pyrone], m.p. 156—157°, and at least two phenolic substances not obtained sufficiently pure for identification. C. HOLLINS.

**Desulphurisation and catalytic hydrogenation of a fraction of primary tar.** J. M. PERTIERRA (Anal. Fis. Quim., 1930, 28, 1435—1450).—The sulphur content (0.70%) of a 180—300° fraction of primary tar was present chiefly as hydrogen sulphide 0.17%, mercaptans 0.09%, thioethers 0.04%, and thiophens 0.14%. On passage of the vapour of the fraction admixed with hydrogen over certain metallic oxides previously reduced by hydrogen and heated at 300—350°, a maximum of 60% of the sulphur was removed; after a second treatment the content of basic substances fell by 64.2% and of phenols by 13.3%. The product of berginisation of the primary tar, after removal of the lighter fractions, and using ferric or molybdenum oxide as catalyst, contained phenols 7.2%, bases 13%, neutral oil 86.6%, and was completely soluble in ether, showing complete removal of the ulmin content (2.6%) of the tar. The initial b.p. was 50°, and 13.9% distilled below 185°; the distillate (50—180°) contained aromatic hydrocarbons (29.1%) and unsaturated hydrocarbons (42.3%). H. F. GILLBE.

**Thermal decomposition of coal-tar constituents.** VII. Reaction products of the thermal decomposition of *m*-cresol. VIII. Reaction products of the thermal decomposition of *o*- and *p*-cresols. IX. Reaction mechanism of the thermal decomposition of cresols. Y. KOSAKA (J. Soc. Chem. Ind., Japan, 1931, 34, 10—12 B, 12—13 B, 13—14 B; cf. A., 1930, 332).—VII. Pyrolysis of *m*-cresol at 700° and 800° in contact with silica and coke, and at 900° without contact material, gave (approx.) 95, 69.5, and 38% of condensate, respectively. The corresponding volumes of gases evolved, based on 1 g.-mol. of *m*-cresol, are: 3.5, 30, and 51 litres. Deposits of carbon were found at the three temperatures, viz., 0.5%, 3.25%, and 15%. A table shows the amounts of compounds identified among the condensates. The amount of cresol undecomposed at 900° is about 0.7%, at 800° 21%, and at 700° 86%. The contact material exerts little influence on the products. As the temperature of decomposition

risers, the yields of benzene, toluene, and phenol appear to reach a maximum, those of naphthalene and anthracene vary little, whilst the production of diphenyl etc. increases.

VIII. *o*- and *p*-Cresols gave decomposition products (800°, with silica) similar to those obtained with the *m*-compound. Tables give a comparison of the amounts of products condensed from the three isomerides (decomp.) at 800° as well as the composition of the gases produced. The chief differences are in the quantities of undecomposed cresols (*o*- 12%, *m*- 8%, *p*- 24%) and the yields of phenol (*o*- and *p*- each 19%, *m*- 4.6%).

IX. A scheme of 8 reactions is given to account for the formation of the substances identified among the products of decomposition. The differences observed in the behaviour of the three isomerides are accounted for by the fact that the methyl group in *m*-cresol is more stable than the hydroxyl group. In the *o*- and *p*-isomerides these relations are reversed.

H. INGLESON.

**Fluorescence analysis of coal-tar and petroleum pitch.** W. TEUSCHER (Chem.-Ztg., 1930, 54, 987).—Coal-tar pitch is detectable in petroleum pitch (asphalt), but not *vice versa*, by its more characteristic fluorescence in 1/50,000 dilution under quartz-lamp illumination (cf. Haitinger and Reich, A., 1929, 1127).

L. J. HOOLEY.

**Mechanism of formation of higher hydrocarbons from water-gas.** D. F. SMITH, C. O. HAWK, and P. L. GOLDEN (J. Amer. Chem. Soc., 1930, 52, 3221—3232).—When ethylene and mixtures of ethylene and carbon monoxide or hydrogen are passed over a cobalt-copper-manganese dioxide catalyst (B., 1929, 82) at 206°, no reaction occurs. Passage of heavy hydrocarbons does not result in cracking; this process or polymerisation of ethylene does not occur in the reaction previously studied (*loc. cit.*). Acetone is not an intermediate product, since only 4—7% is converted into a water-insoluble oil by passage over the catalyst. When mixtures of ethylene, carbon monoxide, and hydrogen (containing more than 10% of the hydrocarbon) are used, higher hydrocarbons and oxygen-containing compounds, b.p. below 100°, are produced. The oxygenated compounds are dehydrated and partly polymerised under the conditions used, and their formation depends on the concentration of the water-gas and not the ethylene (above 10%). When water-gas is passed over an iron-copper catalyst at 256—266°, only small amounts of methane and higher hydrocarbons are produced; carbon dioxide is the predominating reaction product. With a mixture of ethylene and water-gas no oxygen-containing compounds are produced and the ethylene does not react at all. The action of an iron-copper-manganese dioxide catalyst is similar to that of the iron-copper catalyst.

H. BURTON.

**Hydrogenation of petroleum oils.** R. H. MCKEE and A. SZAYNA (J. Inst. Petroleum Tech., 1931, 17, 121—132).—A review of certain published information dealing with this subject on the lines originally adopted by Bergius.

H. S. GARLICK.

**Action of inorganic refining reagents on alkyl sulphides in naphtha.** P. BORGSTROM and J. C.

MCINTYRE (Ind. Eng. Chem., 1931, 23, 321—323).—The effect of the various reagents on naphthas containing the following alkyl sulphides, viz., ethyl, *n*-propyl, isopropyl, allyl, *n*-butyl, isobutyl, *sec*-butyl, *n*-amyl, isoamyl, *sec*-amyl, *sec*-hexyl, *n*-heptyl, *sec*-octyl, and benzyl, has been studied. Alkyl sulphides having two or three carbon atoms in the chain are almost completely removed, whilst those with five or more are but slightly removed, when using mercuric acetate or chloride solution. When a solid such as silica gel, that has adsorptive powers, is used, the extent of the removal is dependent on the length of the carbon chain. Solid mercurous nitrate removes the lower sulphides, but is not so effective with the higher secondary sulphides.

C. B. MARSON.

**What determines the value of absorption oil?** F. L. KALLAM (Chem. and Met. Eng., 1931, 38, 78—81).—A series of curves showing the physical characteristics of new and used absorption oils employed in the petroleum industry are given.

D. K. MOORE.

**Regenerating litharge for sodium plumbite "doctor" [solution for petroleum refining].** (Chem. and Met. Eng., 1931, 38, 76—77).—The spent liquor containing 0.6% PbO as sodium plumbite, 4% PbS as sludge, and 11% caustic soda solution separates on keeping into a top layer of oil, a middle layer of emulsion of caustic soda and naphtha containing the solid lead sulphide, and a layer of alkaline plumbite at the bottom. The emulsion is cut by heating to 66° with the addition of a little sulphur, and the lead sulphide which settles out is mixed with the previously separated alkaline plumbite, heated to 80°, and oxidised by blowing air through the suspension to plumbite with the formation of sodium thiosulphate. The colour is removed from the regenerated liquor by washing with the previously separated oil. The concentration of the sodium thiosulphate is kept below 25% by withdrawing from circulation some of the spent liquor.

D. K. MOORE.

**Luminous stationary flames: quantitative relationship between flame dimensions at the sooting point and chemical composition, with special reference to petroleum hydrocarbons.** S. T. MINCHIN (J. Inst. Petroleum Tech., 1931, 17, 102—120).—The method of Kewley and Jackson (B., 1927, 642) for evaluating the burning of kerosenes has been extended in order to ascertain quantitatively the dependence of the tendency to soot on the composition of the oil. Standard distillation cuts, essentially completely paraffinic, naphthenic, and aromatic, respectively, were prepared by methods described, and the maximum flame height, tendency to smoke, and aniline points were determined on various blends of these and on fractions cut from the various treated and untreated crudes taken every 10° from 150° to 280° through a Hempel column. The results show that the tendency to smoke of a kerosene is directly proportional to its aromatic and naphthene content and, in general, to the boiling range. For the boiling ranges considered, the relative smoking tendencies of paraffins, naphthenes, and aromatics, respectively, are 4.4, 13.1, and 42.4. Various substances were burned in the Weber photometer and their

smoking points and the heights and radii of the flames produced were measured. From the results an equation is derived whereby the tendency to smoke can be calculated for any homologous series. The paraffins show an increased smoking tendency with increase in mol. wt., but in all other homologous series the tendency is for a decrease. For a petroleum mixture the sooting tendency increases with increase in b.p. H. S. GARLICK.

**Ultra-violet spectroscopy of flames of motor fuels. V. Analysis of gasoline for iron carbonyl by means of a small quartz-prism spectrograph.** G. L. CLARK, V. R. HARDY, and H. B. WILLMAN (J. Physical Chem., 1930, **34**, 1924—1929; cf. B., 1929, 504).—A method using the ultra-violet spectra of gasoline flames as an accurate and sensitive means of determining iron carbonyl in gasoline is described. A small quartz-prism spectrograph will detect the carbonyl down to a concentration of  $6.2 \times 10^{-7}$  g. of iron per c.c., whilst a larger spectrograph will detect a concentration of the order of  $5.4 \times 10^{-7}$  g./c.c. The line 2450.4 Å. now recorded has not been identified previously in either the flame or the electric-furnace spectrum, whilst the lines 3760.0, 3651.0, 3634.4, 3611.0, 3005.6, 2776.0, 2731.0, 2710.0, 2509.9, 2502.2, 2479.8, 2462.8, 2453.50 Å. have been identified in a flame for the first time.

L. S. THEOBALD.

**Modified absorption-distillation method for analysis of cracked gasolines.** C. C. TOWNE (J. Inst. Petroleum Tech., 1931, **17**, 134—141).—Using synthetic mixtures of hydrocarbons, three methods for the analysis of cracked gasoline have been developed: (a) a gravimetric (barium salt) method in which absorption in 98% sulphuric acid is followed by decomposition of the unsaturated derivatives with barium hydroxide and determination of the barium in solution corresponding to the aromatics. Means for arriving at an average mol. wt. by similar means are described. (b) Using the above method as foundation, a rapid approximate absorption procedure was developed. Unsaturated compounds are absorbed by treatment with cold 93% sulphuric acid in a 1-min. application. The aromatics are determined by absorption of the residual hydrocarbons in 98% sulphuric acid in 30 min. (c) For quick practical work a final procedure is to determine part of the unsaturated constituents by absorption in 3 vols. of cold 93% sulphuric acid for 1 min., and the remainder by distillation to a predetermined temperature after treatment with 98% acid. The effect of the solvent action of sulphuric acid of different concentrations on a number of pure hydrocarbons is tabulated.

H. S. GARLICK.

**Lubricating properties of mineral, vegetable, and fatty oils.** A. S. T. THOMSON and P. S. CALDWELL (J. Roy. Tech. Coll., Glasgow, 1931, **2**, 490—502).—Comparisons have been made by means of the Deeley and Boulton standard testing machines, and a variable-gear-driven friction machine having a cylindrical, double-ring, oiler-type journal bearing, with an oil-bath. Static and fluid friction and viscosity ( $\eta$ ) tests were made on turbine, double-Shell, sperm, rape, castor, and pure Bayonne mineral oils, and on blends of the above oils with one another and with oleic acid. The static friction

( $\mu$ ) is low for fatty oils (i.e., the "oiliness" factor, 100—100  $\mu$ , is high), whilst blended oils have values intermediate between those of mineral and fatty oils. Since solid-friction conditions almost certainly exist when a machine is started up, it is advisable to add a fatty oil to increase the oiliness of a mineral oil and so reduce the friction. With animal and vegetable oils the decrease of  $\eta$  with rise in temperature (up to 200° F.) is less rapid than with mineral oils, so that at high temperatures the lubricating film can withstand pressure tending to break it and so produce solid-friction conditions. Under viscous-friction conditions the lubricating film forms more readily when  $\eta$  is high, but has a lesser tendency to form and a greater tendency to break down at low rubbing speeds (and *vice versa*),  $\mu$  being directly proportional to this speed. Under fluid conditions  $\mu$  decreases with the load up to a certain value of the latter. Small bearings, which involve relatively high loads, produce less friction than large bearings (low loads), but their films are more liable to breakage by overloading. J. GRANT.

**Amount of lubricating oil burned in the gasoline engine.** C. C. MINTER and W. J. FINN (Ind. Eng. Chem., 1931, **23**, 285).—Commercial electrolytic hydrogen was led into the intake pipe of a single-cylinder engine which had previously been freed from carbon deposits. The engine was lightly loaded and run at 600 r.p.m. The cooling water was kept at 100°. The exhaust gases were sampled and analysed, the amount of carbon dioxide present giving an indication of the amount of oil burned. A negligible quantity of oil was burned when the hydrogen was in excess. An oil of low viscosity showed less burning than one of high viscosity.

C. B. MARSON.

**Effect of carbon black on insulating oils.** W. B. WIEGAND, C. R. BOGGS, and D. W. KITCHIN (Ind. Eng. Chem., 1931, **23**, 273—276).—Carbon black has been used successfully to improve the electrical properties of insulating oils. New transformer oils, when treated either by filtration or by sedimentation with dried carbon black, gave an average improvement in dielectric strength of about 40%. Active carbon black tends to remove moisture, electrolytes, and suspended particles.

C. B. MARSON.

**Pyrogenic decomposition of paraffin oil in the presence of various catalysts and under high hydrogen pressure.** R. ODA (J. Soc. Chem. Ind., Japan, 1931, **34**, 58—62 B).—A paraffin oil (no distillate below 300°/760 mm.; 60% between 83° and 215°/4 mm.) from a North Japanese crude was heated under pressure up to 450° (within 1 hr.) in the presence of metallic oxides, silica, iron, or bleaching earths, the rate of change of pressure being observed for comparison. In the presence of catalysts, after a short halt at 450°, the temperature rose spontaneously by some 30° as a result of exothermic polymerisation of the unsaturated products: in general, catalysts decreased the amount of cracked oil formed and increased the yield of gas and coke; further, cracked oils obtained with a catalyst gave more of the lightest fractions and less pitch on distillation. Similar experiments conducted in a hydrogen atmosphere are detailed. E. LEWKOWITZCH.

**Critical solution temperatures of systems of sulphur dioxide and normal paraffins.** W. F. SEYER and E. TODD (Ind. Eng. Chem., 1931, 23, 325—327).—Investigations into the systems sulphur dioxide with seven hydrocarbons of the  $C_nH_{2n+2}$  series ( $n = 4, 6, 8, 10, 12, 14, 32$ ) showed that the critical solution temperature was a function of the mol. wt. of the hydrocarbon. The amount of hydrocarbon soluble in sulphur dioxide at its b.p.,  $-10^\circ$ , is comparatively small.

C. B. MARSON.

**Liquid-vapour chart.**—See I. **Ammonia synthesis.** **Sulphur in oils.**—See VII. **Baku lubricating oil.**—See XII. **Synthetic resins from lignin.**—See XIII. **Brown coal and growth of crops.**—See XVI.

See also A., April, 419, **Adsorption of ethylene.** 436, **Flame speeds of carbon monoxide-oxygen mixtures.** 441, **Ethylene and acetylene from methane.** 450, **Analysis of mixtures of hydrogen, methane, and ethane.** 460, **Formation of petroleum deposits.** **History of coal.**

## PATENTS.

**Carbonisation process and apparatus therefor.** F. C. GREENE and I. F. LAUCKS, Assrs. to OLD BEN COAL CORP. (U.S.P. 1,771,999, 5.8.30. Appl., 18.9.24).—In a vertical retort fitted with a screw conveyor the coke is prevented from sticking to the conveyor by introducing into the carbonisation chamber, through a channel inside and openings in the conveyor, a suitable lubricant, *e.g.*, oil or powdered graphite, conveyed by a stream of gas or by gas under pressure.

D. K. MOORE.

**Coke oven and other furnace [with corrugated heating surfaces].** C. R. BELLAMY, Assr. to W. H. BLAUVELT (U.S.P. 1,772,413, 5.8.30. Appl., 17.11.26).—The walls of the oven are horizontally corrugated on each side to offer increased surface for heat transmission.

D. K. MOORE.

**Distillation of carbonaceous materials.** W. C. KIRKPATRICK, Assr. to WESTERN GAS CONSTRUCTION CO. (U.S.P. 1,770,984, 22.7.30. Appl., 4.8.21. Renewed 15.12.23).—A continuous vertical retort consists of a central cylindrical portion forming the combustion chambers, surrounded by an annular space forming the retort, and this also is surrounded by an annular space forming distillate chambers. The retort is divided into a number of compartments separated by winged conveyors which, on rotation, feed the material being carbonised from one compartment to the next below and at the same time form a seal. Each compartment communicates with its own separate distillate chamber and condenser. The distillate from each compartment is thus separately removed and cracking is eliminated.

D. K. MOORE.

**Manufacture of fuel briquettes.** C. V. McINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS CO. (U.S.P. 1,772,053, 5.8.30. Appl., 29.5.26).—Satisfactory briquettes containing 8—13% of volatile matter (excluding water) are made by mixing semi-coke with 10—12% of pitch, so that the raw briquette contains 18—20% of volatile matter, and then carbonising by radiant heat at 650—750° for 30 min.

D. K. MOORE.

**Production of carbonised briquettes.** C. V. McINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS CO. (U.S.P. 1,772,189, 5.8.30. Appl., 15.7.24).—Briquettes made from low-temperature coke and pitch or asphalt are heated in a continuous vertical retort to about 700° by admitting near the bottom a mixture of air and combustible gas and higher up more air, but in no case is the supply of air sufficient for complete combustion of the combustible gases. The exit gas preheats the air, and then part of it, enriched by other combustible gas, is used for heating the retort.

D. K. MOORE.

**Manufacture of absorbent materials [from peat].** W. W. ODELL (U.S.P. 1,768,963, 1.7.30. Appl., 24.9.24).—The peat is macerated, dried, and carbonised, all operations being carried out in an atmosphere free from oxygen. The addition of certain substances, *e.g.*, ammonium salts or alkali resins, reduces the tendency of the peat to crack during drying. By varying the conditions under which the operations are carried out the porosity and density of the resulting product may be varied. The absorbent material may be rendered catalytic by mixing in catalysts, *e.g.*, platinum salts, during maceration.

D. K. MOORE.

**Apparatus for producing carbon black.** C. F. CROMMETT, Assr. to L. N. WHEELOCK and W. J. HAWKINS (U.S.P. 1,772,984, 12.8.30. Appl., 15.11.23).—Hydrocarbon gases are decomposed in the absence of air in a tube of nickel or other catalytic material. The tube is mounted in a furnace setting and carries a centrally disposed, rotatable tube which delivers the gas by means of jets on to the inner surface of the tube and also carries scrapers which remove the carbon as soon as it is deposited. The carbon then falls to a cooling chamber.

T. A. SMITH.

**Apparatus for producing carbon black.** W. HUNT (U.S.P. 1,773,002, 12.8.30. Appl., 19.1.28).—Gas for the production of carbon black is passed through a central tube of a burner, through the outer annulus of which a combustible gaseous mixture is passing. A number of such burners deliver into a firebrick furnace and the products of combustion are passed downwards through a cooling chamber where they are cooled with a spray of water. Carbon black is collected on a water-seal at the bottom of the cooling chamber.

T. A. SMITH.

**Gas producers.** H. F. SMITH, Assr. to GAS RESEARCH CO. (U.S.P. 1,772,642—3, 12.8.30. Appl., [A] 17.11.21, [B] 27.4.22).—(A) A gas producer for supplying small gas engines consists of a shell with a centrally disposed offtake for the gas. Combustion is arranged to take place between the grate and offtake, the fuel in the shell acting as insulating material. The absence of masses of insulating brickwork which requires to be heated up makes it possible to bring the producer into action more quickly. A saturator is arranged so that the hot gases vaporise the water by heat-exchange. (B) A large gas producer is fitted with a rotating top carrying two steam-operated pokers and fuel hopper. A master-valve controls the supply of steam to the pokers and turning engine. The exhaust steam from the pokers, engine, etc. is used in the saturator. Cooling is provided for parts of the rotating-head, poking, and fuel-feed mechanism.

T. A. SMITH.

**Producer-gas apparatus.** E. SCHUMACHER, Assr. to FRANKFURTER GASGES. (U.S.P. 1,772,819, 12.8.30. Appl., 24.1.25. Ger., 1.2.24).—Apparatus for the low-temperature carbonisation and drying of fuel is fitted to the top of a producer-gas generator. Two concentric tubes are mounted above a stationary plate, the outer tube being rotatable and fitted with a scraper which delivers fuel to the generator. The fuel is fed into the space between the tubes and is dried and distilled by the hot gases which pass from an annular outer space, through the fuel, to the inner tube, which carries off the gas from the generator. T. A. SMITH.

**Apparatus for manufacturing water-gas.** W. E. STEINWEDDELL, Assr. to GAS MACHINERY Co. (U.S.P. 1,772,789, 12.8.30. Appl., 15.2.28).—Gas from a generator is delivered downwardly to a carburettor, the duct having a centrally disposed oil spray. An air duct also leads to the top of the carburettor. During the "blow" period air is admitted to the top of the carburettor and prevents the end of the spray from being burned off. During the carburetting period the spray is kept cool by means of the oil. Better carburetting with lower maintenance costs is obtained. T. A. SMITH.

**Gas-treating apparatus [for internal-combustion engines].** G. E. COOK (U.S.P. 1,772,746, 12.8.30. Appl., 1.2.28).—Exhaust gases are passed first through a layer of activated charcoal or other drying material and then through a bed of "hopcalite" to absorb carbon monoxide. T. A. SMITH.

**Synthesis of methane from gases.** A. H. WHITE (U.S.P. 1,772,652, 12.8.30. Appl., 5.10.25).—Mixtures of carbon monoxide and hydrogen are passed over a nickel catalyst supported on magnesium oxide or calcium oxide. The reaction  $2\text{CO} + 2\text{H}_2 = \text{CH}_4 + \text{CO}_2$  takes place over the range 230–760°, the carbon dioxide produced combining with the oxide. The temperature is then raised so that the carbonate produced is decomposed, and afterwards lowered to the temperature at which it is capable of producing the synthesis of methane. T. A. SMITH.

**Manufacture of asphalt by oxidising heavy petroleum hydrocarbons.** A. F. MACLACHLAN (U.S.P. 1,774,756, 2.9.30. Appl., 12.5.28).—Petroleum or petroleum residuums (above  $d_{40} 0.909$ ) or such hydrocarbons mixed with natural or prepared asphalt are oxidised by continuously circulating the oxidising gas and resulting fumes through the original charge, adding approx. 5% of the volume of air used per 100 gals. of original charge, and maintaining the volume of gases and fumes circulating through the material at approx. 50 cub. ft. per 100 gals. H. S. GARLICK.

**Manufacture of dispersions of bituminous substances.** N.V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of J. M. FAÏN (B.P. 342,031, 15.11.29. U.S., 21.11.28).—Bituminous emulsions containing up to 75% of bitumen are prepared by adding to the water 0.25–0.75% of mineral paste such as bentonite or clay and 0.6–0.25% of soap or other material to lower the surface tension. T. A. SMITH.

**Treatment of aqueous dispersions of bituminous substances.** N.V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of L. KIRSCHBRAUN (B.P. 341,914,

21.10.29. U.S., 20.10.28).—The tendency of bituminous emulsions containing soap to break down prematurely in use when mixed with such substances as lampblack, asbestos, or cement may be overcome by treating these substances with trialkali phosphate or alkali hydroxide before admixture with the emulsions. T. A. SMITH.

**Catalysts for and methods of destructive hydrogenation [of carbonaceous materials].** H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,215–8, 19.6.29).—(A) Heavy mineral oil and hydrogen under 250 atm. pressure are passed upwards through a steel vessel coated internally with nickel or tin and containing a number of similarly treated, horizontal, perforated trays between which are packed tinplate or nickel-plated iron discs or cylinders. The plated iron is activated by treatment with 5–10% nitric acid containing 5% of carbamide, by immersion in borax at 900–1000°, or by heating at 350° in contact with paraffin wax. (B) The reaction is carried out in the vapour phase at 400–600° under at least 10 atm. pressure. (C) Alloys of tin with iron, copper, magnesium, or phosphorus having m.p. above 500° are used as catalysts in either of the above processes. Phosphor-bronze, gunmetal, and iron-tin alloys with less than 80% Sn are specifically claimed. (D) A porous catalyst such as bog iron ore or a dehydrated mixture of ferric oxide with 1–5% of alumina is used in addition to the metal catalysts mentioned above. A. R. POWELL.

**Oil distillation.** C. F. TEARS, Assr. to TEXAS Co. (U.S.P. 1,769,972, 8.7.30. Appl., 23.4.23).—The apparatus consists of a still and a series of bubble-cap towers. Oil is passed through a heat exchanger to the first bubble-cap tower and returned with the condensate to the still. Condensate is withdrawn from each of the successive bubble-cap towers and passed through a heat exchanger, where it is reheated by the vapours from the previous tower. Vapours from the last tower are passed to a condenser. Sharp cuts are obtained. T. A. SMITH.

**Petroleum distillation.** A. McD. McAFEE and B. H. BARNES, Assrs. to GULF REFINING Co. (U.S.P. 1,774,559, 2.9.30. Appl., 17.9.26).—Acid vapours formed by the distillation of petroleum oils containing acidic substances, e.g., aluminium chloride, are removed from the still at the conclusion of the distillation by blowing a mixture of steam and caustic soda solution into the vapour space and allowing the vapours displaced to escape from the still. H. S. GARLICK.

**Apparatus for converting hydrocarbons.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,767,838, 24.6.30. Appl., 6.6.21. Renewed 21.2.29).—Oil is passed through heating tubes mounted in a furnace where it is heated to cracking temperature, to an expansion chamber to which are connected a vapour line and residue draw-off. Means are provided for separately collecting the residual oil from the vaporising chamber and reflux condensate from the dephlegmator; alternate draw-off lines direct the residue and reflux, together with the raw oil, back to the heating coil in predetermined proportions or direct such products to storage. H. S. GARLICK.

**Cracking of petroleum oils.** N.V. BATAAFSCHE PETROLEUM MAATS., Assees. of W. J. PERELIS (B.P.



343,286, 23.1.30. U.S., 14.2.29).—Increased yield in cracking is obtained by carrying out the operation in two stages. In the first the oil is heated to a temperature at which coking would take place but for the fact that the oil is highly turbulent and the heating carried on only for a short period. The oil is then passed to an evaporator, the portion containing asphalt being removed. The heavy fractions from a dephlegmator are returned for cracking in the second stage, being passed through a series of three heating coils.

T. A. SMITH.

**Cracking of petroleum oil.** J. C. MORRELL and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,767,695, 24.6.30. Appl., 1.2.23. Renewed 4.6.29).—Oil is heated to conversion temperature by passage through a heating coil under superatmospheric pressure and discharged into an enlarged chamber, where the products are kept in turbulent agitation by means of paddles driven by a turbine utilising the expansive force of the vapours to supply the mechanical power.

H. S. GARLICK.

**Cracking of [petroleum] oil.** J. S. HARRISON, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,767,360, 24.6.30. Appl., 26.3.27).—Petroleum oil is heated to cracking temperature by passage through a coil set in a furnace and retained under pressure in a digestion zone. From this zone it is continuously withdrawn in a single stream and discharged under reduced pressure and principally in the vapour phase into a vaporising zone in which tar is separated from hydrocarbon vapours which are bubbled through oil in a stripping zone and partly condensed thereby. A fraction of the vapours containing essentially all the gasoline material passes to a rectifying zone and is separated into a condensate and an overhead distillate, which latter, together with feed oil, is continuously supplied to the stripping zone and continuously withdrawn and passed through the heating zone.

H. S. GARLICK.

**Cracking of [petroleum] oil.** J. C. BLACK (U.S.P. 1,770,098, 8.7.30. Appl., 16.11.26).—Oil mixed with aluminium chloride (2–10%) is heated in a heat exchanger to 315°, whereby all the unsaturated compounds are converted into saturated hydrocarbons. The mixture is then heated to cracking temperature in a cracking coil under pressure and passed to a dephlegmator in which the aluminium chloride is recovered.

T. A. SMITH.

**Cracking of hydrocarbon oils.** E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,772,865, 12.8.30. Appl., 30.3.28).—Desulphurising material is added to crude oil before cracking. The mixture is preheated in the refluxing column and then fed into the vaporising chamber, where it is mixed with the material from the cracking coil. Vapours from the vaporising chamber pass to the refluxing column, the lightest being conducted to a condenser and the reflux circulated through the cracking coil. In this way the carrying over of the sulphur-removing material into the cracking coil is avoided and corrosion is minimised.

T. A. SMITH.

**Cracking of hydrocarbon oils.** H. L. PELZER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,774,291,

26.8.30. Appl., 7.6.27).—Oil is forcibly circulated from a bulk-supply drum through a battery of heating tubes and back to the supply drum. The heating tubes comprise a series of banks of vertically arranged tubes situated in a flue with connexions between the upper ends, except the last in such series, and the lower ends of successive banks, the connexions being arranged externally of the flue. The first bank of heating tubes is arranged to make contact first with the heating gases in the flue, which communicates with a firebox at one end and a stack at the other. Means are provided for recirculating through the flue, together with fresh hot products of combustion, a portion of the heating gases escaping. Situated in the bulk-supply tank below the normal liquid level and between the connexions to and from the heating tubes is a bed of finely-divided solid material (*e.g.*, fuller's earth or coke). H. S. GARLICK.

**Cracking of hydrocarbons.** E. W. ISOM and E. C. HERTHEL, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,774,600—2, 2.9.30. Appl., [A-C] 11.6.29).—(A) Oil is passed in series through a vertical bank of tubes arranged in a flue with transverse baffles so that the heating gases which enter at the top of the flue traverse the tubes several times. The oil is maintained under pressure in the heating coil and passed into a vaporising vessel which is also maintained under pressure. In (B) two or more series of heating coils, through which the oil passes in series, are mounted in the same flue, whilst the vaporising chamber is maintained under reduced pressure. In (C) the flue is divided by vertical baffles, so that the path of the gases is along the tubes, and is so arranged that the flow of oil is countercurrent to the heating gases.

T. A. SMITH.

**Treatment of petroleum hydrocarbons.** R. CROSS, Assr. to GASOLINE PRODUCTS Co., Inc. (U.S.P. 1,775,067, 2.9.30. Appl., 2.8.24).—Cracking stock is passed continuously through heating tubes, where it is raised to cracking temperature without any substantial vaporisation or decomposition taking place, and the heated oil is passed to a digestion chamber in which the cracking reaction is completed. The oil is maintained substantially in the liquid stage, and is passed to an evaporator, where the pressure is reduced and the lighter fractions are distilled off and passed to a series of dephlegmators and a final condenser. The reflux condensate from the final dephlegmator may optionally be recycled to the condensate in the preceding dephlegmating stage in order to distil off the remaining lighter fractions, or may be recycled from the final dephlegmating stage to the unvaporised oil in the bottom of the evaporating stage in order to assist in preventing coking of the unvaporised oil. The reflux condensate remaining unvaporised in the initial dephlegmating stage is recycled through the system. H. S. GARLICK.

**Treatment of petroleum oils.** F. G. RING and P. G. PARIS, Assrs. to BETHLEHEM STEEL Co. (U.S.P. 1,775,052, 2.9.30. Appl., 3.7.24).—Cracking stock is heated under pressure to decomposing temperature and the evolved vapours are removed and subjected to a series of selective partial condensations without loss of pressure by passing them countercurrently into heat-interchange immiscible relation with oil of high b.p.

before submitting the latter to decomposing temperature. The condensates of higher b.p. are selectively assembled and submitted to further decomposition and condensation under pressure independently of the first operation. The remaining uncondensed vapours of both decompositions are expanded into heat-interchange relation with a cooling medium to effect a further series of selective partial condensations, and condensates of lowest b.p. are selected therefrom for the recovery of crude naphtha. H. S. GARLICK.

**Treatment of [hydrocarbon] oils.** G. EGLOFF and H. P. BENNER, ASSTS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,767,839, 24.6.30. Appl., 23.4.21. Renewed 16.7.28).—Oil is passed through heating tubes mounted in a furnace and after being heated to cracking temperature is introduced into a small separate inlet chamber, extending along the top of an expansion chamber and having means for intermittently introducing the heated oil into different portions of the chamber, whereby cracking is promoted and the deposited carbon uniformly distributed in the vapour zone. The residual oil with colloidal carbon in suspension is drawn off through a residuum line, and the vaporised oil passes to dephlegmating and collecting means. H. S. GARLICK.

**Continuous low-temperature treatment of hydrocarbons.** W. L. GOMORY, ASST. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,767,283, 24.6.30. Appl., 24.11.23. Hung., 1.8.23).—Raw material is heated to a predetermined decomposing temperature, while being forced rapidly through a heating coil at such a rate that no substantial decomposition occurs, and then continuously passed through a series of decomposing chambers without distillation, the temperature and pressure being maintained. Any one of the chambers can be cut out of the system for cleaning and recharging without disturbing the continuity of the process, while simultaneously another clean decomposing chamber is connected to the system as the last member of the series. The decomposed material continuously passing through the system passes through the lower-temperature charge of the newly connected convertor, and is then discharged at reduced pressure into a vaporising chamber. H. S. GARLICK.

**Treatment of benzol obtained by pyrolysis.** D. A. HOWES, W. S. G. P. NORRIS, S. T. HENDERSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 343,359, 26.3.30).—The reactive compounds formed with benzol in the pyrolysis of gases such as methane, ethane, etc. are contained in the fraction boiling at 130–150°. This fraction is refined after separation and hydrogenation, and then remixed with the benzol. T. A. SMITH.

**Lubricating oil.** N. MACCOULL, JUN., ASST. to TEXAS CO. (U.S.P. 1,767,147, 24.6.30. Appl., 15.8.28).—An unused lubricating oil for internal-combustion engines (flashpoint above 104°, Saybolt viscosity at least 140 sec. at 37.7°) that has a relatively low loss of viscosity during use, consists of a lubricating oil having a viscosity (Saybolt) over 1000 sec. at 37.7° blended with less than 25% (preferably 7–15%) by vol. of a hydrocarbon (b.p. 149–237°) and about 2% of an oxidised higher hydrocarbon, e.g., paraffin wax, completely soluble therein. H. S. GARLICK.

**Manufacture of lubricants.** W. B. D. PENNIMAN (B.P. 317,406, 15.8.29. U.S., 15.8.28).—The lubricants consist of mineral oils to which has been added a "polar body," a material to produce "mushiness," and an anti-oxidant. The polar bodies may be saponifiable oils, fatty acids, or higher alcohols and ketones. "Mushiness" may be produced by the addition of mixtures of diphenylamine and tallow oil, soap solutions, or sulphonic acids. Lead tetraethyl, phenol disulphide, or a number of other materials or compounds mentioned may be used as anti-oxidants. With these additions a lubricant for heavy machinery may be made from oil of low viscosity (below 135 Saybolt at 100° F.). T. A. SMITH.

**Treatment of [lubricating] oils.** PETROLEUM CONVERSION CORP. (B.P. 341,925, 23.10.29. U.S., 6.11.28).—An effect equivalent to the use of high vacuum is obtained by distilling oil in the presence of hot inert gas. The hot gases are passed upwards through a series of towers down which the oil is sprayed and in which successive reductions of temperature of the gas occur. The temperature of each tower is controlled by cooling the unvaporised oil that is being circulated in the tower. This cooling is carried out in heat exchangers in which the incoming oil is preheated. T. A. SMITH.

**[Lubricating] oil water-washer.** N. E. FUNK (U.S.P. 1,770,736, 15.7.30. Appl., 28.11.24).—Oil is continuously diverted from its use in a circulation system and mixed with water so intimately and in such proportions that acid decomposition products and other impurities are withdrawn from solution and form an insoluble sludge in mechanical admixture with the oil, such mixture being subsequently separated by centrifugal means. H. S. GARLICK.

**Oil-sludge accelerator [for testing lubricating oils].** N. E. FUNK (U.S.P. 1,770,735, 15.7.30. Appl., 28.6.24).—A testing device for intensively subjecting a sample of lubricating oil to the deteriorating influences existing in turbine service is described. The effects of heat, light, and contact with air and water may be used to determine the extent of the deterioration and in comparing such oils. H. S. GARLICK.

**Resolving of [mineral oil-water] emulsions.** F. H. PENN (U.S.P. 1,771,096, 22.7.30. Appl., 2.9.26).—A reagent not readily soluble in the continuous phase of the emulsion and comprising a salt of a weak base and a strong acid, e.g., ammonium chloride, an alkali hydroxide, e.g., caustic soda, and a solvent of such reagent which is also a solvent for mineral oil, e.g., alcohol, is added. H. S. GARLICK.

**Separation of mineral oil from emulsions.** F. G. EDMED and H. NEWINGTON (B.P. 343,487, 16.11.29).—Emulsions such as the fuel residues from ships' tanks are broken by the addition of tar or creosote oils or phenols. The mixture is agitated in a tank and may be heated up to 65°. After settling, the aqueous layer may be drawn off. T. A. SMITH.

**Treatment of petrol and other organic liquids containing water.** R. LANT and W. KORESKA (B.P. 343,105, 10.9.29).—Water is removed from petrol (etc.) by filtration through cellulose esters of the higher fatty acids (e.g., oleic or stearic acid). T. A. SMITH.

**Increasing the viscosity of hydrocarbons, oils, and the like.** T. M. RIGBY (B.P. 343,548, 16.11.29).—The viscosity of lubricants is increased and permanent emulsions for use as fuel are obtained by adding rubber latex to oils and wholly or partly coagulating the rubber by suitable chemical treatment and/or heat.

T. A. SMITH.

**Sweetening of petroleum hydrocarbons.** T. W. CULMER, Assr. to LINCOLN OIL REFINING Co. (U.S.P. 1,772,985, 12.8.30. Appl., 1.3.28).—Gasoline vapours are passed through a bed of material supplying oxygen. The refining mass is surrounded by a jacket through which flue gases may be passed to keep the material hot. Suitable refining materials are oxides of lead, zinc, manganese, or copper, or bauxite which has been fused with soda. The reaction is favoured by high temperature and pressure. The addition of dry ammonia prolongs the active life of the catalyst. T. A. SMITH.

**Treatment of [petroleum] hydrocarbons.** E. B. PHILLIPS and J. G. STAFFORD, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,774,611, 2.9.30. Appl., 22.7.27).—Crude oil or naphtha is treated with a solution of sodium "cuprate" (a clear solution made by saturating dilute caustic soda with black copper oxide at about 140°) during distillation. A mixture of oil and the solution (1–4% on the wt. of oil) is pumped into the dephlegmating column of a still, or raw kerosene or gasoline after treatment with soda may be agitated with the solution. T. A. SMITH.

**Refining of hydrocarbon oils.** A. S. RAMAGE, Assr. to GYRO PROCESS CORP. (U.S.P. 1,771,350, 22.7.30. Appl., 25.10.23. Renewed 12.10.28).—The bulk of the sulphur is removed from such oils by treatment with an aqueous lead compound, and the last traces by adding an alcoholic solution of a lead compound and separating the resultant precipitate. Acetylenic hydrocarbons are removed by mixing the oil with an alcoholic solution of a copper compound and separating the resultant precipitate. Diolefinic hydrocarbons are eliminated by treating the oil with an alcoholic solution of a mercury compound and separating the precipitate, and resinous material by treatment with an alcoholic solution of caustic soda followed by washing with water.

H. S. GARLICK.

**Refining [mineral] oil.** W. JOHNSON (U.S.P. 1,767,459, 24.6.30. Appl., 27.3.22. Renewed 25.10.29).—Oil is refined in an apparatus comprising a heating unit, a combined reservoir and collecting chamber, and a combined condenser and steam generator. The second unit is filled with oil, which is circulated by heat through the coil in the heating unit; the vapours pass above the level of the oil in the second unit into the third unit, which acts as a water condenser. Steam generated in this tank by such conversion is injected into the heating coil and, together with compressed air, aids the distillation and bleaching of the oil and maintains the convolutions free from deposit. H. S. GARLICK.

**Refining of mineral oils and mineral fats.** COMP. PROVENÇALE DE RAFFINAGE DE CORPS GRAS MINÉRAUX (B.P. 342,834, 12.12.29. Fr., 27.7.29).—The oil or fat to be bleached is first dissolved in decahydronaphthalene.

H. ROYAL-DAWSON.

**Treatment of hydrocarbon oils.** R. K. STRATFORD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,768,342, 24.6.30. Appl., 16.10.25).—Refining agent, e.g., fuller's earth, is suspended in oil and pumped on to the trays of a bubble-cap tower in which cracked distillate vapours are being treated. Condensate and refining material are received in a settling vessel at the base of tower and, after settling, the oil is drawn off and returned to the still. T. A. SMITH.

**Separation of oil from bleaching clay.** W. W. TRIGGS. From DEUTS. GASOLIN A.-G. (B.P. 343,311, 14.2.30).—Bleaching clay used for refining mineral oil is heated under pressure with dilute mineral acid to a temperature above the normal b.p. of the acid. The oil separates in a layer which can be run off. If necessary, the clay may first be extracted with a lighter solvent. T. A. SMITH.

**Treatment of [spent] adsorbents [containing hydrocarbon oils].** F. A. BENT, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,770,166, 8.7.30. Appl., 10.3.28).—Used filtering clays are revived by treatment with a polyhydric alcohol or ether boiling above 100°, and having a solvent action on the oil. The clay is then washed with water or heated to drive off the alcohol, and is ready for re-use. Compounds of the glycol type are suitable solvents. T. A. SMITH.

**Production of sulphonic acids [from oils].** S. PILAT and J. SEREDA (B.P. 343,530, 19.9.29. Poland, 1.7.29).—Acid sludge obtained in the refining of oils is neutralised with lime and soluble calcium sulphonates are separated by evaporating the filtered solution. The insoluble sulphonates in the precipitate are recovered by boiling with sodium carbonate and salting out the solution of sodium salts so obtained in known manner. T. A. SMITH.

**Fuel for internal-combustion engines.** H. MENZ (B.P. 343,238, 14.12.29).—The fuel consists of alcohol 50 pts., benzol 10 pts., tetrahydronaphthalene 5 pts., distilled water 30 pts., pulverised sulphate-cellulose extract 1 pt., and sulphonated whale oil 4 pts. The material gives a stable emulsion and does not cause rust formation. T. A. SMITH.

**Liquid combustible.** C. A. BUERK (U.S.P. 1,766,501, 24.6.30. Appl., 4.5.26).—Engine fuels are improved by the addition ( $\frac{1}{2}$ – $\frac{3}{4}$  oz.) of benzoyl peroxide to 13 gals. of fuel. Alternatively, hydrogen peroxide, naphthalene peroxide, or a per-salt may be used. T. A. SMITH.

**[Stabilising] treatment of hydrocarbons.** J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,770,181, 8.7.30. Appl., 15.10.23. Renewed 28.7.27).—Residual oil containing carbon from a cracking plant is passed through a homogeniser or colloid mill to render it more suitable for use as fuel. T. A. SMITH.

**Distillation of tar.** BARRETT Co., Assess. of A. A. MACCUBBIN and J. ZAVERTNIK (B.P. 318,897, 11.9.29. U.S., 11.9.28).—See U.S.P. 1,759,816; B., 1931, 10.

**Doors for gas-generating, coking, and like retorts and ovens.** H. J. LIMBERG (B.P. 345,913, 24.6.30. Ger., 2.6.30).

**Solid polyformaldehyde.** Vanillin.—See III. Carbon monoxide in gaseous mixtures.—See VII.

Weatherproofing stone etc.—See IX. Tanning materials. Adhesive.—See XV. Nitrogenous compounds.—See XVI. Purifying waste waters.—See XXIII.

### III.—ORGANIC INTERMEDIATES.

Utilisation of the soya bean. IV. Electrolytic oxidation of the hydrolysate of proteins. I. Y. TAKAYAMA (J. Soc. Chem. Ind., Japan, 1931, 34, 31—32 B; cf. B., 1930, 753).—Crude soya-bean protein was hydrolysed with sulphuric acid and the filtered hydrolysate electrolysed with a peroxidised lead anode and a lead cathode in an undivided cell (current density 2 amp./dm.<sup>2</sup>) and then steam-distilled. The volatile acids of the distillate (9.6% of the crude protein) contained formic, acetic, and isovaleric acids, and probably also propionic and butyric acids in small amount. Ammonium chloride was the only base recoverable from the residue of distillation. Small amounts of other volatile materials present were probably benzoic acid and an acetone-like substance. Formic, acetic, and valeric acids were obtained similarly from the gluten hydrolysate. E. LEWKOWITSCH.

Effect of carbohydrates and other impurities on the crystallisation of glutamic acid hydrochloride from decomposition products of soya-bean protein. M. MASHINO and T. SHISHIDO (J. Soc. Chem. Ind., Japan, 1930, 33, 421—422 B).—The addition of the methyl alcohol extract from soya-bean cake (cf. Mashino, A., 1928, 436; 1930, 384, 826) considerably reduces the yield of glutamic acid hydrochloride obtainable on recrystallisation: the addition of carbohydrates does not reduce the yield so much. By purifying soya-bean cake with methyl alcohol before decomposition a high yield (90%) of the salt is obtainable, whereas an unpurified cake yields only small amounts of the crystalline product. E. LEWKOWITSCH.

Liquid-vapour chart.—See I. Purifying acetylene.—See II. Alumina catalyst [for ethyl alcohol].—See VII. Toxicity of amyl nitrite.—See XXIII.

See also A., April, 431, System acetic acid-ethyl acetate. 439, Catalysis of hydrogenation. 441, Ethylene and acetylene from methane. 477, Action of bromine on phenols. 479, Preparation of  $\beta$ -naphthol-3-sulphonic acid. 492, Separation of borneol from camphor. 498, Synthesis of aminotoluthiazoles.

#### PATENTS.

Manufacture of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,411, 18.11.29).—Aliphatic  $\alpha$ -glycols are passed at 270—300°/10 atm. over a dehydrating catalyst, *e.g.*, sodium dihydrogen phosphate, red phosphorus, potash alum, alumina activated with copper sulphate, aluminium phosphate, etc. The increased pressure results in increased throughput without loss of yield. [Stat. ref.] C. HOLLINS.

Manufacture of polymerisation products of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 339,243, 30.8.29).—In the polymerisation of diolefines by alkali or alkaline-earth metals, either as single metals, as alloys, or as non-ionised organic

derivatives, the agent is added gradually or in at least 3 portions. In the examples, sodium, sodium amalgam and sodium, and potassium  $\alpha$ -diphenylethane ( $K \cdot CPh_2Me$ ) are so used. C. HOLLINS.

Polymerisation of hydrocarbons of the butadiene series. I. G. FARBENIND. A.-G. (B.P. 342,107, 9.1.30. Ger., 9.1.29).—The butadiene is treated with sodium previously dispersed in a finely-divided solid diluent, *e.g.*, 1.5 pts. of sodium wire ground in a ball mill with 60 pts. of dry sodium chloride. C. HOLLINS.

Production of chlorinated hydrocarbons. IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, and J. MASON (B.P. 342,329, 24.10.29).—Methane and the required amount of chlorine (3 vols.) are passed through a (preferably) packed tube above 650° (*e.g.*, 800—850°) at high space velocity (*e.g.*, 5500 reciprocal min., calc. without the packing), to give 25% conversion (on the chlorine) into methyl chloride, methylene dichloride (15%), chloroform (1.5%), and carbon tetrachloride (1.5%). Methyl chloride and chlorine (5.4 vols.) at 600° gives methylene dichloride (46.7% on the chlorine). C. HOLLINS.

Preparation of combustible, solid polyformaldehyde. W. SCHILT (B.P. 342,668, 3.10.29).—Non-melting solid fuel is obtained by treating formaldehyde or paraformaldehyde with sulphuric acid (5—25% concentration at 55—60°, 20—30% at 15°) or phosphoric acid (40% at 15°). C. HOLLINS.

Preparation of vinyl esters of organic acids. E. I. DU PONT DE NEMOURS & Co. (B.P. 319,589, 23.9.29. U.S., 22.9.28).—Acetylene in excess is passed into an organic acid (*e.g.*, acetic) at 70—90° containing a catalyst (mercuric sulphate) and diluted with an aliphatic hydrocarbon distillate having a b.p. above that of the acid, and preferably lower than that of the ethylidene ester. With kerosene as diluent 11—15% yield of vinyl acetate is recovered in one distillation, compared with 2% without diluent. Vinyl propionate, b.p. 90—110°, and butyrate, b.p. 115—120°, are similarly prepared. Other suitable diluents are medicinal paraffin, "triethylin,"  $\beta$ -ethoxyethyl acetate, b.p. 154°, and high-boiling petrol, b.p. 150—180°. C. HOLLINS.

Manufacture of nitrophenyl derivatives of thiazole compounds [vulcanisation accelerators]. GOOD-YEAR TIRE & RUBBER Co., Assees. of J. TEPPEMA (B.P. 342,330, 25.10.29. U.S., 6.2.29).—Alkali salts of 2-thiolbenzthiazoles are condensed with nitro- or dinitrobenzenes carrying reactive chlorine. The following compounds are described: 2-*op*-dinitrophenylthiolbenzthiazole, m.p. 162°, and its 5-nitro- (m.p. 185—187°), 6-chloro- (m.p. 165°), and 6-chloro-5-nitro- (m.p. 172°) derivatives; and 2-*o*-nitrophenylthiolbenzthiazole, m.p. 110—112°. [Stat. ref.] C. HOLLINS.

Production of vanillin [from peat, lignins, etc.]. H. PAULY and K. FEUERSTEIN (B.P. 319,747, 27.9.29. Ger., 27.9.28).—Peat powder, lignite, brown coal, etc. is oxidised below 90° with the calculated amount of chromic acid, ozone, or other suitable oxidant. A further quantity of vanillin is obtained by hydrolysis of the mother-liquors with acid after extraction of the vanillin.

Thus 100 kg. of peat powder yield 1.1 kg. of vanillin, pine wood sawdust 2 kg., maple wood meal 2.45 kg., and elm wood meal 3.2 kg. C. HOLLINS.

[Manufacture of non-resinous] esters of dibasic acids, particularly esters of polyhydroxy-compounds containing at least two free hydroxyl groups. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,324, 27.7.29. U.S., 27.7.28).—A dibasic acid or anhydride (phthalic anhydride) is heated below 140° with an ether of a polyhydric alcohol still containing free hydroxyl groups to give the acid ester, which is then heated below 160° with excess of an aliphatic alcohol to complete esterification. *E.g.*, phthalic anhydride and glycerol monoethyl ether at 140° give a product which with butyl alcohol and hydrogen chloride at 160° yields  $\beta$ -hydroxy- $\gamma$ -ethoxy-*n*-propyl butyl phthalate, b.p. 231–232°/7 mm. The products are useful as plasticisers for cellulose esters and ethers.

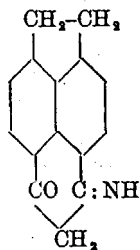
C. HOLLINS.

Manufacture of substitution products [cyanoacetyl derivatives] of aromatic hydrocarbons. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 342,373, 5.11.29).—Cyanoacetyl chloride, prepared in solution from cyanoacetic acid and phosphorus pentachloride in a solvent, is condensed with an aromatic hydrocarbon in presence of aluminium chloride (etc.) and the solvent below 60°. Suitable solvents are carbon disulphide and tetrachloroethane. Acenaphthene gives 5-cyanoacetylacenaphthene, m.p. 163°. Products from toluene (m.p. 104°) and 1-methylnaphthalene (m.p. 127°) are also described.

C. HOLLINS.

Manufacture of cyclic ketones of the acenaphthene series, and of 1:4:5:8-naphthalenetetracarboxylic acid and derivatives thereof. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 342,379, 6. and 7.11.29. Addn. to B.P. 342,373; cf. preceding abstract).—5-Cyanoacetylacenaphthene, obtainable by the process of the parent patent, is treated with aluminium chloride (etc.) at 110–160°, in a diluent (tetrachloroethane) if desired, whereby a cyclic imino-ketone (annexed formula) is obtained. This may be oxidised, *e.g.*, by chromic acid or sodium hypochlorite, to naphthalene-1:4:5:8-tetracarboxylic acid. Similar products are obtained from the 4(or 5)-cyanoacetyl derivative of 3-chloroacenaphthene.

C. HOLLINS.



Preparation of solutions of salts of sulphonic acids [fire-extinguishing agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,399, 11.11.29).—The addition of urea or thiourea increases the solubility of sodium butylnaphthalenesulphonate etc. in water. The fire-extinguishing agents of B.P. 319,083 (B., 1929, 964) may thus be prepared in highly concentrated solutions, readily diluted for use. C. HOLLINS.

Manufacture of 4-halogeno-1-hydroxyanthraquinone-2-sulphonic acids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,551, 19.2.30).—A 4-halogeno-1-aminoanthraquinone-2-sulphonic acid is diazotised and the diazo compound is added gradually

to an aqueous suspension of powdered copper or solution of copper sulphate. Preferably enough copper sulphate is used to precipitate the copper salt of the product.

C. HOLLINS.

Sulphonic acids.—See II.

#### IV.—DYESTUFFS.

Sulphur dyes. I. Preparation of a new series. G. D. PALMER, JUN., and S. J. LLOYD (J. Amer. Chem. Soc., 1930, 52, 3388–3395).—When the vapour of benzene, chlorobenzene, dichlorobenzene, naphthalene, anthracene, furfuraldehyde, or ethylene is passed over or through molten sulphur at about 380°, reddish-brown sulphur dyes (the colours quoted are those on cotton and rayon), soluble in aqueous sodium sulphide and containing 61.34–80.28% S, are produced. Dyes are also obtained from substances such as alcohol (grey-brown), ether, acetylene (grey), phenol, benzaldehyde, acetic acid, toluene, diethylaniline, diphenyl, turpentine (brown), and aniline (green). Dye formation appears to be proportional to the amount of hydrogen sulphide evolved; the compounds used must necessarily contain hydrogen.

H. BURTON.

See A., April, 420, Adsorbents for dyes. 426, Dye sols. 456, Colour measurement. 479, [Azo dyes from]  $\beta$ -naphthol-3-sulphonic acid. 480, Reduction of triphenylmethane dyes. 484, [Dyes from] pyromellitic acid. 494, Closing of heterocyclic rings in indigoid dyes. 495, Dyes derived from thiohydantoin. 497, Zerevitinov determination with pyrrole dyes.

#### PATENTS.

Manufacture of brominated vat dyes of the benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,884, 21.9.29).—The vat dye obtained by alkaline fusion of *N*-(3-benzanthronyl)pyrazolanthrone is brominated below 60° in an inorganic medium, *e.g.*, chlorosulphonic acid, concentrated sulphuric acid, or oleum, in presence of iodine as catalyst. Navy-blue vat dyes result. [Stat. ref.] C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,466, 18.12.29).—A dibenzanthrone or *iso*-dibenzanthrone is treated at 100° with sulphur chloride (4–6 pts.) in presence of aluminium chloride (etc.) to give blue vat dyes. C. HOLLINS.

[Manufacture of] vat dyes [of the dibenzanthrone series]. C. SHAW, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 342,315, 24.7.29).—Grey to black vat dyes are obtained by treating 4:4'-dibenzanthronyl with chromic and sulphuric acids, with or without subsequent alkylation. C. HOLLINS.

Manufacture of yellow wool [azo] dyes. I. G. FARBENIND. A.-G. (B.P. 319,585, 23.9.29. Ger., 21.9.28).—A *m*-aminoarylsulphonarylamide,  $\text{NH}_2\cdot\text{Ar}\cdot\text{SO}_2\cdot\text{NHA}\text{r}'$ , in which Ar does not contain a carboxyl group *ortho* to the amino-group, and in which Ar' does not contain at the same time a sulphonic and a hydroxyl group, or a *m*-aminoarylsulphonanilide,  $\text{NH}_2\cdot\text{Ar}\cdot\text{SO}_2\cdot\text{NHPh}$ , in which Ar may carry only alkyl substituents, is diazotised and coupled with a halogenated arylpyrazolone

(except 1-[2':5'-dichloro-3'-sulphophenyl]-3-methyl-5-pyrazolone), and the product is sulphonated if desired. Examples are: *p*-toluidine-2-sulphonanilide  $\rightarrow$  1-(2'-chloro-4'-sulphophenyl)- or 1-(4'-chloro-2'-sulpho-5'-methylphenyl)-3-methyl-5-pyrazolone; *m*-aminobenzenesulphonanilide  $\rightarrow$  1-(2'-chloro-5'-sulphophenyl)-3-methyl-5-pyrazolone. (Cf. B.P. 12,205 of 1909; B., 1910, 554).

C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Fleece analysis for biological and agricultural purposes. Average fineness of a sample of wool.**

J. A. F. ROBERTS (J. Text. Inst., 1930, 21, T 127—164).—A laboratory sample is selected from the sample the mean fineness of which is to be determined, and the total length of the fibres in it is found. The dry weight of the laboratory sample is determined and the fineness is then expressed as cm. per mg. Since the fineness varies within wide limits over small areas of skin, a system of zoning is adopted so that the sample taken for analysis is made up of a large number of small pieces collected from all over the main sample. The methods of sampling, of computing the total length of the hairs, and of determining the dry weight are fully described, and a statistical survey is made of the standards of accuracy for the whole process.

B. P. RIDGE.

**Reindeer wool.** V. N. PETELINA (Niti [Suppl. Izvest. Tekstil. Prom.], 1930, 1, No. 3, 18—19).—A mechanical analysis of Jamala, Ouralsk, wool is recorded.

CHEMICAL ABSTRACTS.

**Dry weight of cotton.** G. F. DAVIDSON and S. A. SHORTER (J. Text. Inst., 1930, 21, T 165—178).—Determinations have been made, using the desiccator, commercial oven, small oven, box oven, and the bulb methods. True dry weight is defined as the constant weight obtained by prolonged exposure at room temperature to the dry atmosphere produced by phosphorus pentoxide. If any hot-air drying process gives a lower value, subsequent low-temperature drying over phosphorus pentoxide gives less than the true dry weight, indicating the permanent loss of water or other material. Cotton subjected to the action of hot air continues to lose weight for days, or even weeks, but, under the conditions of commercial testing, the rate of loss becomes very slow in a few hours and a stage is soon reached at which apparent constancy of weight is attained. If air which has been artificially dried is used for drying, the dry weight is independent of temperature over a wide range. At temperatures above 100°, however, the weight obtained is less than the true dry weight. If the cotton is heated by undried air, the dry weight given at moderately high temperatures is greater than the true dry weight due to residual moisture in the cotton. The effect of the hygrometric state of the air, the correlation of different apparent dry weights, and the difference between bleached and unbleached cotton are discussed.

B. P. RIDGE.

**Influence of humidity on the elastic properties of cotton. V. Tensile behaviour.** K. C. BROWN, J. C. MANN, and F. T. PEIRCE (J. Text. Inst., 1930, 21, T 186—203).—Breaking load, extensibility at break, stress-strain curves, Young's modulus, etc. have been

determined for single hairs of various cottons (raw, soda-boiled, and mercerised) and for filaments of viscose rayon, under different conditions of atmospheric humidity and temperature. The effects of temperature and humidity, the behaviour of Young's modulus, the effect of length of specimen, etc. are discussed, and the results are examined statistically. Comparisons are made between the elastic properties of the cotton hairs and those of yarns. It is concluded that differences in specific strength, extensibility, etc. between varieties of cotton can be explained largely as the effect of flaws rather than of intrinsic specific differences in the cellulose.

B. P. RIDGE.

**Effect of moisture on the strength and elongation of yarn.** P. D. ERMOLENKO (Izvest. Tekstil. Prom., 1930, 9, No. 3, 39—44).—Yarn tested at R.H. 60—80% was 11—51% stronger and had 8—42% greater elongation than dry yarn tested in a dry atmosphere.

CHEMICAL ABSTRACTS.

**Elimination of *Aspergillus* from cotton.** V. A. KUTIEICHTCHIKOV (Izvest. Tekstil. Prom., 1930, 9, No. 3, 57—60).—The only usual treatment which is efficient is boiling under pressure with sodium hydroxide solution.

CHEMICAL ABSTRACTS.

**Application of neutral salts to the cottonisation of flax.** A. P. ZAKOCHTCHIKOV, I. A. KOHYENIOV, and E. YKIONCHKIN (Izvest. Tekstil. Prom., 1930, 9, No. 3, 91—95).—Sodium or ammonium oxalate, but not oxalic acid, is a good pectin solvent for flax; calcium oxalate is precipitated.

CHEMICAL ABSTRACTS.

**Cottonisation of flax.** A. A. AXIPETROV and G. N. RLOUMN (Izvest. Tekstil. Prom., 1930, 9, No. 3, 89—91).—The moistened flax is steamed for 2 hrs. at 120—150°, washed, and centrifuged. The distillate and drainage are acid; hence the process depends on acid hydrolysis.

CHEMICAL ABSTRACTS.

**Cottonising [of flax].** B. M. LOTAREV (Niti [Suppl. Izvest. Tekstil. Prom.], 1930, 1, No. 1—2, 17—18).—Greatest separation of fibres is effected by mercerising; cooking with sodium hydroxide and oxidation with hypochlorite are next in order. The fibres are not appreciably weakened.

CHEMICAL ABSTRACTS.

**Cooking flax yarn with sodium oxalate.** G. A. KORZHENIOVSKI (Niti [Suppl. Izvest. Tekstil. Prom.], 1930, 1, No. 3, 15).—Sodium oxalate does not clean the fibres as well as does sodium carbonate, but the loss in weight is less and subsequent bleaching more rapid. Oxidisable substances are eliminated with equal ease.

CHEMICAL ABSTRACTS.

**Composition and mechanical properties of "kenaf" fibre as a function of watering of the plants.** V. T. IVANOVA (Izvest. Tekstil. Prom., 1930, 9, No. 1—2, 140—144).—Artificial watering increased the cellulose, lignin, fat, and resin, and decreased the moisture and ash, contents of plants grown in areas subject to drought.

CHEMICAL ABSTRACTS.

**Study of the structure of vegetable fibres by dissolution in sulphuric acid.** A. P. ZAKOCHTCHIKOV (Izvest. Tekstil. Prom., 1930, 9, No. 1—2, 137—140).—The addition of a drop of sulphuric acid (*d* 1.82) to a vegetable fibre is followed by that of a drop of glycerin;

decomposition of the fibre is thereby arrested, and may be studied microscopically. Membranes, forming part of the structure of the fibre but more resistant to sulphuric acid, are observed with flax, hemp, ramie, "kendyr" and "kenaf." The object is preferably treated subsequently with chlorine water or zinc chloride and potassium iodide. Cotton requires lactic acid also.

## CHEMICAL ABSTRACTS.

**Determination of the content of organic fibres in asbestos products.** ANON. (Mitt. Materialprüf., 1931, 141—142).—The usual organic fibres employed in making asbestos string, board, or paper are cotton and silk, the total content of which cannot be determined by loss on ignition as the actual loss on ignition of asbestos itself varies considerably with the variety. Good results are, however, obtained by dissolving out the silk and cotton with cuprammonium solution and weighing the washed residue. For the determination of the silk content the mixture is boiled with dilute sodium hydroxide solution, washed, dried, and weighed; the residue is then treated with cuprammonium solution to extract the cotton and again washed, dried, and weighed. The difference between these two weights plus 3.5% of this difference (allowance for the cotton dissolved in the sodium hydroxide solution) gives the cotton content, and the difference between this and the sum of the cotton and silk previously determined gives the silk content.

A. R. POWELL.

**Cellulose production with the aid of chlorine. I. From bagasse. II. Course of the chlorination process.** J. KAWAMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 392—395 B, 396—398 B).—I. After pretreatment with caustic soda solution of various concentrations, bagasse (from Formosa) mixed with an equal weight of water was subjected to the action of chlorine gas and the lignin chloride was subsequently removed by means of 1% caustic soda solution. The amount of lignin remaining in the bagasse depended on the duration of the chlorination and on the concentration of the alkali used in the pretreatment, more complete removal of lignin resulting from the use of 2% than of 1% caustic soda. The pentosan content was slightly higher after chlorination, but was reduced by subsequent extraction with 10% sodium hydroxide solution.

II. During the chlorination process the ratio of chlorine converted into hydrochloric acid in the reaction to the chlorine combining with the lignin is approximately constant (7 : 3).

F. R. ENNOS.

**Behaviour of cellulose with bisulphite as compared with sulphurous acid solution. II. Experiments at 150°.** T. NAKASHIMA and S. OHORA (J. Soc. Chem. Ind., Japan, 1930, 33, 390—392 B; cf. B., 1930, 857).—Sulphurous acid attacks cellulose at 150° with formation of sulphuric acid and sulphur, the liquid increasing in colour with time of treatment; sugars which are also formed increase in quantity at first and afterwards diminish owing to oxidation by the sulphurous acid. Under similar conditions bisulphite solution has much less action on cellulose. The proportion of  $\beta$ - and  $\gamma$ -cellulose increases with time of treatment in the case of bisulphite, but not with sulphurous acid.

F. R. ENNOS.

**Action of alkali on cellulose. IV. Mechanism of oxidation of alkali-cellulose in ripening.** K. ATSUKI and H. SOBUE (J. Soc. Chem. Ind., Japan, 1930, 33, 511—513 B; cf. B., 1931, 107).—The chemical reactions occurring between cellulose and alkali are considered to be represented by the following equations: (1)  $R \cdot OH + Na^+ + OH^- + H_2O = R \cdot ONa + 2H_2O$ ; (2)  $R \cdot OH + NaH_2O^+ + OH^- = R \cdot OH, NaH_2O^+ + OH^-$ ; (3)  $R \cdot OH, NaH_2O^+ + OH^- = NaH_2O^+ + R(OH)_2^-$ ; (4)  $R \cdot OH, NaOH = R(OH)_2^- + Na^+ = RO^- + NaH_2O^+$ . Reactions (1) and (2) take place with concentrations of alkali at or below that required for mercerising, respectively, and (3) and (4) occur in more concentrated alkali solutions, the cellulose complex taking on a negative charge, whereby oxidation, which is stated to be the most important chemical reaction in ripening, readily occurs.

F. R. ENNOS.

**Solubility of cellulose in caustic soda.** T. NAKASHIMA (J. Soc. Chem. Ind., Japan, 1930, 33, 388—390 B).—When cotton and sulphite-cellulose are extracted with successive portions of 10% caustic soda solution in an atmosphere of hydrogen, to avoid formation of oxycellulose, the solubility gradually diminishes and finally becomes constant. The high initial solubility is probably due to dissolution of impurities such as hemicelluloses.

F. R. ENNOS.

**Detection of oxy- and hydro-cellulose.** ANON. (Mitt. Materialprüf., 1931, 142).—Owing to the similarity in behaviour of oxy- and hydro-cellulose, none of the known qualitative tests is capable of distinguishing between the two compounds in woven goods. The most sensitive tests are the methylene-blue, Prussian-blue, and the ammoniacal silver nitrate methods, particularly the last. Fats and waxes do not interfere with the tests, but the degradation products of starch, lignified constituents, and pectin should be removed before testing; if this is not possible, the defects in the cloth can be detected only by strength tests.

F. R. ENNOS.

**Determination of cellulose.** K. KÜRSCHNER and A. HOFFER (Chem.-Ztg., 1931, 55, 161—163, 182—184).—1 G. of cellulosic material is distilled under reflux on a water-bath with a mixture of 20 c.c. of 96% alcohol and 5 c.c. of concentrated nitric acid for 1 hr., and, after removal of the liquor by decantation, is heated for a further hour with another 25 c.c. of alcoholic acid. The crude cellulose is then filtered, washed with alcohol and water, dried, and weighed; it is tested for lignin and pentosans with phloroglucinol hydrochloride and, if the former be present, the extraction is repeated until the cellulose is freed from lignin. The method was applied to a large number of cellulose-bearing materials with satisfactory results, the reagent having no effect on the cellulose. A numerical relationship was found to hold between the amount of pentosans originally present in the material and that remaining in the cellulose after extraction.

F. R. ENNOS.

**Acetolysis of bamboo cellulose.** S. OGURI and M. NARA (J. Soc. Chem. Ind., Japan, 1930, 33, 516—517 B).—The yield of cellobiose octa-acetate obtained from bamboo cellulose is very similar to that from cotton cellulose, and the m.p. and  $[\alpha]$  of the products are nearly identical.

F. R. ENNOS.



**Velocity of swelling of cellulose acetate film. Effect on the mechanical properties of the film.**

K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 497—499 B).—The swelling of acetone-soluble cellulose acetate in various liquids was determined by steeping the dried film in the liquid at ordinary temperature, shaking with water to replace the swelling agent with water, wiping to remove adhering water, and weighing. The increase in weight, expressed as a percentage of the dry weight, is called the "swelling degree" of the film. Experiments showed that the velocity of swelling of the material in water may be expressed by  $x = 1/z \times \log [w_\infty / (w_\infty - w)]$ , where  $w_\infty$  is the limiting swelling degree,  $w$  the swelling after time  $z$ , and  $x$  is a constant. For alcohol-water mixtures the maximum swelling is obtained with a solution containing 20% of the former and 80% of the latter, and the elongation of the film for a given load is directly proportional to the swelling degree; when the swelling degree exceeds 20 the film becomes plastic. The swelling degree and mechanical properties of the film with other liquids are also given.

F. R. ENNOS.

**Influence of plasticiser on the mechanical properties of cellulose acetate film.**

K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 499—500 B).—Cellulose acetate film containing triacetin shows both elastic and plastic elongation, the load-elongation curves resembling those of cellophane steeped in acetone-water mixtures. The relation between the critical load, viz., that corresponding to the transition point, and the percentage of triacetin is linear. The tensile strength of a film containing the plasticiser was unaltered by immersion in water.

F. R. ENNOS.

**Peptisation and coagulation of cellulose nitrate and acetate.** A. Y. DREIBERG (J. Appl. Chem., Russia, 1930, 3, 915—950).—The phenomena depend on the concentration, dispersing medium, temperature, and the degree of depolymerisation of the cellulose ester.

CHEMICAL ABSTRACTS.

**Viscosity of cellulose esters. I. Relation between concentration and viscosity of cellulose nitrate solution. II. Viscosity of mixed cellulose nitrate solutions.** K. ATSUKI and M. ISHIWARA (J. Soc. Chem. Ind., Japan, 1930, 33, 506—508 B, 508—511 B).—Experimental results for viscosities of cellulose nitrate solutions are in good agreement with those calculated by the formulæ: (I)  $\eta_s/\eta_o = (1 + ac)^k$  for separate nitrate solutions, or (II)  $\eta_s/\eta_o = \{1 + a(nc' + c)\}^k$ , for mixed solutions of two different cellulose nitrates, where  $\eta_s$  and  $\eta_o$  represent the viscosities of solution and solvent, respectively,  $c$  and  $c'$  the concentrations of the esters,  $\eta = c'/c$ , and  $a$  and  $k$  are constants. (Cf. Baker, B., 1913, 991.)

F. R. ENNOS.

**Electrical insulating paper. I. Effect of beating on its electrical insulating and mechanical properties. II. Dielectric strength of cellophane and of papers impregnated with plastics.** K. ATSUKI and K. MATSUOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 385—387 B, 387—388 B).—I. Determinations of tensile, bursting, and folding strengths, oil absorption, porosity, and dielectric breakdown voltage were made on sheets of paper produced from spruce sulphite pulp which had

been beaten for varying times. The results indicate that, whilst oil absorption and porosity diminish with time of beating, there is an optimum beating degree which gives high insulating power and the maximum mechanical strengths to paper. II. Cellophane shows lower porosity and higher dielectric breakdown voltage than paper made from sulphite spruce pulp. Paper impregnated with bakelite or pollopas is brittle and has a higher dielectric breakdown voltage and lower mechanical strengths than the original paper. Casein, owing to its hygroscopic character and mineral content, lowers its value as a dielectric, but does not appreciably affect the mechanical properties.

F. R. ENNOS.

**Analysis and composition of vegetable parchment for packing dairy products.** P. ARUP (Analyst, 1931, 56, 149—161; cf. Weiss, B., 1924, 230).—Details of analysis of 41 samples of vegetable parchment and two samples of greaseproof paper are given, the results being tabulated. Additional standards tentatively suggested are: water 10.0% (max.); ash 0.45%; water-soluble extract 1.30%; bursting strength (by Ashcroft tester) min. 25 lb./in.<sup>2</sup> for parchments of 25 lb. per ream, and 18 lb./in.<sup>2</sup> for those of 18 lb. per ream. The strength immediately after wetting should be at least 33% of that of the unwetted sample. The water-soluble extract of genuine vegetable parchments was found to consist of degradation products of ligno-cellulose, and is the material on which the growth, or otherwise, of moulds depends. The presence of added sugar may be detected by its copper-reducing power.

T. McLACHLAN.

**Lignin-resins.**—See XIII. Flax.—See XVI. Fermented rice straw.—See XVIII.

See also A., April, 469, Purification of cellulose fibres. Preparation of fibrous cellulose nitrate.

## PATENTS.

**Preparation of cellulose from plants of annual growth such as cornstalks.** CORNSTALK PRODUCTS Co., INC., Assecs. of E. R. DARLING (B.P. 343,115, 15.10.29. U.S., 13.12.28).—After comminuting and washing, the plant material is softened by heating with a solution containing 1% of sodium hydroxide and 0.1—1% of sodium sulphite at a temperature corresponding to a steam pressure of 10 lb. above atmospheric pressure; the entire mass, including the plant material and softening solution, is afterwards pumped into a digester where it is digested at 10—30 lb. super-atmospheric pressure.

F. R. ENNOS.

**Production of cellulosic pulp.** CORNSTALK PRODUCTS Co., INC., Assecs. of E. R. DARLING (B.P. 343,429, 15.10.29. U.S., 13.12.28).—Cellulosic agricultural waste, particularly cornstalks, is digested with 1% caustic soda, fresh concentrated soda being added periodically to maintain the digesting solution at its initial concentration throughout the operation.

F. R. ENNOS.

**Manufacture of cellulose esters and cellulose ester products therefrom.** H. DREYFUS (B.P. 342,738—342,740 and 343,986, [A—C] 2.8.29, [D] 16.8.29. Addns. [B] to B.P. 312,098 and [C] to B.P. 309,201; B., 1929, 750, 593).—(A) The process for esterifying cellulose in

the presence of a ferric halide as catalyst (cf. B.P. 312,095; B., 1929, 750) is modified in that the proportion of iron in the catalyst mixture is greater than the chemical equivalent of the halide radical. Thus a basic ferric halide may be used as catalyst, or suitable quantities of an alkali or of ferric hydroxide may be added to the ferric halide before or during the esterification process. (B) The process of the prior patent is extended to include pretreatment of the cellulose with less than 2% of hydrogen halide (calc. on wt. of cellulose). (c) The prior process is extended to include the use of mixed catalysts of the type hydrogen halide-ferric halide when either or each of the components is present in quantities less than 2% (on the wt. of cellulose). (d) The cellulosic material used in the pre-treating and esterifying processes described in [A—C] and also in B.P. 309,201, 312,095—6, and 312,098 (B., 1929, 593, 750) may with advantage contain more than 8% of water, *e.g.*, up to 30%. [Stat. ref.]

D. J. NORMAN.

**Manufacture of cellulose esters.** KODAK, LTD., Assees. of C. J. STAUD and E. E. BEETON (B.P. 344,151, 22.1.30. U.S., 22.1.29).—Cellulosic material is hydrated by treatment with 68% nitric acid for 1 hr., followed by esterification at 100—170° with an aliphatic acid in the absence of a catalyst.

F. R. ENNOS.

**Manufacture of cellulose esters.** W. W. GROVES. From SOC. CHEM. IND. IN BASLE (B.P. 343,889, 19.11.29).—Cellulose is partly acylated by means of an acid (acetic) anhydride in the presence of a basic agent (caustic alkali, pyridine, sodium acetate), and is afterwards treated with an organic or inorganic esterifying agent, in presence or absence of an acid catalyst (zinc chloride, sulphuric or hydrochloric acid).

F. R. ENNOS.

**Manufacture of esters of cellulose or of its transformation products or of other carbohydrates.** I. G. FARBERIND. A.-G. (B.P. 343,655, 20.12.29. Ger., 20.12.28. Addn. to B.P. 301,036; B., 1930, 504).—The esterification in a medium of liquid sulphur dioxide is carried out with addition of a limited quantity of an oxidising agent (chromic oxide, hydrogen peroxide, potassium permanganate), followed by treatment with a hydrolysing agent (water) if desired.

F. R. ENNOS.

**Production of organic acid esters of cellulose.** L. CLEMENT and C. RIVIÈRE (B.P. 343,712, 31.1.30).—Cellulose in the form of cotton linters is hydrolysed by heating at 100—145° under pressure with water, which may contain 1—2% of acetic acid; after washing and drying, the hydrocellulose is converted into fatty or aromatic esters in the usual manner. F. R. ENNOS.

**Preparation of cellulose ester solutions.** J. K. WEIDIG, Assr. to CELLULOSE UTILITIES CORP. (U.S.P. 1,775,179, 9.9.30. Appl., 24.3.23).—The viscosity of cellulose ester solutions is reduced without deleteriously affecting the strength of the resulting film or coating by treating the cellulose ester (before, during, or after treatment with the solvent) with a regulated quantity of a basic reagent, *e.g.*, sodium bicarbonate, chalk, zinc oxide, anhydrous ammonia, or diethylamine. The treatment may continue with agitation for 1—10 days,

depending on the particular reagent used, and the reagent may then be neutralised or removed by filtration. Anhydrous ammonia (<1%, *e.g.*, 0.15—0.33% on the total solvent) dissolved in ethyl acetate is particularly suitable.

D. J. NORMAN.

**Manufacture of cellulose ethers.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. S. LEVESLEY (B.P. 343,873, 14.8.29).—Cellulose glycol ethers which are soluble in water and insoluble in the common organic solvents are obtained by treating alkali-cellulose, prepared in the usual way and matured for 48—96 hrs. at 10—35°, with solid caustic soda and ethylene chlorohydrin and heating the mixture for about 8 hrs. at 100°. Further quantities of caustic soda and ethylene chlorohydrin may then be added and the heating continued. The cellulose ether when isolated from the yellow sticky reaction mass by dialysis and subsequent precipitation with acetone, is obtained as a white fibrous material readily soluble in water, giving a solution of high viscosity.

D. J. NORMAN.

**Manufacture of cellulose ethers.** H. DREYFUS (B.P. 343,521, 20.11.29).—Organic-soluble cellulose ethers of hydroxycarboxylic acids or their derivatives, particularly celluloseglycolic acids, are prepared by treating cellulose in stages with sufficient halogenated organic acids or their derivatives (*e.g.*, chloroacetic acid) to introduce more than one ether group into the  $C_6H_{10}O_5$  molecule; the reaction is carried out in the presence of an amount of caustic alkali exceeding 3 times the wt. of the water present, which should be 1.5—4 times the wt. of the cellulose, and also, if desired, in the presence of a diluent (benzene or alcohol) and lime.

F. R. ENNOS.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. in BASLE (B.P. 343,748, 21.2.30. Switz., 1.6.29).—Alkali-cellulose or cellulose which has been impregnated with aqueous or alcoholic potash solution is treated with carbon disulphide, and simultaneously or subsequently with a solution of cyanogen halide in benzene in the presence of an organic base; the product has a marked affinity for acid dyes.

F. R. ENNOS.

**Purification of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 344,327, 21.7.30. U.S., 30.7.29).—The cellulose derivative, *e.g.*, acetate, is ground to pass 5-mesh and is then either graded through a series of screens (6—200-mesh) or simply separated into two fractions on a screen of about 40-mesh. The coarser the screen the higher is the purity of the cellulose acetate retained thereon, most of the impurities being in the fraction which passes 100—200-mesh. The coarse particles of cellulose acetate give solutions of high clarity and are particularly suitable for photographic films.

D. J. NORMAN.

**Softener for cellulose derivatives.** H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. Co. (U.S.P. 1,761,813, 3.6.30. Appl., 18.1.29).—The cellulose derivative (*e.g.*, nitrocellulose) is plasticised with a condensation product of a polyhydric alcohol (glycerol) with pimelic, suberic, azelaic, or sebacic acid.

F. R. ENNOS.

**Dry-spinning of cellulose derivatives.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 342,712, 3.10.29).—After formation of a skin-like layer

by partial evaporation of the solvent, the extruded filaments are treated with a liquid solvent or softening agent, subjected to stretching which is wholly or partly confined to the softened portions of the filaments, and finally set by further evaporation of the solvent followed by the application of an additional stretching. The initial partial setting, the softening treatment, and the final setting are carried out in separate compartments of a single cell or chamber. F. R. ENNOS.

**Production of viscose for manufacture of artificial silk.** W. HARRISON (B.P. 342,779, 9.11.29).—The raw material, after being prepared according to the process described in B.P. 268,505 (B., 1927, 473), while bringing it to any point almost up to the stage where prior to the oxidation process it becomes soluble in water, is treated with caustic soda solution of over 15% concentration, together with a reducing agent (*e.g.*, sodium sulphide or sulphite, glucose, etc.) or with hydrogen or gases containing hydrogen in the presence of a hydrogenating catalyst (*e.g.*, oxides of nickel, manganese, or iron). The product is then pressed until it retains two or more times its weight of caustic soda, disintegrated if necessary, treated with carbon disulphide, and finally dissolved in water with or without addition of caustic alkali. F. R. ENNOS.

**Production of artificial silk from viscose.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 344,751, 3.7.30. Ger., 15.7.29).—The first washing and drying under tension of freshly-spun viscose silk may be omitted without appreciable shrinkage of the thread if the spinning bath contains at least 5 g. of zinc sulphate per litre and the sodium sulphate to sulphuric acid ratio is in excess of that required for the formation of bisulphate, the absolute sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) content being not less than 200 (preferably not less than 230) g./litre. The zinc sulphate content of the bath should be adjusted according to the sulphuric acid content, and for acid concentrations of 100, 110, and 135 g./litre should be 10, 15, and 25 g./litre, respectively. D. J. NORMAN.

**Preparation of voluminous artificial silk from viscose.** A. C. DE WILDE (B.P. 343,604, 5.10.29).—Hollow viscose filaments are obtained by using un-matured or but slightly matured viscose of normal composition and viscosity and dispersing therein at any time prior to spinning not more than 0.1% of an insoluble substance to provide nuclei for the evolution of gases (hydrogen sulphide, carbon dioxide, etc.) within the filament during the spinning operation. Naphthalene, nitronaphthalene, camphor, anthracene, diphenylamine, or a mixture of these may be used, and is conveniently dispersed before addition to the viscose by dissolving it in, *e.g.*, pyridine and adding the solution to a 1% aqueous saponin solution. D. J. NORMAN.

**Preparation of artificial silk from viscose by the "centrifuge" system.** W. W. TRIGGS. From ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 343,118, 16.10.29).—In order to prevent damage to the thread by vibration and to lower the resistance due to a long spinning trajectory, the guide funnel is kept filled with liquid, which, if desired, may be similar to the coagulating liquid, for the purpose of completing the setting of the thread. F. R. ENNOS.

**Manufacture of artificial filaments, threads, etc. by wet-spinning methods.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 342,651, 3.10.29).—Filaments of viscose, cuprammonium cellulose, or organic derivatives of cellulose are partly coagulated in a suitable liquid to form a skin-like layer and, after washing free from coagulating liquid if necessary, are softened by means of a solvent in liquid or vapour form and subjected to a stretching process which is confined to the softened portion. The setting of the product is afterwards completed by liquid coagulating agents or by evaporation, a further stretching being applied if required. F. R. ENNOS.

**Manufacture of films, sheets, etc. from cellulose nitrate.** CELLULOID CORP. (B.P. 343,962, 25.11.29. U.S., 24.11.28).—A solution of cellulose nitrate (10.7–11.35% N; viscosity 10–75 sec.), which may contain a plasticiser (camphor) and a stabiliser (urea) if desired, is applied to a surface, and, after evaporation of the solvent, the layer is stripped off. F. R. ENNOS.

**Production of artificial filaments, films, and like materials.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 343,062, 6.8.29 and 15.4.30).—Solutions of organic derivatives of cellulose are wet-spun into coagulating media containing relatively concentrated aqueous solutions of solvents for the cellulose derivative (diacetone alcohol, diethyl tartrate, ethyl lactate) in the presence of substances which tend to reduce the solubility of the solvent in the non-solvent coagulating medium (water) and are capable of exerting an osmotic pressure (sugars, salts, etc.); alternatively, the solvents which are less volatile than the non-solvent medium may be wholly or partly incorporated in the spinning solution. F. R. ENNOS.

**Manufacture of [hollow] artificial filaments.** BRIT. CELANESE, LTD. (B.P. 318,629, 5.9.29. U.S., 8.9.28).—Solutions of cellulose derivatives are extruded into an atmosphere having a low evaporative power, *e.g.*, one maintained at a temperature below the b.p. of the solvent employed or one substantially saturated with the solvent vapour, to produce a slow initial evaporation of the filaments etc., which are then passed through an atmosphere having a high evaporative power, *e.g.*, one maintained at a temperature considerably above the b.p. of the solvent, or one having a low concentration of solvent vapour, to produce rapid solidification. F. R. ENNOS.

**Manufacture of artificial filaments, yarns, or threads by the dry or evaporative method.** BRIT. CELANESE, LTD., and R. P. ROBERTS (B.P. 343,973, 18.11.29).—Filaments of cellulose derivatives having cross-sectional characteristics periodically varying along their length are produced by periodically varying the evaporative conditions, *e.g.*, the temperature of the spinning solution or of the evaporative atmosphere, the concentration of the solvent present in the evaporative atmosphere, or the rate at which the evaporative atmosphere is carried away from the spinning chamber. F. R. ENNOS.

**Production of artificial filaments.** C. RIMINGTON, and BRIT. RES. ASSOC. FOR THE WOOLLEN & WORSTED INDUSTRIES (B.P. 343,838, 9.5.30).—Keratinous material

(*e.g.*, wool, wool waste, hair) is dissolved in ammoniacal copper solution, wholly or partly reduced by an agent which will not cause deposition of copper (sodium hyposulphite or sulphite), and the solution, with or without addition of cuprammonium solution of cellulose, is spun into filaments and coagulated in an acid bath.

F. R. ENNOS.

**Obtaining threads from protein colloids in solution.** SOC. D'APPLICATIONS ET DE RECHERCHES SCIENTIF. ET INDUSTR. (S.A.R.S.I.) (B.P. 342,821, 2.5.11.29. Fr., 24.11.28).—The viscous solution is allowed to fall by gravity through a large spinning nozzle and the filament drawn out by the falling drop is detached from the drop and collected on a spool. The filament denier is determined by the speed of winding and the rate of supply of the spinning solution. Suitable apparatus is described.

D. J. NORMAN.

**Manufacture of artificial silk.** BREDA-VISADA, LTD., and R. O. JONES (B.P. 342,812, 21.11.29).—The spinning solution is delivered to the jets by pumps constructed to give periodic variations of pressure at constant speed of operation, whereby filaments are produced in which thin and thick portions of substantial length alternate.

F. R. ENNOS.

**Spinning of artificial filaments and the like.** BRIT. CELANESE, LTD., W. I. TAYLOR, and H. DREYFUS (B.P. 343,936, 25.11.29. Addn. to B.P. 324,061; B., 1930, 318).—The filament, after extrusion into a closed chamber under reduced pressure, is led through an aperture the size of which can be adjusted so that it does not substantially exceed the cross-section of the filament; the aperture is surrounded by a hood or cowl which maintains a supply of heated dry air in the region of the aperture, thereby preventing moist air from entering the chamber.

F. R. ENNOS.

**Manufacture of films.** KODAK, LTD., ASSEES. OF S. E. SHEPPARD and J. G. McNALLY (B.P. 344,170, 4.2.30. U.S., 8.2.29).—In order to prevent shrinkage during drying, cellulose ester films, after coagulation from solution by suitable liquids or steam or after swelling of the dried film, are stretched and dried under tension.

F. R. ENNOS.

**Treatment of artificial silk yarns after spinning.** F. J. GANLERT (B.P. 343,358, 26.3.30. Ger., 26.3.29. Addn. to B.P. 283,752; B., 1928, 228).—A winding-off spool having a regulatable braking effect applied to it is so arranged as to impart a tension to the yarn passing over or around the rollers.

H. ROYAL-DAWSON.

**Treatment of threads, filaments, etc. [in cake form].** COURTAULDS, LTD., W. H. STOKES, and E. A. MORTON (B.P. 343,730, 11.2.30).—A strip of textile material is placed along the top outside surface of the horizontally-supported spun cake and another along its lower inner surface; the cake is then washed by allowing water to drip on to the upper surface of the upper strip of textile material.

F. R. ENNOS.

**Papermaking.** J. NEUMANN (U.S.P. 1,775,405, 9.9.30. Appl., 2.11.27. Renewed 8.2.30).—Cellulosic materials such as flax, wood, straw, etc. are freed from incrustants without attacking the cellulose by digestion for about 2 hrs. at 5—125 lb./in.<sup>2</sup> with a liquor containing 2—20% (on the weight of dry material) of a 4 : 1 mixture

of sodium silicate ( $d$  1.297—1.842) and caustic potash. For materials other than cereal straws 2—20% of sodium sulphide is introduced into the cooking liquor after the first  $\frac{1}{2}$  hr. of the digestion. After washing and bleaching, the resulting pulp is suitable for high-grade papers or artificial silk.

D. J. NORMAN.

**Adhesive papers and the like.** PAPETERIES NAVARRE (B.P. 343,248, 20.12.29. Fr., 17.1.29).—The paper is coated with a layer consisting of a mixture of barium sulphate, gelatin, and dextrin; such a paper is rendered adhesive by being dipped in water.

D. J. NORMAN.

**Imitation leather.** J. HOLMES (BIAS FABRICS), LTD., and J. HOLMES (B.P. 342,673, 26.10.29).—The fabric base is built up of two plys or webs connected together so as to leave a small space between them. When the cellulose-mica coating solution is applied it percolates through the top ply into this space and, on drying, firmly anchors the film to the base.

D. J. NORMAN.

**Laminated [fibrous] materials.** BAKELITE CORP., ASSEES. OF V. H. TURKINGTON (B.P. 343,572, 21.11.29. U.S., 30.11.28).—Sheets etc. of fibrous material are pressed and heated with potentially reactive phenolic resinoid bonding agents in the presence of a plasticiser, *e.g.*, butyl phthalate, which is substantially non-volatile at the temperature of hardening of the resinoid. The resulting product can be "cold-punched" even in thick layers.

S. S. WOOLF.

**Manufacture of vulcanised fibre.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,089, 13.11.29).—A porous and absorbent raw material (paper) is worked at 60° with a solution of zinc chloride ( $d$ <sub>20</sub> 1.98), to which another salt (calcium or ammonium chloride) has been added.

F. R. ENNOS.

**Rubber products.**—See XIV. Nitrogenous substances.—See XVI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Dyeing and cleaning.** Some problems of the industry. F. W. LAKE (J. Soc. Dyers and Col., 1931, 47, 68—73).—Deteriorated materials cannot be restored so as to have their original dyeing properties by any treatment. Stripping usually precedes re-dyeing: stripping treatments additional to those usually employed are the removal of indigoid vat dyes by boiling with caustic soda and potassium ferri-cyanide, and the treatment of mordant dyeings on wool with a boiling solution of oxalic acid for removal of the metallic radical. When dyed under ordinary conditions, faded wool has a much greater affinity than that of the normal fibre for dyes, the difference being least marked with acid dyes. It is not recommended to re-dye faded wool materials at the boil in a sharply acid bath since the cotton stitchings may become tendered, the seams be poorly penetrated, and even the wool itself damaged; it is preferred, as the result of experiments with about 100 dyes, first to strip in a boiling chromic acid bath and then dye with a level-dyeing colour, using the normal amounts of assistants. Of the group of acid dyes which can be dyed from a neutral or faintly alkaline bath, the great majority yield fuller shades on the faded portion of a woollen

garment, and variation in the temperature of dyeing does not appreciably affect this phenomena. Cardinal Red JS gives a solid shade on faded and non-faded wool. Substantive dyes which give almost solid shades on mixtures of normal wool and cotton give nearly solid shades when dyed at the boil on faded and non-faded wool; Chlorazol Black SDS, Chrysophenine GS, and Benzo Fast Bordeaux 6BL show this effect particularly. Dyeing difficulties caused by fading are not met with in "dry dyeing" by means of a liquor prepared by dispersing in "white spirit" (a petroleum fraction,  $d$  0.780—0.810, flash point 30—35°, b.p. 130—210°), and by the aid of an ammonium soap, a solution of a suitable dye in a mixture of methylated spirit and water; "Stoddart solvent" (a petroleum fraction, b.p. 138—210°, flash point 57°) may be used in place of "white spirit." *cyclo*Hexanol is very suitable for use with dry-cleaning soaps.

A. J. HALL.

**Control of the operation of quick-steaming apparatus for aniline-black.** P. P. SAZANOV and N. N. PETROVA (Izvest. Tekstil. Prom., 1930, 9, No. 1—2, 127—135).—A humidifying system must be provided in advance of the steaming apparatus in order to counteract heating. The maximum evolution of heat in the formation of aniline-black occurs at 75—80°, and the oxidation is the more rapid the higher is the acidity of the bath.

CHEMICAL ABSTRACTS.

**Bismarck-brown in aniline-black dyeing.** J. FEIGUIN (Izvest. Tekstil. Prom., 1930, 9, No. 1—2, 155).—Bismarck-brown catalyses the production of aniline-black; moreover by its use the tensile strength of the fabric is increased by 8—18%.

CHEMICAL ABSTRACTS.

**Treatment of aniline-black subsequent to ageing.** J. L. HANKEY (J. Soc. Dyers and Col., 1931, 47, 59—68).—The reactions which occur in aged fabric during after-chroming depend on whether or not all the aniline originally padded into the fabric has been oxidised to aniline-black, for it appears possible for excess of aniline to combine with nigraniline. On extracting with water an aged fabric containing excess of aniline this excess is found to be present as *p*-chloroaniline and not as free aniline. Aged fabrics, whether or not containing excess of aniline, yield violet-blue-black shades by oxidation (dichromate) after extraction with cold water, and these turn green when treated with 1% sulphuric acid; the pigment in the fabrics thus appears to be quite different from a normal aniline-black pigment and resembles emeraldine products. Treatment of a developed (oxidised) black with cold water may affect its shade, but treatment with solutions of sodium sulphite and sodium thiosulphate is particularly effective. After boiling for  $\frac{1}{4}$  hr. in a 10% solution of sodium sulphite crystals the black is reduced to a greenish-grey shade which only partly recovers to the original full shade during storage for several months; after boiling for 2 min. in a 1% solution the black becomes bluer and greener, and bronziness is entirely removed. Treatment for 5 min. at the boil with a 10% solution of sodium thiosulphate removes all bronziness and leaves a rich greenish-blue-black. The odour of phenylcarbylamine is liberated when aniline-black is treated with

acids or alkalis. Safranine is formed during extraction with hot 1% hydrochloric acid if aniline or an aniline derivative was present in the chrome bath used for previous oxidation. Immediately after ageing, the dyed fabric contains an appreciable amount ( $\equiv$  1.0—1.5% HCl) of hydrochloric acid, so that mineral acid as well as inorganic salts accumulate in the chroming bath; this renders continuous methods of chroming difficult to control. The shade of a chromed aniline-black is related to its chrome content (the higher this content the redder is the black) and this is influenced by the conditions of chroming; numerous results obtained by varying the conditions of chroming for the purpose of improving the resulting shade of black are given. To ensure uniform results each batch of aged fabric should be chromed hot under standard conditions, the amount of dichromate added being almost all used up in the oxidation.

A. J. HALL.

**Nitroso-colours.** M. F. POZDIEV (Izvest. Tekstil. Prom., 1930, 9, No. 4—5, 86—88).—For the production of iron lakes ferrous sulphate (1 mol. to 5 mols. of nitroso- $\beta$ -naphthol) is preferably used. Steaming is carried out for 3—5 min. at 103—105° with fabrics dyed with vat dyes. Vaseline oil is added to the dye bath.

CHEMICAL ABSTRACTS.

**Action of nitrous acid on animal fibres, and effect of azo components on the product.** M. J. VAN TUSSENBROEK (Chem. Weekblad, 1931, 28, 62—65).—The mechanism of the formation of dyes on animal fibres which have been treated successively with nitrous acid and phenolic or amino-compounds is discussed. The properties of the dyed fabrics are described, and utilisation for photographing on to wool or silk is suggested.

S. I. LEVY.

**Properties of insoluble azo colours on the fibre in relation to their constitution. Fastness to kier-boiling and the method of testing.** F. M. ROWE (J. Soc. Dyers and Col., 1931, 47, 29—35).—The development of the insoluble azo colours (of Naphthol AS type) and the relation between the original and what have been called the "selection" patents for these colours is described. Details are given of the dye combinations and fastness tests carried out on behalf of the Petitioners (Imperial Chemical Industries, Ltd.) in the action for the revocation of B.P. 193,834, 193,866, and 199,771 (I.G.) to test the advantages alleged in these three patents, *i.e.*, greater fastness to kier-boiling and to light and a greater capacity for being discharged to a good white. Special attention was paid to kier-boiling. For this the "a" test of the German Fastness Commission, consisting essentially in 6 hrs.' boiling with 0.36% caustic soda solution, with undyed cotton was used. The results were not classified according to the Commission types, however, as these are not all azoic colours, but five new grades were chosen. In addition to laboratory tests, cuttings were also included in a large-scale kier and boiled with 0.36% caustic soda solution for 6 hrs., and also with 0.5% soda ash instead of caustic soda. The following conclusions are drawn: (1) the laboratory caustic soda test is much less stringent than the large-scale; (2) the 0.36% caustic soda boiling is a very

convenient one for the laboratory and is much more stringent than the 0.5% soda ash, but sometimes excludes colours which would pass in practice; (3) nitro-groups affect the kier-boiling adversely, and the caustic soda test is too severe for estimating suitability for a large-scale soda ash boil; (4) the selected combinations exhibited as much variation among themselves as did the unselected; (5) resistance to kier-boiling is a specific property of the coupled colour and not of the components; (6) some azoic dyeings are sufficiently fast to kier-boiling as carried out in this country with coloured goods.

L. J. HOOLEY.

**Action of boiling caustic soda (kier-boiling) on insoluble azo dyes on the fibre.** F. M. ROWE (J. Soc. Dyers and Col., 1931, 47, 35—39).—Cotton yarn dyed with (a) Fast Yellow G Base and Naphthol AS-RL, (b) Fast Red BL Base and Naphthol AS-RL, (c) Para Red, tested by plaiting with an equal weight of undyed cotton and boiling under reflux with 0.36% caustic soda solution (ratio of liquor to cotton 10:1) for 6 hrs., showed the following results. (a) With 2.9% of colouring matter originally on the fibre, 0.78% of unaltered colour could be removed by squeezing after boiling, whilst 1.7% remained on the fibre, although, owing to change in physical condition, the dyed cotton appeared to be almost stripped; from the liquor, chloroaniline and *p*-anisidine were recovered, upwards of 14% of the colouring matter originally present on the fibre being destroyed during the test, partly by reduction of the azo group and partly by hydrolysis of the arylamido-group. (b) With 2.8% of colouring matter, 1.6% remained on the fibre chemically unaltered and a new crystalline azo compound was squeezed out of the fibre, viz., *p*-amino-*o*-tolueneazo- $\beta$ -hydroxynaphthoic *p*-anisidide (black needles from toluene, m.p. 248°; acetyl derivative, m.p. 292°), formed from the original colour by reduction of the nitro-group. From the alkaline liquor *p*-anisidine was isolated. Upwards of 43% of the colouring matter originally present is destroyed partly by reduction to the aminoazo compound and partly by hydrolysis to the arylamino-group. No *p*-diamine could be detected, and if formed it would probably be destroyed by the boiling alkali. (c) With 3.46% colour on the fibre, 2.5% remained unaltered, although the fibre was practically colourless and the new azo compound formed by reduction of the nitro-group, viz., 4-aminobenzeneazo- $\beta$ -naphthol (blackish-violet needles from alcohol, m.p. 148°) were isolated; 28% of the colouring matter on the fibre was also destroyed by reduction to the aminoazo compound and possibly partly by reduction to the azo group, although no *p*-diamine could be detected. The reducing agent is cellulose in the presence of caustic soda, but small amounts of dextrose formed from impurities in grey cotton have some effect in increasing the reducing action.

L. J. HOOLEY.

**Action of diazonium salts on silk.** P. SISLEY, A. SIMONNET, and J. P. SISLEY (Bull. Soc. chim., 1930, [iv], 47, 1389—1394).—When they are diazotised and the diazonium solution is neutralised and allowed to act on silk for  $\frac{1}{2}$  hr. in the cold and in presence of sodium acetate, *o*- and *m*-chloroaniline, 2:5-dichloroaniline, *o*-, *m*-, and *p*-nitroaniline, 4-chloro-2-nitroaniline, 4-chloro-

2-anisidine, 4(?)-nitro-2-anisidine, 4-chloro-*o*-toluidine, 5-nitro-*o*-toluidine, 4-nitro-*o*-toluidine, and *p*-aminoacetanilide yield yellow, orange-yellow, or brownish-yellow dyeings which in some cases have technical value on account of their fastness to scouring and bleaching by hydrogen peroxide. Similar shades are obtained by tetrazotising thioaniline and dianisidine and coupling under similar conditions. Sulphonated amines, in general, yield shades which are not so fast. In alkaline solution nitrosoamines are formed, *p*-nitrobenzene-diazonium hydroxide yielding *p*-nitrophenylnitrosoamine (orange on silk, not fast to soaping). Aminoazo-benzene and -toluene, ethoxybenzeneazoethoxyaminobenzene, aminodimethoxyazobenzene, aminomethoxyethoxyazobenzene, and aminobenzeneazo-*p*-aminotoluene similarly couple with silk when diazotised, part of the diazonium azo compound also dyeing the silk without coupling, and, on soap-boiling, being partly converted into a hydroxy-derivative which by coupling with the undecomposed diazo compound forms a complex dye. In the case of primuline, which yields a fast yellow shade, the azofibroinazoprimumine compound preponderates. Since when *p*-nitrobenzenediazonium chloride neutralised with calcium carbonate is coupled with fibroin in presence of sodium acetate the amount of diazonium compound absorbed corresponds with 5.4%, or about half of the tyrosine present in the silk, the coupling is regarded as conditioned by the hydroxy-group of the tyrosine, and probably occurs in the *o*-position. Diazotised *p*-nitroaniline couples with tyrosine itself to give an almost theoretical yield of an orange-yellow dye,  $C_{15}H_{14}O_4N_5$ , which dyes silk and cellulose acetate. Reduction of the azo compounds from diazotised *o*- and *p*-nitroaniline and fibroin with sodium hyposulphite affords aminofibroin and *o*- and *p*-phenylenediamine, respectively, whilst reduction with dilute sodium sulphide gives yellow shades which by diazotisation and development with  $\beta$ -naphthol, in the case of *p*-nitroaniline, yield a fast red shade. Developed with  $\alpha$ -naphthylamine a violet-black is obtained which can be rediazotised and coupled with *m*-tolylenediamine to give a black. The shades obtained are very similar to those produced by coupling with much simpler compounds than fibroin, e.g., *p*-cresol, and accordingly the complex molecule has little effect on the shade, its size and insolubility merely increasing the fastness to washing (cf. A., 1927, 1212).

R. BRIGHTMAN.

**Relations between light-fading and depth of shade of dyed materials.** II. SOMMER (Z. angew. Chem., 1931, 44, 61—65).—Measurement of the rate of fading of 0.25, 0.5, 1.0, 2.0, and 4.0% dyeings of Anthracene Blue SSWG extra on scoured wool (Melton fabric), using a Zeiss step photometer, the Ostwald system of colour annotation, and Victoria Blue paper (cf. Krais, A., 1930, 1260) for determination of the bleaching quality of the light effective during fading, indicates that, contrary to the conclusions of Barker, Hirst, and Lambert (B., 1927, 811), the heaviest dyeing (4%) darkens, whereas the paler dyeings become lighter in shade. During fading the wool fibre is protected from deterioration by the dye present, this protection being approximately proportional to the depth of shade.

A. J. HALL.

**Rapid fixation of chrome colours.** V. BORISOV and N. SOKOLOV (Izvest. Tekstil. Prom., 1930, 9, No. 3, 101).—Sodium dichromate (250 g.) is dissolved in hot water (550 c.c.) and glycerin (200 g.) is added; the mordant is used when it has become dark green.

## CHEMICAL ABSTRACTS.

**Impregnation of wool fibre with fatty substances in aqueous emulsion.** L. MEUNIER and J. CORBIÈRE (Compt. rend., 1930, 191, 661–663).—Particles of 1 $\mu$  and less pass through pure wool used as a filter for aqueous emulsions of olive oil, in which a protecting emulsifier is formed *in situ* by addition of oleic acid to the oil and ammonia to the water used for dispersion. With 0–8.5% of soap, the amount of fatty matter fixed decreases owing to increase in the stability of the emulsion; for 8.5–14.5% there is a break in the fixation curve, corresponding with equilibrium between the adsorptive powers of the wool and the dispersive powers of the soap, whilst above 15% the latter effect preponderates. For less than 6.5% the composition of the adsorbed matter is the same as that of the emulsion, but for higher amounts the former is richer in oleic acid. The amounts adsorbed decrease slowly (from 67 to 60%) with increase in  $p_H$  up to 3, and then rapidly (to 5%) at the isoelectric point ( $p_H$  4.5) of the wool; they increase linearly with the mean diameter of the particles of emulsion. For  $p_H$  values up to 4.5, using 10% oleic acid emulsions, the adsorbed matter also contains 10% of oleic acid, but at higher  $p_H$  values the proportion of oleic acid fixed gradually increases. J. GRANT.

**Bleaching process.** A. P. ORANSKI (Izvest. Tekstil. Prom., 1930, 9, No. 1–2, 136–137).

**Continuous bleaching process.** N. V. FILIPPOV and B. S. VORONKOV (Izvest. Tekstil. Prom., 1930, 9, No. 1–2, 135).

**Aniline-black in the printing of naphtholated goods.** B. A. RAZUNOV and M. F. CHMEKEV (Izvest. Tekstil. Prom., 1930, 9, No. 1–2, 145–146).

**Textile oils.**—See XII.

See also A., April, 456, Colour measurement.

## PATENTS.

**Dyeing of filamentous textile substances.** E. J. ABBOTT (U.S.P. 1,775,543, 9.9.30. Appl., 10.7.29).—In dyeing oily wool yarn by the method of circulating a dye liquor through it, the yarn is first scoured by circulation of a solution of sodium sulphate or chloride followed by a solution of the acid which is subsequently used in the dye liquor; uneven deposition on the wool of the impurities removed is thus prevented and the wool is left in a condition favourable to dyeing. Suitable apparatus is described. A. J. HALL.

**Dyeing of animal fibres.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,407, 14.11.29).—Wool is dyed with sulphuric esters of leuco-vat dyes in presence of an alkali or base, *e.g.*, potassium carbonate, triethanolamine, and a reducing agent, *e.g.*, formaldehyde-sulphoxylate, the colour being developed with an acid oxidant, *e.g.*, nitrous acid, or persulphate and sulphuric acid. A wax resist may be used. C. HOLLINS.

**Manufacture of [coloured] artificial filaments, threads, bands, etc.** COURTAULDS, LTD., and H. J. HEGAN (B.P. 342,696, 2.11.29).—A solution of a dye (*e.g.*, Oil Yellow ADS) in a liquid which is insoluble in viscose (*e.g.*, high-boiling petroleum or sperm oil) is thoroughly mixed with viscose before spinning.

F. R. ENNOS.

**Varying the dyeing properties of cellulosic fibrous material.** SOC. CHEM. IND. IN BASLE (B.P. 342,689, 1.11.29. Switz., 3.11.28).—Cellulosic fibres are treated first with 15% caustic soda solution to form alkali-cellulose and then with an allyl halide (bromide or chloride) in gaseous form or in the presence of a solvent; the fibres are thus partly converted into allyl cellulose and become resistant to direct dyes, although they acquire an increased affinity for basic dyes. Should hydrolysis occur in the treated fibres during subsequent storage, the products of hydrolysis can exert no tendering action. A. J. HALL.

**Production of colour reserves under vat dyeings.** DURAND & HUGUENIN A.-G., Assees. of I. G. FARBENIND. A.-G. (B.P. 340,085, 5.11.29. Ger., 5.11.28).—Coloured reserves, using ester salts of vat dyes, are obtained under vat dyeings by a process which is a combination of the known method, in which heavy-metal salts are used, with previously described processes (B.P. 220,964, 281,336, 306,800, and 298,088; B., 1925, 879; 1929, 280, 320; 1930, 320); the coloured reserve paste contains the ester salt of a leuco-vat dye made into a paste with glycol or one of its derivatives, a heavy-metal salt, an oxidising agent, and a substance capable of liberating an acid during steaming, such as dichlorohydrin and potassium methyl sulphate. A. J. HALL.

**Coloration of materials made of or containing cellulose derivatives.** BRIT. CELANESE, LTD., and R. H. PARKINSON (B.P. 343,124, 8.11.29).—Materials composed of cellulose esters or ethers are coloured by immersion in a colloidal solution of a suitable metal oxide (*e.g.*, the oxide or hydroxide of zinc, titanium, chromium, aluminium, iron, or tin), whereby absorption (cf. B.P. 343,121; following abstract) occurs, followed by dyeing with a mordant dye. Alternatively, the order of these treatments may be reversed.

A. J. HALL.

**Treatment of materials made of or containing cellulose derivatives.** BRIT. CELANESE, LTD., and R. H. PARKINSON. In part from C. DREYFUS and H. PLATT (B.P. 343,121, 6.11.29).—Fibres composed of cellulose esters or ethers are delustred, weighted, or made resistant to hot-ironing by treatment with colloidal solutions of inorganic substances (cf. B.P. 343,124; preceding abstract) such as the oxides of aluminium, titanium, tin, silicon, zinc, cerium, etc., barium carbonate or sulphate, zinc sulphide, china clay, and insoluble silicates, whereby absorption of these substances occurs. Swelling agents may be present to facilitate absorption. A. J. HALL.

**Manufacture of [dull-lustre] viscose silk.** BREDAVISADA, LTD., H. SAGAR, E. VAN WEYENBERGH, and R. O. JONES (B.P. 344,288, 9.5.30).—Dull-lustre viscose silk is obtained by spinning viscose solutions to which has been added up to 1% (calc. on the wt. of viscose) of a



difficultly hydrolysable ester of a hydrogenated phenol and a mono- or poly-carboxylic acid, such as the methyl-cyclohexanol ester of cinnamic acid, methyladipic acid, and oleic, stearic, and palmitic acids. A. J. HALL.

**Manufacture of artificial silk filaments [of modified lustre].** J. A. SINGMASTER (B.P. 342,743, 9.8.29. U.S., 18.5.29).—To the solution of the cellulosic material is added a solution of a non-volatile oleaginous substance (heavy white petroleum oil) in a solvent non-reactive with the cellulose solution, together with a pigment, preferably titanium dioxide of particle size  $0.75\mu$  or less, the whole being passed through at least part of the filtering system before spinning.

F. R. ENNOS.

**[Delustring] treatment of textile [cellulose ester and ether] materials.** BRIT. CELANESE, LTD. (B.P. 343,698, 20.1.30. U.S., 21.1.29).—Knitted cellulose acetate (etc.) garments are delustred by the usual methods (e.g., by boiling in a soap solution, or by precipitation within the fibres of barium sulphate or zinc sulphide) while stretched on suitable supports for the purpose of avoiding the formation of wrinkles and distortion.

A. J. HALL.

**[Apparatus for] scouring and carbonising wool and other hair and fibrous materials.** L. A. and A. E. BOWLER (B.P. 343,120, 5.11.29. Austral., 14.8.29).—Wool is treated with scouring or acid carbonising liquors by passage through a bath of the liquor, the wool being carried between two endless moving brattices; balling and felting of the wool is thereby avoided or reduced.

A. J. HALL.

**Manufacture of [electric] insulating fabrics.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 342,848, 27.12.29).—Mercerised cotton fabrics are impregnated with suitable cellulose derivative lacquers, such as may be prepared from a mixed ester of cellulose with butyric, lauric, and acetic acids.

A. J. HALL.

**[Preparation of water-resistant] textile filaments and fabrics formed of cellulosic material.** R. LANT and W. KORESKA (B.P. 343,104, 10.9.29).—Fabrics are esterified to an extent such that their woven structure is not substantially destroyed by treatment with a higher fatty acid chloride in the presence of pyridine at  $80-120^\circ$ .

A. J. HALL.

**Sizing of [cellulose] acetate silk.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 341,516, 15.10.29).—Water-soluble hydroxyalkyl ethers of carbohydrates (starch or cellulose), which are obtained by the interaction of alkylene oxides (e.g., propylene and ethylene oxides) with such carbohydrates in the presence of aqueous alkalis at moderate temperatures until the products are soluble in water and in alcohol, are used after neutralisation with acids for sizing cellulose acetate silk; the size can subsequently be removed by treatment with water.

A. J. HALL.

**Production of ornamental [cellulose acetate] fabric.** BRIT. CELANESE, LTD. (B.P. 344,155, 28.1.30. U.S., 5.2.29. Addn. to B.P. 342,075; B., 1931, 391).—Fabric having a cockled or pebble appearance is produced by weaving in such a way that yarns of cellulose ester or ether alternate with other yarns in the warp or

weft, and then subjecting the fabric to a wet treatment (e.g., immersion in caustic soda, or a soap boil) capable of causing the last-named yarns (e.g., cotton, wool, and real silk) to shrink in length.

A. J. HALL.

**[Production of effects on] filaments, yarns, fabrics, and other materials made of or containing cellulose esters.** BRIT. CELANESE, LTD., and R. P. ROBERTS (B.P. 343,953, 18.11.29).—Cellulose esters are made to exhibit different degrees of resistance to hydrolysis by suitable means, such as varying the solvent content of the evaporative medium. The filaments (etc.) or fabric made therefrom, after differential hydrolysis, are cross-dyed with cotton dyes.

F. R. ENNOS.

**Production or treatment of cellulosic or cellulose-derivative materials or natural textile materials.** H. DREYFUS (B.P. 343,949, 23.10.29).—Metallic radicals other than alkali-metal radicals are introduced into threads or films, particularly those composed of a cellulose ester or ether, by treatment with solutions (usually in organic solvents) of organo-metallic compounds, in which the metal is directly united to carbon or in which the metal replaces the hydrogen of a hydroxyl group, or of the organic esters of metallic acids; zinc diethyl, lead tetraethyl, zinc methoxide, aluminium phenoxide, and titanium compounds represented by the formulae  $\text{TiO}(\text{OH})(\text{OEt})$ ,  $\text{TiO}(\text{OEt})_2$ ,  $\text{Ti}_2\text{O}(\text{OEt})_6$ , and  $\text{Ti}_2\text{O}(\text{OPh})$ , are suitable compounds. Alternatively, such organo-metallic compounds may be introduced into the solutions used in spinning the threads or films. The treated threads or films may be further treated by various methods for decomposition of the organo-metallic compounds so that a metallic residue remains within the materials, thereby modifying its lustre, weight, affinity for dyes, and resistance to hot-ironing.

A. J. HALL.

**Cellulose derivatives.**—See V. Adhesive.—See XV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Chamber [sulphuric acid] process. XXII. Automatic nitric acid feeder to the chamber system—photoelectric method.** M. MATSUI and T. NODA (J. Soc. Chem. Ind., Japan, 1930, 33, 518—521 n; cf. B., 1931, 110).—The absorption of light by nitrogen tetroxide has been measured by means of a photoelectric (potassium hydride) cell; as the extinction, or the photoelectric current, is quantitatively related to the pressure or concentration of the gas, very dilute mixtures of nitric oxides can be analysed by this method. The photoelectric current, if suitably amplified, could be used as an automatic control for the supply of nitric acid to the chamber system.

E. LEWKOWITZCH.

**Electrolytic cells for chlorine and caustic soda.** C. L. MANTELL (Chem. and Met. Eng., 1931, 38, 88—90).—The cells may be divided into three classes: (1) mercury cells, e.g., those of the Castner type; (2) rectangular-diaphragm cells, e.g., the Hargreaves-Bird cell; and (3) those having a cylindrical diaphragm, as in the Gibbs cell. The cell containers are usually built of steel or concrete, with concrete or slate covers. The cathodes, except for those of class (1), are of steel in

the form of perforated plate or gauze, the anodes of graphite, and the diaphragms usually of asbestos. Generally, purified saturated brine heated to the optimum temperature is fed into the cells. Details of the performances of different cells are given.

D. K. MOORE.

**Ammonia synthesis by the low-pressure Mont Cenis process.** I. W. F. SCHOLVIEN (Chem. and Met. Eng., 1931, 38, 82—86).—Coke-oven gas is freed from sulphur compounds, compressed to 12 atm. pressure, and denuded of carbon dioxide by scrubbing with water and caustic soda. The gas is cooled to  $-40^{\circ}$  in heat exchangers and a refrigerator, and then washed in a Linde-Bronn separator with liquid nitrogen. Its constituents, with the exception of hydrogen and part of the nitrogen, are liquefied, and after mixing with nitrogen obtained by the liquefaction of air contains 0.05% CO and 0.05% O. This gas is compressed to 100 atm., heated in heat interchangers to  $300^{\circ}$ , and passed over a nickel catalyst to convert the carbon monoxide into methane and water, and the oxygen into water. The gas is cooled in heat exchangers, and part of its moisture is deposited and mixed with gas coming from the ammonia reaction chamber. After cooling, the ammonia is liquefied at  $-55^{\circ}$ . The gas, containing 0.2—0.5%  $\text{NH}_3$ , is heated in heat interchangers to  $320^{\circ}$  and passed into a catalyst chamber, where it is partly converted into ammonia at  $370^{\circ}$ , the issuing gas containing 12—13%  $\text{NH}_3$ .

D. K. MOORE.

**Process giving high yields in the manufacture of urea.** A. KLEMENC (Z. Elektrochem., 1930, 36, 799—802).—A summary of work already published in detail (cf. A., 1930, 1387).

H. F. GILLBE.

**Preparation of potassium nitrate and alumina by double decomposition of potassium chloride and aluminium nitrate.** I. The system:  $3\text{KCl} + \text{Al}(\text{NO}_3)_3 \rightleftharpoons 3\text{KNO}_3 + \text{AlCl}_3$ . II. Treatment of clay with nitric acid. H. TANAKA (J. Soc. Chem. Ind., Japan, 1930, 33, 488—492 B, 493—495 B).—I. The solubilities of aluminium chloride and nitrate from 0 to  $100^{\circ}$  have been determined. The data obtained for the former are in disagreement with the figures given by Gerlach (cf. "Solubilities of Inorganic and Organic Compounds," Seidell, 1919), and the nitrate shows abnormal changes in solubility between  $0^{\circ}$  and  $10^{\circ}$  (minimum about  $5^{\circ}$ ). The isothermal data at  $0^{\circ}$  and  $10^{\circ}$  have been obtained and plotted for the four ternary systems from aluminium and potassium nitrates and chlorides consisting of water and two salts with a common ion, and for the quaternary system  $(\text{K}, \text{Al})(\text{Cl}, \text{NO}_3)$ —water at  $10^{\circ}$  (at  $0^{\circ}$ ,  $15^{\circ}$ , and  $20^{\circ}$  in the original) and the diagrams have been applied to determine the range of solutions to be employed in the process.

II. The dissolution velocity in nitric acid of alumina (from a Chosen clay) is much the same as in sulphuric acid. Similarly the effect of temperature on the velocity constant can be expressed by the Arrhenius formula (temperature coefficient about 4460), and the yield of alumina is only slightly affected by the concentration of the nitric acid.

E. LEWKOWITSCH.

**Enriching Khibinsk apatite by flotation.** G. O. ERCHIKOVSKI (Udobr. Urozhai, 1930, 2, 109—114).—

With crude birch tar (1.3 kg., with 0.3 kg. of potassium hydroxide per ton) 85% of apatite was extracted. Guaiacol or oleic acid (0.7—0.13 kg. per ton) also gave good results.

CHEMICAL ABSTRACTS.

**Thermophosphates from Khibinsk apatite-nepheline rocks.** S. N. ROZANOV (Udobr. Urozhai, 1930, 2, 105—109).—When fused with sodium carbonate at  $1000^{\circ}$  for 1 hr. (Wiborg's method) material containing 11.55 or 31.52%  $\text{P}_2\text{O}_5$ , respectively, required 25% or 50% of sodium carbonate for complete conversion of the phosphorus into the citrate-soluble form. Volter's, Easthard's, and the Rhenania methods were less satisfactory.

CHEMICAL ABSTRACTS.

**Polymerisation and solubility of phosphates.** S. S. DRAGUNOV (J. Appl. Chem., Russia, 1930, 3, 797—805).—The salt  $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$  was heated for 1.5 hrs. at various temperatures; its solubility in boiling aqua regia and the rate of dissolution in water at  $25^{\circ}$  were determined. The fused salt is a good fertiliser.

CHEMICAL ABSTRACTS.

**Reduction reactions with calcium hydride. II. Determination of sulphur in sulphur[—containing] oils and rubber.** W. E. CALDWELL and F. C. KRAUSKOPF (J. Amer. Chem. Soc., 1930, 52, 3655—3659; cf. A., 1929, 1411).—A mixture of the oil or finely-divided rubber and calcium hydride is heated in a bomb at  $700^{\circ}$ , and the calcium sulphide determined in the residue by acidifying and titrating with iodine. The purity of sulphur and the sulphur content of sodium thiosulphate can be determined by the same method. H. BURTON.

**Composition of commercial calcium arsenate.** C. M. SMITH and C. W. MURRAY (Ind. Eng. Chem., 1931, 23, 207—208).—The usual practice of calculating the total arsenic in insecticides to  $\text{Ca}_3(\text{AsO}_4)_2$  is known to be probably incorrect, and results of analyses of sixteen commercial products showed that total  $\text{As}_2\text{O}_5$  ranged only from 40.3 to 44.4%. Calcium carbonate and calcium oxide contents averaged 6.4% and 6.6%, with a somewhat wider range. Calculated on a molecular basis,  $\text{CaO}/\text{As}_2\text{O}_5$ , the ratios were 3.0—4.1 (aver. 3.4), which demonstrates that arsenic in commercial calcium arsenate practically never exists solely as tricalcium arsenate, but that a considerable portion of it is in the form of a basic arsenate the composition of which is undetermined, but may possibly consist of a mixture of  $3\text{CaO}, \text{As}_2\text{O}_5$  and  $4\text{CaO}, \text{As}_2\text{O}_5$ .

C. A. KING.

**Active alumina catalyst.** E. V. ALEXEEVSKI and I. G. PREIS (J. Appl. Chem., Russia, 1930, 3, 859—862).—Air-dried hydrated alumina (70%, sieve 900 per cm.<sup>2</sup>) and kaolin (30%) are mixed and steamed for 1 hr.; a paste with water is formed into rings 12 mm. in diam. and 20 mm. high, which are dried at  $150^{\circ}$ . The catalyst is used for dehydration of ethyl alcohol.

CHEMICAL ABSTRACTS.

**Adsorption phenomena on the surface of a vanadium catalyst.** I. E. ADADUROV (J. Appl. Chem., Russia, 1930, 3, 833—842).—Oxygen is not adsorbed; the adsorption of sulphur dioxide, whereby  $\text{V}^{\text{v}}$  is reduced to  $\text{V}^{\text{iii}}$ , depends on the moisture content of the gases. Since the velocity of the gases does not influence the efficiency of contact, the reaction may take place in the gaseous phase.

CHEMICAL ABSTRACTS

**Sulphur monochloride. III—V. Determination of total chlorine and sulphur in a naphtha solution of sulphur monochloride.** H. ENDOH (J. Soc. Chem. Ind., Japan, 1930, 33, 287—290 B, 290—292 B, 292 B).—III. Total chlorine is determined by adding 1% sodium nitrate solution, 10% copper sulphate solution, and water, with shaking. The liberated sulphur remains in the lower, aqueous layer. The mixture is heated on a water-bath and then boiled until nearly all the naphtha is removed. The sulphur and remaining naphtha are filtered off and the chloride in the filtrate is determined as silver chloride. Total sulphur is determined in the following two ways: (a) by adding glacial acetic acid and bromine, followed by hot water. The solution is heated on a water-bath until all the bromine is driven off, and filtered. Sulphate in the filtrate is determined gravimetrically as barium sulphate. The relative amounts of naphtha and glacial acetic acid are very important, the most satisfactory results being obtained with 10 c.c. of the former and 20 c.c. of the latter. (b) The solution in naphtha is added to a concentrated ethyl alcoholic solution of sodium ethoxide, heated until naphtha and alcohol are completely expelled, and evaporated to dryness. The residue is washed out into a crucible, evaporated to dryness, fused with a mixture of sodium carbonate and potassium nitrate, dissolved in water, and filtered. The sulphate in the filtrate is determined as barium sulphate. Free sulphur dissolved in the sulphur chloride may be determined by either of the two methods.

IV—V. The method of Dubosc ("Les Caoutchoucs Factices ou Huiles Vulcanisées," p. 199) for determining sulphur and chlorine is modified for use in the determination in sulphur monochloride. C. W. GIMBY.

**Flow of gases in pipes.**—See I. Litharge.—See II. Corrosion by phosphoric acid. Germanium.—See X. Magnesium arsenate insecticide.—See XVI. Calcium carbonate hydrate.—See XVII.

See also A., April, 419, Adsorptive capacity of silica gels. 423—4, Colloidal silver sols. 424, Colloidal lead, lead phosphate, and selenium. 431, Melting in the system  $Z + O_2$ -BeO. System  $KNO_3$ - $Ca(NO_3)_2$ . 432, System  $RbNO_3$ - $RbCl$ . 439, Catalysts for hydrogenation. 440, Synthetic ammonia. Reduction of arsenic acid and arsenates. 444, Stable blue cupric hydroxide. Silver iodide. Zinc and barium ferrates. 445, Anhydrous aluminous chloride. Silicic acids. 446, Titanium white. 448, Pure perchloric acid. Working up iodine residues. 451, Detection of chlorate in presence of perchlorate and nitrate. Determinations of sulphuric acid, sulphur, persulphate, and nitrites. 452, Determinations of phosphoric acid, carbon monoxide, helium and neon, potassium, and magnesium. Separation of beryllia and alumina. 455, Density determinations with salts.

#### PATENTS.

**Catalyst carriers [for the contact sulphuric acid process].** L. F. NICKELL (B.P. 343,441, 14.11.29. U.S., 8.8.29).—A porous material, such as kieselguhr, is mixed into a paste with sodium silicate solution and

pelleted, the pellets after drying being heated at 400° in a current of an acid gas, e.g., from sulphur burners. A matrix of silica is thus produced. W. J. WRIGHT.

**[Manufacture of stable products containing] nitrosylsulphuric acid.** A. KLEIJN, and N.V. CHEM. IND. VAN HASSELT (B.P. 344,213, 27.2.30).—Nitrosylsulphuric acid for use, e.g., in bleaching flour, is stabilised by grinding with absorptive material, e.g., charcoal or silica gel, and mixing the product with alkaline-earth compounds capable of forming non-corrosive products with sulphuric acid, e.g., calcium carbonate, phosphate, or hydroxide. L. A. COLES.

**Concentration of nitric acid.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,684, 2.8.29. U.S., 2.8.28).—Vapours of aqueous nitric acid are passed up a tower, without admission of steam, in countercurrent to a mixture of dilute nitric acid and fuming sulphuric acid, which is not preheated. The vapours of nitric acid (95%  $HNO_3$ ) leaving the top of the tower are condensed, the liquid from the bottom being boiled and its vapours dephlegmated and returned to the dilute nitric acid supply vessel. W. J. WRIGHT.

**Manufacture of phosphoric acid and gaseous mixtures suitable for the synthesis of ammonia.** SOC. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTRI., Assees. of E. VOUTURON (B.P. 343,683, 9.1.30. Ger., 9.1.29).—Phosphorus vapour, e.g., that present in the gas mixture evolved from an electric furnace for the production of the element, is oxidised by a mixture of carbon dioxide and nitrogen, together with oxygen, if desired, and the carbon monoxide produced is treated with steam to yield carbon dioxide and hydrogen. The phosphorus oxidation products may be removed as the oxides before the treatment with steam or afterwards as phosphoric acid, and the carbon dioxide and residual monoxide are removed by the usual means from the final gas mixture, to yield a mixture suitable for the synthesis of ammonia. L. A. COLES.

**Manufacture of sodium cyanide.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 342,703, 4.11.29).—For the production of briquettes of high mechanical strength, the dry powder is heated to 50° and, while hot, subjected to a pressure of 700—1000 kg./cm.<sup>2</sup> W. J. WRIGHT.

**Production of potassium nitrate.** KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 343,796, 1.4.30. Ger., 19.4.29).—A mixture of potassium chloride with a quantity of crystalline aluminium nitrate 70—75% in excess of that required theoretically (or less if the mixture is stirred continuously) is heated until no more oxides of nitrogen or chlorine-containing gases are evolved; potassium nitrate is extracted from the product by hot potassium nitrate solution, and the residual aluminium oxide is converted into the nitrate for re-use by treatment with the oxides of nitrogen obtained in the first stage, after these have been treated with hot lime to remove chlorine compounds. L. A. COLES.

**Production of potassium sulphate and soda.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 343,691, 15.1.30. Ger., 5.3.29).—A solution containing ammonium chloride

and bicarbonate obtained by the ammonia-soda process is stirred with calcium (or strontium) sulphate and, after removal of the calcium (or strontium) carbonate formed and subsequent addition of crude sylvinit to the solution, the liquor is treated with ammonia to precipitate a mixture of glaserite ( $3K_2SO_4 \cdot Na_2SO_4$ ) and potassium chloride, which is removed and treated with water to yield solid potassium sulphate and sodium chloride solution. After removal of the potassium sulphate, the solution is added to the mother-liquor from the filtration of the glaserite-potassium chloride mixture, and the mixed solution, after removal of ammonia by suction or heating and of ammonium chloride by cooling and filtration, is used in the ammonia-soda process.

L. A. COLES.

**Production of alkali phosphates.** METALLGES. A.-G. (B.P. 343,829, 29.4.30. Ger., 1.2.30).—Sodium chloride is heated with ferrous sulphide in presence of oxygen and steam, the sodium sulphate produced being converted into trisodium phosphate and ferrous sulphide by heating it with ferrophosphorus. The ferrous sulphate is utilised for further conversion of sodium chloride, and the ferric oxide produced by this conversion is heated with a mineral phosphate, *e.g.*, tricalcium phosphate, and coal so as to form ferrophosphorus.

W. J. WRIGHT.

**Treatment of phosphate rock and the like.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 344,135, 13.1.30. Norw., 14.1.29. Addn. to B.P. 339,340; B., 1931, 201).—Nitric acid in the mother-liquor remaining after removal of the calcium nitrate crystals in the prior process is removed by distillation *in vacuo* before or after residual calcium has been precipitated and removed as its sulphate by the addition of sulphuric acid or ammonium or an alkali sulphate; the residual phosphoric acid is concentrated or worked up to ammonium or alkali phosphates etc. Alternatively, the mother-liquor is concentrated and the remaining calcium precipitated and removed as the nitrate by the addition of concentrated nitric acid, the solution being worked up to nitric and phosphoric acids as before. L. A. COLES.

**Treatment of solutions obtained in leaching phosphate rock with nitric acid and ammonium sulphate.** KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G. (B.P. 343,701, 21.1.30. Swed., 28.11.29).—The solutions are neutralised with ammonia to a point such that mono- or di-ammonium phosphate is present in solution together with ammonium nitrate, and the phosphate formed is recovered alone or in admixture with ammonium nitrate by evaporation to a suitable concentration followed by cooling and filtration; residual phosphates in solution (1.5–2%) are then precipitated and removed by the addition of calcium nitrate and a little ammonia, leaving a solution which on evaporation yields pure ammonium nitrate. L. A. COLES.

**Production of detergent.** A. H. COWLES, ASST. to ELECTRIC SMELTING & ALUMINUM Co. (U.S.P. 1,774,872, 2.9.30. Appl., 2.3.26).—Alkali carbonate (0.75–1.25 mols.) is fused with silica (1 mol.). The ground product is mildly hygroscopic and dissolves readily in water. If the silica contains iron or aluminium compounds as impurities, their equivalent in alkali chloride is added

before fusion, whereby these metals are volatilised as the chlorides. E. LEWKOWITSCH.

**Manufacture of double salts of calcium nitrate.** LONZA ELEKTRIZITÄTWERKE U. CHEM. FABR. A.-G. (B.P. 343,636, 9.12.29. Ger., 31.12.28).—An alkali (potassium) or ammonium nitrate (1 mol.) is added to a hot 65–82% solution containing 5 mols. of calcium nitrate and the solution is cooled to effect crystallisation of a double salt,  $Ca(NO_3)_2 \cdot MNO_3 \cdot 10H_2O$ , suitable for use as a fertiliser. Alternatively, the added nitrate may be replaced by another alkali or ammonium salt, *e.g.*, potassium chloride, in quantity such that, by interaction of it with the calcium nitrate, a mixture of nitrates in the desired ratio is obtained. L. A. COLES.

**Manufacture of bleaching powder.** IMPERIAL CHEM. INDUSTRIES, LTD., and C. CARTER (B.P. 344,012, 26.11.29 and 10.4.30).—A suitable quantity of water to control the temperature and to prevent premature drying is sprayed into rotary tubes etc. for the manufacture of bleaching powder, diluted chlorine being introduced into the tubes in a hot and dried or partly dried condition.

L. A. COLES.

**Regeneration of zinc chloride lyes containing calcium chloride.** I. G. FARBENIND. A.-G. (B.P. 342,775, 8.11.29. Ger., 9.11.28).—The calcium chloride in the lyes is precipitated by adding zinc sulphate, either before or during evaporation.

W. J. WRIGHT.

**Manufacture of aluminium chloride, and apparatus therefor.** IMPERIAL CHEM. INDUSTRIES, LTD., J. W. PRITCHARD, and D. F. DOUSE (B.P. 343,785, 21.3.30).—Molten aluminium is caused to combine with chlorine in an insulated vessel, the hot vapours being led into a separate vessel in which alumina and carbonaceous matter are treated with chlorine, which may be preheated. The temperature of this latter reaction is thus maintained without external heating.

W. J. WRIGHT.

**Production of solid carbon dioxide.** P. STAPP (B.P. 343,012, 7.11.29).—Liquid carbon dioxide is cooled almost to the triple-point temperature, whereupon by slow release of the gas pressure above the liquid, the latter is caused to freeze to carbon dioxide ice.

H. ROYAL-DAWSON.

**Manufacture of hydrogen.** W. P. ROGERS (B.P. 344,043, 29.11.29).—The steam and reducing gases in the process described in B.P. 251,124 (B., 1926, 584) are preheated by passing along a duct in the outer wall before they enter the inner duct from which they pass transversely into the charge; the issuing gases similarly traverse the whole length of an outer duct before they leave the retort.

L. A. COLES.

**Detection and determination of the proportions of gases such as carbon monoxide in gaseous mixtures.** C. S. W. GRICE (B.P. 343,724, 7.2.30).—A measured volume of the gaseous mixture is drawn into a cylindrical chamber by means of a piston and forced into a vessel containing an absorbent for water and a catalyst for the oxidation of carbon monoxide. From this vessel it passes through a tube with a Schrader valve into a glass vessel containing dilute alkali solution

and an indicator. When a standard colour change occurs, the number of piston strokes is recorded, and from previous calibration the corresponding percentage of carbon monoxide in the gaseous mixture is determined.

W. J. WRIGHT.

Imitation ice.—See IX. Acid-proof alloys.—See X. Fertilisers.—See XVI.

### VIII.—GLASS; CERAMICS.

Coloration of glasses and related materials by  $\beta$ - and  $\gamma$ -rays. J. HOFFMAN (Glastech. Ber., 1930, 8, 482—490; Chem. Zentr., 1931, i, 338).—Brown coloration probably arises from the presence of potassium and barium. Colourless glasses containing manganese and iron become blue and violet; these colours do not appear if the glass is coloured green or brown with iron. Crystal quartz is converted into smoky quartz; sintered quartz glass becomes grey. Glasses which by irradiation have become yellow or brown become grey on devitrification. The grey colour disappears when the material is heated or crushed. Reddish irradiation colours were observed only with lead glasses; these also fluoresce in ultra-violet light.

A. A. ELDRIDGE.

Insulation of pyrex glass after heating *in vacuo*. J. H. MITCHELL (Phil. Mag., 1931, [vii], 11, 748—753).—An apparatus is described for measuring the change in conductivity produced by heating for 30 min. and cooling for 16 hrs. in pyrex glass which had previously been baked-out in a vacuum. A general increase of conductivity was found for 55—80°, a decrease for 250—300°, and a marked increase for 350—400°; the second effect is attributed to thorough drying and the third to a surface change. Attempts to restore low conductivity by immersion in liquid air produced no effect; allowing to remain in air or heating to 500° produced partial, and treatment with hydrofluoric acid produced complete, restoration of the original low conductivity.

N. M. BLIGH.

Influence of firing temperature and furnace gases on the development of ceramic colours and glazes. E. BERDEL (Farben-Chem., 1930, 1, 13—16; 1931, 2, 12—15).—The firing technique is described in general outline for the different classes of ware in relation to the various methods of decorating pottery, viz., coloured engobes or slips, coloured glazes, and on-glaze and under-glaze colours. The subject is treated under the headings: bricks and coarse pottery, majolica and faience, earthenware, vitreous earthenware (stoneware), and porcelain.

F. SALT.

Electrical dewatering of clay suspensions. C. E. CURTIS (J. Amer. Ceram. Soc., 1931, 14, 219—263).—Electrophoresis and its practical applications to the purification and dewatering of clays is reviewed in detail. Investigations by the author on Mica (Washington) kaolin showed that (a) the concentration of electrolyte which gives the best results in electrophoresis was that giving the greatest dispersion as judged by settling tests; (b) addition of a deflocculant greatly increases the efficiency of dewatering, the best results being obtained by addition of dry sodium silicate (0.6% or less, on the weight of dry clay in suspension); (c) the area of the

cathode has little effect; (d) in most cases electrophoresis raises the  $p_H$  of the suspension, but decreases that of the deposited clay to neutrality or lower. In the case of very fine-grained clays, dewatering by electrophoresis is considered more economical than filter-pressing.

J. A. SUGDEN.

Suggested relations of sodium silicate, adhesion tension, and angle of contact to liquid absorption in clays. G. I. WHITLATCH (J. Amer. Ceram. Soc., 1931, 14, 154—161).—The addition of small amounts of sodium silicate to clays free from excess of lime and other fluxes causes a decrease in the amount of water absorbed, the effect passing through an optimum as the silicate content increases. If lime or other type of flux is present in excess, the absorption of silicate-water solution is equal to or greater than that with water alone. The type of silicate required to give the maximum wetting effect for a minimum water absorption is specific for each clay, and the amount of silicate required to obtain the maximum reduction of absorption is considerably less than that usually required for maximum deflocculation of clay slips. These effects can be explained by changes in the adhesion tension and angle of contact of the liquid-solid interface. The bearing of this hypothesis on flocculation and plasticity is indicated.

J. A. SUGDEN.

Measurements of the plasticity of clays. G. W. S. BLAIR (J. Physical Chem., 1931, 35, 374—382).—An empirical test for the plasticity of a clay, based on the rolling into a thin thread, is described. The ordinary flow constants, mobility and shearing strength (or yield value), of different clays flowing through a capillary tube under stress at the same concentration depend on the hydration capacity of the clay rather than on its plasticity. By comparing the flow of clay pastes at shearing stresses low enough for the material to flow as a solid plug and at constant shearing strength of the water envelope surrounding the plug, values are obtained for the critical shearing stress of material, which depend on a property closely allied to plasticity. This property has been named "flow-plasticity." The effect of electrolytes on the flow-plasticity of kaolin is described. A mixture of sodium hydroxide and sodium chloride has a marked plasticising effect, as has also sulphuric acid or a mixture of sulphuric acid and aluminium sulphate. With hydrochloric acid plasticity increases up to an optimum concentration of 0.44% HCl and then decreases.

L. S. THEOBALD.

Effects of autoclave treatments on ceramic bodies and clays. H. H. HOLSCHER (J. Amer. Ceram. Soc., 1931, 14, 207—218).—Comprehensive tests showed that autoclave treatment causes an increase in volume of porous bodies with a roughly proportional increase in weight; the effect is greater with bodies than with pure clays, and therefore cannot be due to rehydration of the clay molecule. Again, as only part of the water is removed from the treated ware at 110°, it is concluded that the water taken up cannot all be merely filling the pores. Further tests indicate that the effect is due to a combination of adsorption and partial filling of the pores. The reaction to autoclave treatment is a function

of both the composition and the absorption of the body, but with bodies of the same composition increased absorption is accompanied by increased change in length.

J. A. SUGDEN.

**Influence of time on the maturing temperature of whiteware bodies.** I. F. H. NORTON and F. B. HODGDON. II. C. L. NORTON, JUN. (J. Amer. Ceram. Soc., 1931, 14, 177—191, 192—206).—I. The maturing (decrease in porosity and increase in shrinkage) of a number of earthenware and whiteware bodies was found to follow the equation  $\log(k_2/k_1) = A[(1/T_1) - (1/T_2)]$ , where  $k_1$  is the reaction velocity at the temperature  $T_1$ , and  $A$  a constant depending on the particular body. The firing times ranged from 10 to 10,000 min. and the temperatures from 1045° to 1300°.

II. Microscopical examination of the matured bodies showed that time is an important factor in firing. Although long-time low-temperature and short-time high-temperature firings may give the same degree of maturity, the former gives a structure with more quartz dissolution and mullite development. The growth of mullite is by the dissolution of the quartz in the pseudomorphic feldspar grains and subsequent crystallisation within the grain boundary. In no case were the crystals seen to pierce the grain boundary.

J. A. SUGDEN.

**Electric-furnace production of high-heat-duty refractories.** F. W. SCHROEDER (Ind. Eng. Chem., 1931, 23, 124—126).—Primarily adapted for use in glass furnaces, a uniform mixture of clays of high alumina content, adjusted by adding kaolin, is first calcined in a gas-fired rotary kiln and then in an electric furnace from which molten aluminium silicate is tapped and cast in moulds built up from sand slabs. The clay is fused at about 2200° and tapped at 1870°; the castings in the moulds are annealed for 6—10 days. The refractory material obtained is equivalent to Seger cone 37 and does not deform gradually under load-temperature conditions below the melting range. It is vitreous and cannot be worked to shape except by grinding. The coefficient of expansion is approximately half that of firebrick. The slabs for moulds are made from sand and linseed oil, which are baked and trimmed to size.

C. A. KING.

**Refractory natural stone for industrial furnaces.** H. KALPERS (Giesserei, 1930, 17, 1045—1047; Chem. Zentr., 1930, ii, 3835).—Schlenkermann's stone ( $\text{SiO}_2$  90.59,  $\text{Al}_2\text{O}_3$  6.85,  $\text{Fe}_2\text{O}_3$  0.21,  $\text{K}_2\text{O}$  0.22,  $\text{Na}_2\text{O}$  0.90, loss on ignition 1.10%) has m.p. about 1770—1790°. The properties and use of the stone are described.

A. A. ELDRIDGE.

**Special refractories for metallurgical research.** D. TURNER (Trans. Faraday Soc., 1931, 27, 112—124).—Different types of refractory materials, which have been developed for use in various investigations, and their methods of production are described and discussed. Their treatment in the manufacture of crucibles and gastight muffles for use at high temperatures is dealt with, as well as the application of crucibles and other apparatus made of these materials for various purposes and processes, e.g., the study of the iron-oxygen system, melting in the high-frequency induction furnace, alloys

of iron and manganese, tubes for the protection of thermocouples, and the production of moulded articles of complex design.

O. J. WALKER.

**Effect of titania in refractory materials.** H. HIRSCH (Tonind.-Ztg., 1930, 54, 773—776; Chem. Zentr., 1930, ii, 1115—1116).—Titanium dioxide in the amounts in which it is usually present (0.5—2%) has little effect on the refractoriness or resistance to pressure of clays, grogs, or silicate rocks, and can be regarded as alumina.

L. S. THEOBALD.

**Slagging of refractory materials.** IV. Corrosive action and constitution of iron smelter slags. H. SALMANG and F. SCHICK (Arch. Eisenhüttenw., 1930—1, 4, 299—316; Stahl u. Eisen, 1930, 50, 1814. Cf. Miksch and Salmang, B., 1930, 189).—The corrosive action on refractories of the bases in metallurgical slags decreases in the order: lime, ferrous oxide and magnesia, manganese oxide. Ferric and manganic oxides behave like acids and retard the corrosive action of the bases, and at high temperatures the presence of alkalis in a slag is not nearly so dangerous to refractory linings as is the presence of much lime. The highly corrosive action of lime is ascribed to the considerable dissociation which takes place in calcium silicates at high temperatures; alkali silicates, on the other hand, are much more stable. Phosphorus pentoxide in small quantities retards the corrosive action of the bases, but in large proportions it has a highly corrosive action at temperatures above 1500°. The slagging action ( $K$ ), measured in mm. of refractory dissolved, of basic iron smelter slags may be expressed by the following: at 1410°,  $K = 1.25 \times [7(\text{CaO}) + 7(\text{MgO}) + 5(\text{FeO}) + 4(\text{MnO})]/[(\text{Al}_2\text{O}_3) + 2(\text{SiO}_2) + (\text{Fe}_2\text{O}_3) + 5(\text{P}_2\text{O}_5)]$ , and at 1500°,  $K = 1.8 \times [7(\text{CaO}) + 4(\text{MgO}) + 4(\text{FeO}) + 3(\text{MnO})]/[(\text{Al}_2\text{O}_3) + 2(\text{SiO}_2) + 0.5(\text{Fe}_2\text{O}_3) + 2.5(\text{P}_2\text{O}_5) + (\text{TiO}_2)]$ .

A. R. POWELL.

**Decomposition of refractory silicates by fused ammonium fluoride and its application to the determination of silica in glass sands.** A. C. SHEAD and G. F. SMITH (J. Amer. Chem. Soc., 1931, 53, 483—486).—Silica is rapidly removed from finely-powdered quartz and silicates by the action of molten ammonium fluoride. The silica in 1 g. of powdered glass sand is determined by two successive fusions with 5-g. portions of ammonium fluoride.

J. G. A. GRIFFITHS.

**Determination of zinc in refractory bodies and slags.** E. E. MARBAKER (J. Amer. Ceram. Soc., 1931, 14, 162—175).—Comparative results are given for the separation of zinc from iron and aluminium by the basic acetate and by the ammonia-ammonium chloride methods, but the separation of iron and zinc with hydrogen sulphide in an ammoniacal solution containing citric acid is recommended as a rapid method, full details for which are given.

J. A. SUGDEN.

**Classical methods of analysis of silico-aluminous, calcareous, alkaline materials.** (Rocks, bricks, bauxites, slags, glasses, siliceous minerals.) J. DELFORGE (Bull. Soc. chim. Belg., 1930, 39, 539—550).—The difficulties encountered in the analysis of these materials after attack by alkali carbonates or by hydrofluoric acid are discussed. The hydrofluoric acid

method is preferred and details are given of an improved technique for this purpose. Comparative analyses obtained by the two methods are given. J. W. SMITH.

See A., April, 445, Kaolin. Decomposition of carborundum. 458, Silvered mirrors.

#### PATENTS.

**Working of glass and like substances.** INTERNAT. DE COMP. GOBELTERIE INÉBRÉCHABLE, Assees. of J. VIATOUR (B.P. 343,030, 11.11.29. Belg., 13.11.28).—A hot flame good for quick melting, *e.g.*, for fire-finishing or cutting off glass ware, is obtained by burning pure acetylene with air or oxygen under suitable pressure, *e.g.*, 10–15 cm. of water for acetylene, or 1–2 kg. for oxygen, in a suitable burner, which is described. The flame should be slightly oxidising so that no free carbon is deposited on a test piece of platinum foil held therein.

M. PARKIN.

**Silica crucible.** W. H. JONES, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,769,841, 1.7.30. Appl., 8.6.29).—The crucible is made by first forming the tubular wall and then attaching to the narrow end (the bottom) a lattice work of silica rods. These may be fixed to a retaining ring, which is then fused into the bottom of the crucible.

F. SALT.

**Manufacture of glass [transparent to ultra-violet rays].** J. HOCHHUT (B.P. 342,936, 13.3.30).—Stannous chloride (up to about 1%) is added to the raw materials for the manufacture of glass with a low iron content; cobalt oxide may also be added if glass capable of absorbing visible light is required.

L. A. COLES.

**Manufacture of compound glass.** BRIT. CYANIDES Co., LTD., and E. C. ROSSITER (B.P. 344,047, 29.11.29).—The adhesive comprises a mixture of about 70% of gelatin with 30% of a synthetic resin, *e.g.*, a urea- or thiourea-formaldehyde resin (cf. B.P. 266,028; B., 1927, 305) or a dicyanodiamide-formaldehyde resin (cf. B.P. 314,358; B., 1929, 728), adhesion after assemblage being effected by compression at about 80°.

L. A. COLES.

**Manufacture of reflecting surfaces [for glass].** GLASFABRIKEN U. RAFFINIEREN J. INWALD A.-G., and O. INWALD-WALDTREU (B.P. 343,309, 11.2.30).—Instead of the usual electrodeposited copper backing of the silvered surface, nickel is plated from a bath containing nickel salts, conducting salts, and an agent to control the acidity. A suitable bath contains, per litre of water, nickel sulphate crystals 211–260 g., nickel ammonium sulphate 17–20 g., sodium sulphate 110–150 g., glacial acetic acid 30–50 g., and cadmium chloride 0.1–0.3 g. The hydrogen content of the deposit should be 0.0009–0.01% to prevent peeling. With such deposits the mirror surface is brighter than with copper and withstands a higher temperature so that the varnish backing can be burned on better.

M. PARKIN.

**Clay compound.** T. S. CURTIS (U.S.P. 1,768,545, 1.7.30. Appl., 5.9.25).—Raw pyrophyllite is heated under reducing conditions to about 980°, then cooled quickly to prevent reoxidation, and crushed to a finely-divided state, the impurities such as lime, magnesia,

iron and titanium oxides being removed from the crushed material. The addition of 50% of this material increases the fired strength of English ball clay about 140 times.

F. SALT.

**Obtention of kaolin from preredefined suspensions by fractional precipitation.** E. MÜLLER (B.P. 340,142, 4.1.30).—Kaolin which has already been separated from the raw material, *e.g.*, by fractional precipitation, is formed into a suspension having  $d$  1.06–1.12 and subjected to further fractional separation by setting it aside for about 12 hrs., the height ( $h$ ) of the suspension in the vessel being given by the formula:  $h = 3.2/(d-1)$  cm. The suspended material is then poured off and separated by filter-pressing.

F. SALT.

**[Continuous] preparation of chamotte.** FELLNER & ZIEGLER A.-G. (B.P. 343,837, 7.5.30. Ger., 28.8.29).—Crushed or broken clay is burned at 1100–1300° in rotary furnaces which, in the case of very damp or bituminous materials, should be expanded in the temperature zone 800–1000° before the clay is dissociated. Economy of fuel is claimed.

H. ROYAL-DAWSON.

**Furnace lining composition.** T. H. GRAY (B.P. 340,141, 1.1.30).—A mixture for making or repairing furnace linings contains used foundry sand which, after being screened and damped, is made into a paste with fireclay, in the proportions of 3 pts. by vol. of sand to 2 pts. of clay.

F. SALT.

**Composite refractory brick.** J. D. MORGAN, Assr. to DOHERTY RES. Co. (U.S.P. 1,769,708, 1.7.30. Appl., 10.10.27).—Bricks consisting of a main body portion of heat-insulating material and a surface layer of highly refractory composition attached to one end are built up so that the refractory layer forms the inside wall of the furnace chamber. The main body of the bricks may be of diatomaceous earth, puffed clay, cellular zircon, zirconia, or clay, and the refractory layer of zircon (native zirconium silicate) or of electric-furnace refractories. The bricks may be fired in two parts (and the parts cemented together) or in one piece.

F. SALT.

**Refractory mixture.** J. M. LAMBIE and D. W. ROSS (U.S.P. 1,769,297, 1.7.30. Appl., 30.10.26).—The firing shrinkage of refractory mixtures is counteracted, over a wide temperature range, by the addition of aluminous materials such as cyanite, natural sillimanite, dumortierite, and corundum, the aluminous material added to the clay and grog varying from 5% to 60% according to conditions. The process is particularly suitable to the manufacture of glass tank blocks.

F. SALT.

**Production of refractory materials.** T. R. HAGLUND (B.P. 343,896, 22.11.29. Addn. to B.P. 302,087; B., 1929, 250).—In the molten mixture of magnesium, aluminium, chromium, and silicon oxides the percentage of alumina is such that the tripled sum of the oxides of silicon and iron, or this sum plus the excess of alumina over chromium oxide, if there be an excess, does not exceed 60. Preferably the rule of not exceeding 60 remains satisfied when the excess of alumina over half the chromium oxide is computed.

C. A. KING.

**Glass furnace.**—See XI.



## IX.—BUILDING MATERIALS.

**Production of Portland cement. III. Action of sulphur and its compounds.** A. J. BLANK (Concrete, 1930, 37, No. 5, 89—90; Chem. Zentr., 1931, i, 339—340).—A study of the sulphur distribution in the furnace system was made. The greater part of the sulphur in the clinker was derived from the furnace gases. More sulphur is retained in an oxidising than in a reducing process. A. A. ELDRIDGE.

**Thermochemistry of cement.** W. A. ROTH (Zement, 1930, 19, 628—630; Chem. Zentr., 1930, ii, 1116).—Indirect methods only for its investigation are available. The heat of setting ("Abbindungswärme"), determined by dissolution of the cement powder and of the set product in 100 pts. of 20% hydrochloric acid and 15 pts. of 20% hydrofluoric acid, may be a measure of the quality of cement. Calorimetric and bomb methods applicable to cement are also described. L. S. THEOBALD.

**Cement bacillus and its occurrence in the destruction of concrete by sulphate solutions.** A. GUTTMANN and F. GILLE (Tonind.-Ztg., 1930, 54, 759—762; Chem. Zentr., 1930, ii, 1116).—The calcium sulphoaluminate,  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$ , formed in the destruction of concrete by sulphate is identical with ettringite. A compound  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot \text{H}_2\text{O}$  is sometimes formed. The latter is unstable in acid, magnesium salt, or carbonate solutions, or above  $40^\circ$ . A. A. ELDRIDGE.

**Analysis of silico-aluminous materials.**—See VIII. **Painting of cement. Painting and glueing of wood.**—See XIII. **Glues.**—See XV.

See also A., April, 437, **Hydrates of calcium sulphate.**

## PATENTS.

**Composition for use with concrete, cement, etc.** F. GUY (B.P. 344,025, 28.11.29).—A proofing composition for use with cement mixtures contains approx. 4 pts. of caustic soda, 26 pts. of oleic acid, 224 pts. of sand, and 56—112 pts. of potash alum. A soap is first made to which the mixture of sand and alum is added. C. A. KING.

**Treatment of slag [for building blocks or bricks].** NORSKE A./S. FOR ELEKTROKEM. IND., Assees. of T. BARTHOLEMEW (B.P. 344,309, 4.6.30. U.S., 4.6.29).—Molten slag supersaturated with gas is agitated to release the gas and then formed into blocks. Slag, which may be supersaturated artificially, may also be caused to foam during release of the gas and thus form a cellular brick on casting, or intermediate degrees of density may be obtained. [Stat. ref.] C. A. KING.

**Weatherproofing and colouring stone, wood, etc.** B. REDLICH (B.P. 343,891, 20.11.29).—Building materials are coated or impregnated with a colloidal aqueous solution of wax, paraffin, ceresin, or other wax-like substances containing a small quantity of emulsifying agent. When dry the coating will not swell or dissolve in water. Suitable colouring pigments may be incorporated. C. A. KING.

**Treatment of limestones to give them the appearance of marble.** J. GIOVANNINI (B.P. 342,914,

17.2.30. Belg., 15.2.29).—Articles shaped out of limestone are heated at  $250^\circ$  in a kiln for about 1 hr., and are then steeped for about 24 hrs. in a bath containing 3.4 kg. of iron sulphate and 2.5 kg. of magnesium carbonate per 100 litres of water, after which they are dried and polished. L. A. COLES.

**Imitation ice surfaces for skating and similar purposes.** G. LEXTON and T. C. EARNSHAW (B.P. 342,866, 11.1.30).—A fused mixture of washing soda crystals and Glauber's salt is poured over reticulated material. L. A. COLES.

**Bituminous dispersions.**—See II.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Reaction velocity and equilibrium conditions in steel smelting.** F. SANDELIN (Jernkontorets Ann., 1930, 113, 519—544; Chem. Zentr., 1930, ii, 1122).—Previous theories are discussed. The oxidation of silicon and manganese in acid open-hearth melts containing moderate amounts of these elements follows mainly the equation for a unimolecular reaction when the other variables are kept constant. The concentration of ferrous oxide in steel remains constant. Steel may take up carbon from the furnace gases in the prerefining period. L. S. THEOBALD.

**Reheating of tempered high-speed steels.** A. MICHEL and P. BENAZET (Compt. rend., 1931, 192, 163—166).—Rods, 7 mm. in diam., of a high-speed steel (C 0.72, Si 0.33, Mn 0.26, Cr 5.20, W 18.83, V 1.02, Co 11.09%), oil-tempered at  $1325$ — $1350^\circ$ , and machined thereafter, were reheated for varying periods at  $600^\circ$ , and dilatation and hardness determined. Austenitic conditions predominated throughout the reheating and subsequent cooling to about  $200^\circ$ , when marked dilatation indicated transformation into martensite, accompanied by increased hardness on complete cooling. A single reheat for 4 hrs. at  $600^\circ$  did not effect complete transformation, but two successive reheats for 20 min. each did so. Reheats to a higher or lower temperature effect the transformation more or less rapidly: thus seven reheats to  $500$ — $520^\circ$  are required, the transformation then occurring below  $100^\circ$ . Prolonged reheating (e.g., for 24 hrs. at  $600^\circ$ ) causes the transformation partly to take the form  $\gamma \rightarrow \alpha + \text{carbides}$ , giving a product of lower hardness. Reheating to just below the Ac point has the same effect. Similar results have been obtained with high-speed steels containing from 0 to 16% of cobalt. C. A. SILBERRAD.

**Effect of small quantities of arsenic on the properties of mild steel.** O. BAUER (Mitt. Materialprüf., 1931, 131—132).—Arsenic, within the usual limits of 0.02—0.05% in which it occurs in mild steel, is without effect on the dynamic or static properties of the metal. With slightly higher amounts, e.g., 0.11%, it reduces the impact strength to a small extent, especially when the sulphur or phosphorus content is relatively high. A. R. POWELL.

**Soft steel deposited in the electric arc.** D. ROSENTHAL and M. MATHIEU (Compt. rend., 1930, 191, 484—486).—The mechanical properties of such deposits

depend to a great degree on the extent to which the metal is protected from atmospheric oxidation. Thus, *e.g.*, the Charpy resilience value rises from 0.81 to 1.0 for an unprotected soft-steel wire electrode to 5.85–7.60 kg./cm.<sup>2</sup> for a protected electrode. Metallographical methods show that in the latter case the grains are oriented more regularly, whilst X-ray diagrams (molybdenum K-radiation) indicate the presence of internal stresses in the unprotected, but not in the protected, deposit.

J. GRANT.

**Steels for aircraft engine valve springs.** A. SWAN, H. SUTTON, and W. D. DOUGLAS (Inst. Mech. Eng., Feb., 1931. Advance copy. 39 pp.).—Most of the springs failing by reason of surging and fatigue showed pronounced decarburisation at the surface, and the removal of the surface layer increased the apparent safe range of stress. The best endurance properties were attained by quenching in oil from temperatures relatively low but sufficiently high to secure effective hardening, followed by normal tempering. High quenching temperatures resulted in the formation of longitudinal cracks not always noticeable before testing.

C. A. KING.

**Oxidation of copper in refining in the reverberatory furnace, and significance of the oxide test.** P. SIEBE (Metall. u. Erz, 1930, 27, 549–557; Chem. Zentr., 1931, i, 350).—A high state of purity is characterised microscopically by long dendrites in the eutectic mass; short dendrites and round nuclear inclusions of cuprous oxide indicate the presence of impurities. The dendrites are shortened by the presence of tin, arsenic, antimony, and lead. Poling is indicated by the appearance of annular deposits of cuprous oxide.

A. A. ELDRIDGE.

**Effects of impurities on copper.** VII. Effect of antimony on copper. VIII. Combined effect of antimony and arsenic on copper. S. L. ARCHBUTT and W. E. PRYTHERCH (Inst. Metals, Mar., 1931. Advance copy. 33 pp.; cf. B., 1930, 422).—The alloys studied are antimonious copper (less than 0.85% Sb) and antimony–arsenic–copper alloys (up to 0.5% each Sb + As); other impurities have been kept as low as practicable—below 0.02% in the case of oxygen. Billets 1½ in. in diam. have been cast, mainly from melts made *in vacuo* (<0.5 mm. Hg) in the high-frequency induction furnace and have been rolled according to schedules employed previously (*loc. cit.*). The methods of casting and rolling have been examined and determinations made of the density, hardness, tensile properties at room temperature and 250°, fatigue and impact values, electrical conductivity, softening temperatures of cold-worked material, and microstructure. In general, the properties of copper are markedly improved by additions of antimony, especially in the presence of arsenic. The electrical conductivity is reduced by antimony, but to a less pronounced extent than by arsenic. The solubility of antimony in copper, redetermined over the range 620–400°, was found to be 9–10%. Neither antimony nor arsenic appears to effect any change in the macrostructure of copper. Although all the alloys considered were within the solid-solubility range, a second constituent was invariably detected in the microstructure, occurring as small globules in the

worked and annealed materials. This is thought to be a complex of copper oxide with antimony (and arsenic, when present). No micro-constituent arising out of reactions between antimony and arsenic was found in any of the alloys examined.

E. H. BUCKNALL.

**Rolling of alloys of copper and phosphorus containing up to 5% P.** O. W. ELLIS (Inst. Metals, Mar., 1931. Advance copy. 6 pp.).—The copper–phosphorus alloys may be hot-rolled at 450–650° from chill-cast billets without difficulty, if light passes are given. By such treatment the copper–copper phosphide eutectic is broken up and the material may be subjected to considerable reduction by cold-work without the appearance of cracks. Suitable rolling schedules are described. Strip (0.018 in. thick, 1.2 in. wide) containing 5.05% P had a tensile strength of approx. 40 tons/in.<sup>2</sup> This alloy is suitable for use as a high-melting solder for electrical work. Additions of manganese or nickel render the alloys brittle in the hot-rolling operation.

E. H. BUCKNALL.

**Cold-working and annealing of  $\alpha$ -brasses.** J. N. ALCÁZER (Anal. Fís. Quím., 1930, 28, 1420–1434).—The changes of crystalline structure produced in brasses (68 and 65.1% Cu) by cold-working and by annealing at a series of temperatures between 250° and 700° have been investigated by means of Laue diagrams and photomicrographs, and after annealing at temperatures up to 1000°, by hardness tests. The X-ray diagrams show that the fibrous structure commences to break down even at 250° and that the transformation to a coarsely crystalline condition proceeds to an increasing extent at temperatures up to 700°. The hardness (Brinell) of these alloys increases on hammering and becomes approximately constant when the cross-sectional area has been reduced to about 60%; the hardness–annealing temperature curves of the hammered brasses exhibit peaks at 250° and 400°. Correlation of these results with Erichsen tests shows that the annealing process takes place in four stages: (1) up to 250° the structure changes slightly and the deformation diminishes; (2) at 250–500° the greater part of the fibrous structure disappears and the deformation increases very considerably; (3) at 500–700° the coarsely crystalline non-oriented structure appears; (4) at higher temperatures the crystals increase in size and the deformation increases to a maximum at about 900°. The recommended annealing temperature for the  $\alpha$ -brasses is 800–900°.

H. F. GILLBE.

**Some properties of silicon–aluminium bronzes.** L. J. BRICE (Inst. Metals, Mar., 1931. Advance copy. 18 pp.).—The mechanical properties and microstructure of three typical aluminium–copper alloys containing, respectively, 5, 7.25, and 10% Al, with the addition to each of up to 5% of silicon, have been examined in the chill-cast condition, and also after subjection to mechanical and heat treatments. With 5% Al the addition of up to 2% of silicon improves the general properties; further addition of silicon gives progressively increased hardness, but reduces toughness and ductility, although considerable improvement in these properties may be effected by heat-treatment. With 7% Al additions of silicon up to 4% increase the yield point and maximum stress and hardness, but

reduce ductility and toughness. With more than 3% Si ductility and toughness are so low in all conditions of treatment that the alloys are of little practical use. With the 10% aluminium alloys, 1% Si is the maximum content with which the alloys are at all ductile. The properties of some of the more promising alloys are tabulated, and photomicrographs of a number of the alloys are reproduced. E. H. BUCKNALL.

**Effect of artificial ageing on the resistance of super-duralumin to corrosion by sea-water.** K. L. MEISSNER (Inst. Metals, Mar., 1931. Advance copy. 15 pp.).—Strips of super-duralumin (duralumin containing 0.8% Si) were annealed for 20 min. at 500°, quenched in cold water, aged for 5 days at room temperature, and tempered for 20–40 hrs. at temperatures ranging from 50° to 200°. The strips were subsequently exposed to intermittent corrosion by sea-water between high- and low-water marks for 3 months. The degree of corrosion was then determined by loss in weight and reduction in tensile properties of the strips. Microscopical examination of the samples showed that inter-crystalline corrosion had occurred in all cases, but strips tempered at 125–145° (especially at 140°) were notably subject to this form of attack. In these strips severe pitting also was observed. Strips aged above or below these temperatures retained a comparatively smooth surface, free from pitting. E. H. BUCKNALL.

**Effect of temperature and the cooling conditions in the casting of zinc.** O. BAUER and P. ZUNKER (Mitt. Materialprüf., 1931, 134).—Charges of 7 kg. of zinc containing 1.1% Pb were cast at various temperatures in steel moulds having walls 0.5, 2, or 40 mm. thick, and the resulting crystal structures examined and compared with those obtained in a clay mould with walls 40 mm. thick. With increasing casting temperature ingots cast in steel moulds showed progressively coarser crystals and deeper and broader pipes. Castings made in preheated clay moulds had an extraordinarily large crystal structure and a very deep pipe. The crystals of ingots cast in steel moulds were all perpendicular to the walls, whereas those of ingots cast in clay moulds were directed towards the apex of the pipe. Ingots made by casting zinc at 420° into thin-walled steel moulds cooled in water had a very fine structure of small, irregularly oriented crystals and only a small flat pipe. Rapid casting increased the pipe, but decreased the grain size. A. R. POWELL.

**Resistance of metals and alloys to attack by salt solutions.** I. E. MAASS and W. WIEDERHOLT (Korrosion u. Metallschutz, 1930, 6, 218–223; Chem. Zentr., 1931, i, 146).—Comparative experiments with sylvine, carnallite, and sodium sulphate solutions and various metals are recorded. A. A. ELDRIDGE.

**Effect of temperature on the solubility of nickel, tin, copper, chromium steel, and zinc in raw cow's milk.** G. N. QUAM (Proc. Iowa Acad. Sci., 1929, 36, 261–262).—The solubility curves for nickel, copper, and zinc show a maximum within the range 75–85°. With tin a corrosion product accumulated above 75°; chromium steel was unchanged.

CHEMICAL ABSTRACTS.

**Corrosion of metals by phosphoric acid.** P. R. KOSTING and C. HEINS, JUN. (Ind. Eng. Chem., 1931, 23, 140–150).—Accelerated corrosion tests on 52 metals and alloys, using 10–85% phosphoric acid, both pure and commercial, indicated that aluminium, nickel, tin, and zinc possessed high corrosive rates. Chromium was resistant to pure acid and might be used for plating apparatus to be used for cool dilute acid. The resistance of copper was destroyed to a great extent by aeration of the acid, as was also that of lead, with which, however, sulphuric acid acted as an inhibitor. Iron and alloys of iron and carbon dissolved rapidly, but excellent resistance was offered by both silver and tantalum. Very few non-ferrous alloys exhibited low corrosion rates in either pure or crude phosphoric acid, which latter contains some inhibitor of corrosive effect on iron. Pine oil, mucilage, pyridine, arsenic, and possibly other types of organic materials serve as inhibitors, whilst sulphuric, hydrofluoric, and fluosilicic acids increase corrosion. Rise in temperature and increase in concentration generally tend to more rapid corrosion. It was indicated that 18% Cr should be present in stainless irons to resist pure acid, preferably with 8%, but not more, of nickel. A corrosion rate of  $1 \times 10^{-5}$  cm. per day may be regarded as satisfactory. C. A. KING.

**Active platinum catalyst.** E. V. ALEXEEVSKI and I. D. MAKAROV (J. Appl. Chem., Russia, 1930, 3, 857–858).—Kaolin-clay-charcoal rings are ignited at 950° and soaked in chloroplatinic acid solution until they absorb 8% of platinum; they are dried at 100° and reduced at 120–150° with electrolytic hydrogen containing formaldehyde. CHEMICAL ABSTRACTS.

**Active palladium catalyst.** E. V. ALEXEEVSKI, A. P. MUSAKIN, and I. D. MAKAROV (J. Appl. Chem., Russia, 1930, 3, 863–866).—Porous asbestos cloth soaked for 12–15 hrs. in a solution of palladous chloride in 2*N*-hydrochloric acid is dried at 100° and reduced during 12 hrs. in an electric furnace by hydrogen saturated with moist formaldehyde, and then cooled in hydrogen. Subsequent washing with hot and then cold (12 hrs.) distilled water is important. The optimal concentration of palladium is 8%, but 2% is the economic limit. CHEMICAL ABSTRACTS.

**Germanium. XXXVI. Extraction of germanium and gallium from germanite.** I. W. I. PATNODE and R. W. WORK (Ind. Eng. Chem., 1931, 23, 204–207).—Air-dried germanite is chlorinated at 340–360° in an iron still and the distillate collected. Crystals of ferric chloride collecting on the cooler parts of the still are saved also for the later extraction of its gallium content. The distillate consists essentially of a mixture of germanium tetrachloride (b.p. 83°), silicon tetrachloride, arsenic trichloride, and chlorides of sulphur. By fractional distillation to 120° a ruby-red liquid, *d* 1.75, is obtained which contains almost all the germanium chloride. This is hydrolysed by pouring on to a relatively large quantity of ice, thus forming a heavy light-yellow suspension containing also silica, arsenic oxide, and hydrochloric acid. When distilled in chlorine germanium tetrachloride commences to volatilise at about 68° and the temperature rises to 104–107°, after which the chloride is again hydrolysed on ice to a white

hydrated dioxide, which is filtered and washed. The oxide is spectroscopically pure and represents 85–90% of the total germanium in the ore. Details of the process for extraction of the gallium from the solid residues from chlorination are to follow in a later paper.

C. A. KING.

**Comparative measurement of throwing power in electroplating practice.** L. K. WRIGHT (Metal Ind., N.Y., 1930, 28, 522–523).—Iron or brass tubes are plated for 2–20 min., and the depth of the “throw” is measured by means of a throw-scale calibrated in 0.05 in.

CHEMICAL ABSTRACTS.

**Electrolytic refining of silver.** F. VOGEL (Metallbörse, 1930, 20, 1209–1210, 1265–1266; Chem. Zentr., 1930, ii, 1124).—A discussion of the development of processes.

A. A. ELDRIDGE.

**Electrodeposition of silver-cadmium alloys.** C. G. FINK and B. G. GERAPOSTOULOU (Metal Ind., N.Y., 1930, 28, 519–521, 562–563).—An increased Cd:Ag ratio in the complex cyanide bath causes an increased ratio in the deposit. High current density increases the proportion of cadmium; rise of temperature or agitation causes a decrease in the cadmium content of the deposit. The deposit contains silver-rich (or pure silver) and cadmium-rich solid solutions. Silver-cadmium alloys of high silver content do not resist tarnish by hydrogen sulphide better than pure silver.

CHEMICAL ABSTRACTS.

**Cadmium as a rust preventive.** B. PLANNER (Z. Elektrochem., 1931, 37, 33–49).—In the electrodeposition of cadmium as a rust preventive from a cadmium potassium cyanide solution, the best results are obtained with a current density of 50–150 amp./m.<sup>2</sup> and a bath temperature of 40–60°. Under these conditions a satisfactory deposit is obtained with or without addition of colloids and the current yield may be upwards of 95%. If an acid bath is employed, the current yield normally increases with increase in the temperature and current density, but at the same time the deposit becomes more and more coarse in grain. Addition of substances with surface activity and colloids and increase in the mol. wt. of the anion of the electrolyte, however, tend to render the deposit fine-grained, and by using such an electrolyte as cadmium phenolsulphonate with suitable additions, a current density of 100–150 amp./m.<sup>2</sup>, and a temperature of about 40°, a high current yield and an excellent deposit can be obtained. Tests of porosity and resistance to corrosion by salt water show that cadmium exerts a far greater protective action than either zinc or nickel, a film 0.004 mm. in thickness affording adequate protection to a smooth surface.

R. CUTHILL.

**Influence of other metals on nickel-plate.** B. ŠETLIK (Chem. Listy, 1931, 25, 123–124).—Satisfactory nickel-plating cannot be performed in the presence of 0.25% Cu, 0.2% Zn, or 0.6% Fe in the electrolyte. Copper may be separated electrolytically, whilst iron and zinc must be separated by chemical methods.

R. TRUSZKOWSKI.

**Refractories. Zinc in slags.**—See VIII. **Lead for accumulator grids.**—See XI. **Corrosion by fruit.**—See XIX. **Photography on copper.**—See XXI.

See also A., April, 414, Alloy AuCu<sub>3</sub>. 416, Hardening of metals. 418, Copper-arsenic alloys. System Fe–C–Si. System Li–Ag. System Cu–Sn. Binary alloys of the transition elements with zinc, cadmium, and aluminium. 423–4, Colloidal silver. 424, Colloidal lead. 429, Colloidal gold. 432, Anodic behaviour of copper. 433, Electrolysis of alloys. 435, Magnesium. 441, Deposition of reactive metals on noble metals. 447, Sintered tungsten rods. 452, Determination of small quantities of silver. 453, Determination of zinc in lead, and of cadmium. 454, Determination of copper and cobalt. 455, Determination of nickel. 457, Furnace for metallographic examination.

#### PATENTS.

**Regenerators of Siemens–Martin furnaces.** F. FIORELLI (B.P. 343,871, 22.1.30. Addn. to B.P. 341,721; B., 1931, 398).—The vertical subdivision of the chequerwork of each regenerator is effected by means of a single partition wall.

H. ROYAL-DAWSON.

**Introduction of waste gases from blast furnaces, and other gases produced in the vicinity thereof, into blast furnaces.** F. W. DRÜCKER (B.P. 343,055, 8.11.29).—The waste and other gases are drawn from a container by the injector action of an air blast and are introduced directly into the smelting zone of a blast furnace. The air blast is preheated solely by contact with the gases.

C. A. KING.

**Apparatus for complete roasting of highly sulphidic ores.** METALLGES. A.-G., and H. KLENCKE (B.P. 343,047, 7.10.29).—The usual width of a Dwight-Lloyd roasting chamber is reduced to about 60 cm., or even to 30 cm., so that the heat generated from a rich sulphide ore is dissipated rapidly without sintering the charge. Auxiliary cooling means may be incorporated.

C. A. KING.

**Arrangement for annealing metal articles.** A.-G. BROWN, BOVERI & Co. (B.P. 343,262, 7.1.30. Ger., 7.1.29).—Annealing pots are caused to travel on trucks through a longitudinal chamber to an external furnace from which they are cooled by passing in opposite direction through a similar adjacent chamber. A current of air enters at the material discharge opening, cools the hot pots, and then heats the pots advancing to the furnace, finally leaving at the material charging end.

C. A. KING.

**Manufacture of wrought iron.** A. M. BYERS Co., Assees. of F. WILLE (B.P. 344,277, 28.4.30. U.S., 28.1.30).—In the Ashton process of manufacture, molten steel is poured from a ladle in such a way that the point on the surface of the slag bath at which the metal enters is continually changed, preferably through a predetermined path, which is accomplished by automatic plant.

C. A. KING.

**Production of spongy iron.** F. KRUPP A.-G., FRIEDRICH-ALFRED-HÜTTE (B.P. 342,659, 31.10.29. Ger., 6.11.28. Addn. to B.P. 340,641; B., 1931, 255).—Gas from the blast furnace is reintroduced into the furnace below the place where it is taken off, in order to be carburetted and heated.

H. ROYAL-DAWSON.

**Acid-proof [iron] alloys.** SKODA WORKS (B.P. 343,003, 5.9.29. Czechoslov., 31.10.28).—Iron-silicon-titanium alloys containing up to 25% (2–4%) Ti and up to 20% (16%) Si are claimed. Such alloys should contain 1–20 pts. by wt. of titanium per 8 pts. of silicon.

H. ROYAL-DAWSON.

**Production of a welding material suitable for use as welding wire.** W. ALBERTS (B.P. 343,414, 27.6.30. Ger., 26.8.29).—A more homogeneous mixture of ingot iron and slag is made by adding liquid slag at the time of tapping or casting the molten iron, which should contain as maximum 0.05% C. A slag containing 30–50% FeO is preferred.

C. A. KING.

**Condensation of zinc vapour.** NEW JERSEY ZINC Co., Assees. of E. H. BUNCE (B.P. 344,059, 2.12.29. U.S., 16.10.29).—Gases containing zinc vapour are conveyed through baffled cooling chambers in which the zinc vapour is cooled to liquid metal which is allowed to flow by a direct route to a sump at the exit of the chamber. The required heat dissipation is controlled by the degree of insulation of the chamber.

C. A. KING.

**Manufacture of tubes of highly refractory material [tungsten].** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 342,648, 26.9.29).—Tungsten,  $\delta$  19.0 and thickness 500  $\mu$ , is applied on to a molybdenum core by electrolysis at 950° in a sodium sulphate bath containing a 10% surplus of tungsten trioxide or by heating at 1700° in tungsten hexachloride vapour, and the core is removed by dissolution in a mixture comprising 10 vols. of nitric acid, 6 vols. of sulphuric acid, and 4 vols. of water.

L. A. COLES.

**Manufacture of aluminium by electrolysis [of alumina].** COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES, ET CARMARGUE (B.P. 344,292, 16.5.30. Fr., 21.5.29).—In the preparation of the cell baths for the process the cryolite which serves as a flux is wholly or partly replaced by a mixture of dry or moist aluminium fluoride with sodium or potassium chloride.

H. ROYAL-DAWSON.

**Casting of molten metals or alloys [of relatively low m.p., e.g., of lead].** H. HARRIS (B.P. 345,409, 29.1.30).

**Tempering metals.**—See XI. **Coating surfaces with rubber.**—See XIV.

## XI.—ELECTROTECHNICS.

**Use of antimonial lead for accumulator grids: a cause of self-discharge of the negative plates.** J. T. CRENNELL and A. G. MILLIGAN (Trans. Faraday Soc., 1931, 27, 103–112).—The "ageing" that occurs in the negative plates of lead accumulators, i.e., the increased tendency to self-discharge and sulphation as the cell becomes older, has been investigated. The evolution of hydrogen from the plate is used as a measure of the self-discharge and of the efficiency of the plate. Comparison of cells made up of lead plates with and without antimony shows that the ageing of the negative plate in the usual type of accumulator is due to dissolution of antimony from the alloy of the positive grid and its deposition on the negative plate. The low hydrogen overvoltage at an antimony surface—about 0.1 volt

lower than at a lead surface—allows local action to occur, which discharges the lead plate. In the absence of a source of antimony the rate of sulphation or of self-discharge of the negative plate is negligibly small at all stages of its life, and antimonial lead should, therefore, not be used for the grids of lead accumulators. Although some of the antimony can be removed from the negative plate by reverse charging, the improvement in the performance of the cell is only partial and temporary, and this procedure is of no practical value.

O. J. WALKER.

**Self-discharge of dry cells.** S. MAKINO (J. Soc. Chem. Ind., Japan, 1930, 33, 521 B).—Experiments on the prevention of self-discharge were made by immersing (1) only the positive electrode in an electrolyte, and (2) the same electrode as an anode in an electrolyte with a usual negative electrode; a Japanese and a Javanese manganese dioxide were used as anodes. The decrease of *E.M.F.* and capacity on open circuit was found to occur first at the anode, and then gradually at the cathode, to a degree depending on the amount of iron present as impurity in the manganese dioxide.

E. LEWKOWITSCH.

**Insulating oils.**—See II. **Soya-bean oxidation.**—See III. **Insulating paper.**—See V. **Chamber sulphuric acid.** **Electrolytic cells.**—See VII. **Insulation of pyrex glass.** **Dewatering of clay.** **Refractories.**—See VIII. **Soft steel.** **Copper-phosphorus alloys.** **Electroplating practice.** **Silver.** **Silver-cadmium.** **Cadmium.** **Nickel.**—See X.

See also A., April, 409, **Copper oxide rectifiers.** 424, **Colloidal lead.** 432, **Anodic behaviour of copper.** 433, **Electrolysis of alloys.** **Conductivity of electrolytes for cells.** 435, **Magnesium.** 440, **Synthesis of ammonia.** **Reduction of arsenic acid and arsenates.** 441, **Formation of ethylene and acetylene from methane.** **Ionisation in insulating materials.** 449, **Radioactivity method of examination of powders.**

## PATENTS.

**Induction furnace.** F. LINNHOFF, ASSR. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,775,351, 9.9.30. Appl., 7.12.28. Ger., 2.3.28).—A coreless induction furnace comprises a crucible the upper part of which is of greater diameter than the lower, induction coils surrounding the respective parts of the crucible and separated by laminated iron rings, a three-phase source of current, and a Scott connexion, whereby a two-phase Scott circuit may be connected to the coils.

J. S. G. THOMAS.

**Electric furnaces [for tempering metals].** BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 343,529, 19.9.29).—A relatively small air-delivery orifice and an air-extraction orifice are arranged respectively above and below the treatment zone of the furnace, and electrical resistances are arranged in a chamber placed within the heat-insulating walls of the furnace, in circuit with these orifices, and to one side of the treatment chamber. The circuit includes a fan for circulating air in the treatment chamber.

J. S. G. THOMAS.

**[Glass-melting] electric furnaces and electrodes therefor.** GEN. ELECTRIC Co. LTD., R. F. PROCTOR, and

H. C. BIGGS (B.P. 343,563, 21.11.29).—Conducting blocks of graphite-fireclay mixture of thickness substantially equal to that of the furnace wall and protected by shields from oxidation, are used as electrodes in an electric furnace in which molten glass forms the resistance.

J. S. G. THOMAS.

**[Cored carbon] electrodes for arc lamps.** W. H. HARDMAN and F. GIRVIN, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,775,078, 2.9.30. Appl., 6.7.25).—The cores consist of a mixture of boric oxide 1%, cerium oxide 56%, carbon 37%, and tungsten carbide 6%, with coal tar as binder.

J. S. G. THOMAS.

**[Cored carbon] electrodes for the electric arc.** E. K. DEWEY, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,774,964—5, 2.9.30. Appl., [A, B] 6.7.25. Renewed [A] 1.7.30).—The cores consist of (A) cerium oxide 72%, titanium carbide 8%, boric oxide 1%, and carbon 19%; and (B) cerium oxide 27%, cerium fluoride 18%, tungsten carbide 5%, boric oxide 1%, and carbon 49%. In each case tar is used as binder.

J. S. G. THOMAS.

**Magnetic cores for use in loading coils and the like.** AUTOMATIC TELEPHONE MANUFACTURING CO., LTD., and P. N. ROSEBY (B.P. 343,620, 30.11.29).—Finely-powdered nickel-iron alloy is mixed with about 6% of calcined magnesia added in two stages, between which the mixture is treated with an insulating ("Bakelite") varnish and compressed.

J. S. G. THOMAS.

**Electrodes for electric accumulators.** A. G. OWENS (B.P. 343,048, 9.10.29).—The accumulators comprise a negative lead electrode filled with active material (litharge paste) mixed with sugar and ammonium sulphate, and a positive lead electrode filled with active material (lead peroxide), sugar, ammonium sulphate, and finely-divided glass wool; the electrodes are wrapped in layers of spun silica threads, which retain the electrolyte in contact with the active materials.

J. S. G. THOMAS.

**[Filling for] electric-discharge [rectifying] tubes.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABRIK (B.P. 342,715, 31.10.29).—The filling consists of helium and liquid mercury.

J. S. G. THOMAS.

**Electric-discharge devices.** GRAMOPHONE CO., LTD., and W. F. TEDHAM (B.P. 343,927, 25.11.29).—A process of introducing alkali vapour, *e.g.*, caesium, into electric-discharge tubes, comprising heating in an iron container a mixture of a carbide or hydride of an alkaline-earth metal, *e.g.*, calcium carbide, and a nitrate or carbonate of an alkali metal, *e.g.*, caesium carbonate, by low-frequency current, is claimed.

J. S. G. THOMAS.

**Photoelectric cells.** N.V. PHILIPS' GLOEILAMPENFABRIK (B.P. 343,930, 25.11.29. Holl., 16.5.29).—Particles of electrically-conducting material, *e.g.*, tungsten, or photoelectric material are distributed throughout the intermediate layer, supporting the photoelectric material, and consisting of a compound which is preferably a better absorber of the photoelectric substance than is the support for the intermediate layer. Thus, *e.g.*, the intermediate layer may be formed by a chemical reaction between substances, *e.g.*, tungsten, calcium fluoride, and silica, placed on the support. Alternatively, a mixture of caesium chromate, zirconium, and sodium fluoride may be heated on the support.

ture of caesium chromate, zirconium, and sodium fluoride may be heated on the support.

J. S. G. THOMAS.

**Stabilisation of light-sensitive [selenium] films.** F. H. CONSTABLE, and RADIOVISOR PARENT, LTD. (B.P. 343,939, 25.11.29).—Selenium is alloyed with a substance of the same crystalline form and having a low vapour pressure, *e.g.*, tellurium, bismuth, black phosphorus, or thallium, so that the vapour pressure of the alloy is reduced. Thus, *e.g.*, selenium may be alloyed with 1–5% of tellurium.

J. S. G. THOMAS.

**Electro-optical [Kerr] cells.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 343,853, 6.6.30. Ger., 8.6.29).—The "gap" of Kerr cells is formed between quartz or quartz-glass electrodes partly or wholly coated with metal, *e.g.*, nickel or gold, by cathode sputtering or by deposition from vapour. Nicol prisms for controlling the luminous flux are directly fixed to plates arranged on the gap bodies.

J. S. G. THOMAS.

**Electrolytic [low-frequency] rectifier.** LE R. P. BENSING, Assr. to G. P. KOELLIKER (U.S.P. 1,775,417, 9.9.30. Appl., 23.5.25).—An active ferrosilicon electrode, composed principally of silicon, immersed, with an inactive electrode, in an aqueous electrolyte, is claimed.

J. S. G. THOMAS.

**Apparatus for passing electric currents through gases, and regulating the same.** G. G. A. BRION and A. J. KRUTZSCH (B.P. 319,217, 17.9.29. Ger., 17.9.28).—Two sets of electrodes, respectively connected to separate sources of current, are arranged in the gas stream so that their electrical fields are substantially perpendicular to one another, the one field ionising the gas and the other causing migration of ions to the precipitating electrodes.

J. S. G. THOMAS.

**Preparation of [insulating] compositions.** ELECTRICAL RES. PRODUCTS, INC., Assees. of F. S. MALM (B.P. 343,567, 21.11.29. U.S., 20.12.28. Addn. to B.P. 307,906; B., 1930, 452).—Crude rubber or uncoagulated latex is mixed with about 4 pts. of 1% sodium chloride solution and heated at 130–240° for 1–30 hrs. If desired, a softener, *e.g.*, petrolatum, pine oil, or palm oil, is added to the latex before washing, in order to remove entrapped sodium chloride.

J. S. G. THOMAS.

**[Suspension of] elements for electric furnaces.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 345,874, 15.5.30. Ger., 15.5.29).

**[Earthing the charge in] electrical induction furnaces.** N. R. DAVIS, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 343,601, 26.11.29).

**Dry primary electric batteries.** E. R. TROUGHTON and E. C. HAMILTON (B.P. 343,158, 15.11.29).

**X-Ray tubes [with rotating anticathode].** N.V. PHILIPS' GLOEILAMPENFABRIK (B.P. 345,914, 28.7.30. Holl., 2.8.29. Addn. to B.P. 286,436).

**Photoelectric cells.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 345,436, 17.2.30. Ger., 15.2.29).

[Temperature compensation of] light-sensitive [selenium] bridges or cells. W. ENGELKE and H. L. READ (B.P. 343,885, 14. and 30.11.29 and 4.2.30).

Thermoelectric couples [for measuring electric currents]. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 343,452, 1.10.29).

Manufacture of apparatus comprising a thermoelectric couple. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 342,649, 30.9.29).

[Hard-paper] electric insulators. INTERNAT. GEN. ELECTRIC CO., INC., ASSECS. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 345,604, 5.8.30. Ger., 3.8.29).

Insulating fabrics.—See VI. Reflecting surfaces.—See VIII. Aluminium. Tungsten tubes.—See X. Conductive varnishes.—See XIII. Cheese.—See XIX. Vitamin-D.—See XX.

## XII.—FATS; OILS; WAXES.

Fatty acids and component glycerides of Indian ghee. R. BHATTACHARYA and T. P. HILDITCH (Analyst, 1931, 56, 161—170).—Two samples of cow ghee were examined in detail and their fatty acids found to resemble those from New Zealand and English cow butters reported previously by Hilditch and co-workers (B., 1929, 253; A., 1930, 1308). Two specimens of buffalo ghee gave similar figures to those for cow butter, and, although butyric acid is present in slightly larger amount and the proportion of linoleic to oleic acid is slightly lower, the chief differences consist in the greater content of stearic acid and the presence of arachidic acid. The glyceride structure shows that all these milk fats belong to the "heterogeneous" type, there being no evidence of any segregation of any particular acid to form a simple triglyceride. T. McLACHLAN.

Catalytic fat-hardening. I. Decrease of hydrogenation velocity with progressive saturation. II. Selective hydrogenation of train oil. L. UBBELOHDE and H. SCHÖNFELD (Z. angew. Chem., 1931, 44, 184—187).—The decrease in hydrogenation velocity (observed by Ubbelohde and Svanoe, B., 1919, 870 A) sets in when a certain iodine value, critical for each oil, has been attained, which is lower when more catalyst is employed, but is independent of the time taken to reach such iodine value; further, a catalyst which appears to have become inactive in use in one particular batch of oil recovers almost its full activity if applied to a fresh sample of oil (rape or train oil). Hence the decreased velocity of hydrogenation must be a consequence of the changing composition of the oil: the total unsaturation, however, is not the governing factor, since admixture of 20% of tallow to train oil does not affect the velocity of hydrogenation. Clupanodonic acid is hydrogenated more rapidly than the mono-, di-, and tri-unsaturated acids present in train oil. The degree of selectivity depends largely on the method of hardening employed, and is the less marked (*i.e.*, acids giving ether-insoluble bromides, together with the fishy odour, persist to lower iodine values of the oil) the more rapid is the hardening process used. By selecting suitable conditions an odourless oil free from clupanodonic acid, but still highly unsaturated (iodine value 120), can be prepared. E. LEWKOWITSCH.

New compounds produced during hydrogenation of fish oils. II. Higher alcohols. S. UENO and R. YAMASAKI (J. Soc. Chem. Ind., Japan, 1931, 34, 35—37 B; cf. B., 1931, 125).—The methyl alcohol extract from the unsaponifiable matter (1 kg.) obtained from the volatile hydrogenation products of fish oils consisted of higher alcohols (20 g.), with traces of malodorous aldehydes, but no ketones were present. The alcohols were fractionated by distillation and appeared to consist of C<sub>9</sub>, C<sub>10</sub>, and C<sub>13</sub> isoaliphatic primary alcohols of the saturated and olefine series. By catalytic reduction of one fraction (b.p. 150—163°, iodine value 55, mol. wt. 205) a saturated (C<sub>13</sub>) alcohol (liquid at —10°) was obtained which yielded a crystalline, m.p. 139—140°, and a crude, f.p. 17°, phenylurethane, and on oxidation with permanganate furnished a crude carboxylic acid, m.p. 36°, f.p. 17°.

E. LEWKOWITSCH.

Hardening of linseed oil at very high hydrogen pressure. H. I. WATERMAN and J. A. VAN DIJK (Rec. trav. chim., 1931, 50, 279—282).—"Solidification" of linseed oil by hydrogenation at high pressure is effected by relatively small quantities of hydrogen. A relationship between the iodine and thiocyanogen-iodine values of the partly hydrogenated oil is indicated, and this differs from the similar relationship for oils which are hydrogenated at low pressure.

J. D. A. JOHNSON.

Twitchell reagent. VII. Darkening of the fatty acid obtained by the Twitchell process. I. K. NISHIZAWA and K. FUJIMOTO (J. Soc. Chem. Ind., Japan, 1930, 33, 500—504 B. Cf. B., 1930, 870; 1931, 399).—The discoloration produced in olive oil (or its fatty acids) by treatment with fat-splitting reagents (7 varieties, under various conditions) was studied systematically. Sulphuric acid alone produced less colour in the fatty acids than did water, but hydrochloric acid had 2—3 times the darkening effect. The colour produced by 1% aqueous solutions of the reagents was intensified to a greater extent by the addition of hydrochloric acid than of sulphuric acid. When the fatty acids were heated with about 0.5% of the reagents alone the darkening varied with the particular reagent employed, but in all cases increased considerably with the time of heating, and was greater than in the case of aqueous solutions. (Some of the colour could be removed by washing with water after the experiments.) Discoloration still occurred, although to a less degree, when the tests were conducted *in vacuo*; in these cases the colour produced in fatty acids was about double that developed by oil under the same conditions. The previous conclusions (*loc. cit.*) are confirmed, that addition of strong acids is not a direct cause of increased discoloration. Strong acids accelerate hydrolysis, and the fatty acids formed react with the reagent and the depth of colour apparently increases, although the intensity of colour, as measured by the formula deduced in the previous papers, actually decreases with the progress of hydrolysis. The salting-out effect of strong acids, which causes more intimate contact between fatty acids and reagent and consequently increased colour, is a secondary phenomenon.

E. LEWKOWITSCH.



**Solubility of fats in various solvents.** Solubility of (I) camellia oil, (II) rape oil, in ethyl alcohol of various concentrations. K. HASHI (J. Soc. Chem. Ind., Japan, 1931, 34, 64—66 B, 66—67 B).—Charcoal-bleached, alkali-refined oils were used and the clearing and turbidity temperatures of solutions in aqueous alcohols are plotted. For both oils the curves connecting turbidity temperatures of solutions of stated oil content and the concentration of the alcohol were almost straight lines, especially near the critical concentration. For alcohols of concentrations (wt.-%) 99.76, 95.5, 90.45, and 85.5, the following values are given for critical temperature: (I) 71°, 99.8°, 129.8°, 155°; (II) 86.3°, 113.0°, 140.3°, —; and for critical concentration (oil %): (I) 40.8, 41.5, 44.0, 48.0; (II) 38.5, 40.3, 43.0, —. For both oils straight lines are obtained when the mean alcohol contents of both phases are plotted against temperature. E. LEWKOWITSCH.

**Rapid determination of total fat.** C. P. HARRIS (Ind. Eng. Chem. [Anal.], 1930, 2, 410—413; cf. Schwarz, B., 1930, 1036).—The fat content of seed, chocolate, etc. is determined by measuring the sp. gr. of a solution of the fat obtained by treating the material (100 g.) with a standard amount of a non-inflammable, non-volatile solvent (o-dichlorobenzene). Manipulation suitable for rapid routine use by unskilled operators is devised. E. LEWKOWITSCH.

**Combinations of raw materials for toilet soap.** I, II. B. NAKAGAWA (J. Soc. Chem. Ind., Japan, 1931, 34, 22—27 B, 28—31 B).—I. The effect of the addition of various soaps to a standard base (80% of tallow, 20% of coconut oil soda soaps) has been studied, using 0.5% solutions. A new value—the “lathering coefficient” (lathering number  $\times$  lathering volume)—is introduced to measure the lathering power, and this value is used, in conjunction with the rate of disappearance and the sp. vol. of lather, in appraising the soaps. Addition of hardened fish oil soap was disadvantageous; palm oil and arachis oil soda soaps, tallow potash soap, and especially castor oil soda soap have a good effect on the lathering phenomena; Chinese vegetable tallow is usable as a substitute for tallow; 40% is the optimum addition of lard soda soap. At 40° the presence of a hardened soya-bean oil (iodine value 67.7) soap is slightly favourable, but at 20° tends to the reverse.

II. The solid soaps were tested for hardness and solubility. A modification of Leimdörfer's apparatus (Seifensieder-Ztg., 1927, 54, 489) was used for determining the attrition or “rubbing solubility,” which is decreased by hardened fish oil soap, and to a smaller extent by Chinese vegetable tallow soap; it increases with additions of arachis-soda, tallow-potash, and especially castor oil-soda soaps. The standard soap is softened by additions of the three last-named soaps and hardened by additions of Chinese vegetable tallow and hardened fish oil soaps. E. LEWKOWITSCH.

**Solubility of metallic soaps of naphthenic, oleic, and stearic acids.** T. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 412 B).—The solubility of oleates and naphthenates on the whole are similar, but whilst mercury and lead naphthenates are easily soluble in alcohol at 5° (over 33% and 29%, respectively), the

corresponding oleates are practically insoluble (<0.1%, 0.2%). Acetone at 5° dissolves over 22% of zinc naphthenate and less than 0.4% of zinc oleate.

E. LEWKOWITSCH.

**Time of drying of linseed oil.** J. RINSE (Chem. Weekblad, 1931, 28, 131—132).—It is shown that the time of drying is much affected by humidity and by acid gases present in the atmosphere. Oxides of nitrogen decrease the time to a marked extent. S. I. LEVY.

**Influence of various conditions on the preservation of crude soya-bean oil.** V. M. SUICHOV (Chinese East Rly. Dept. Trans. Agric. Branch, 1928, No. 8, 3—116).—Least variation of sp. gr. occurs in oils kept in a dark, damp place; addition of 10% of water favours constancy. The viscosity of the oil increases with the density. The oil is decolorised by light; the action is accelerated by neutralisation, rise of temperature, or the presence of air and prevented by non-fatty impurities or water. Increase in acidity is due chiefly to fungi. The presence of insoluble impurities in the oil increases the free acidity, iodine value, and tendency towards decomposition. The oil should not contain insoluble organic impurities, and the water content should not exceed 0.2%. CHEMICAL ABSTRACTS.

**Determination of acidity of soya-bean oil.** N. I. MOROZOV (Chinese East Rly. Dept. Trans. Agric. Branch, 1928, No. 7, 38—44).—The sample (5 c.c.) is treated with a solution of magenta in petroleum and the colour is compared with that of standards. CHEMICAL ABSTRACTS.

**Colour reactions of sesamé oil.** T. PAVOLINI (Ind. Ol. Min. Grassi, 1930, 10, 41—42; Chem. Zentr., 1931, i, 179).—Rancidity etc. interferes with the usual colour reactions for the recognition of sesamé oil in mixtures. The following test is applicable: the oil (5 c.c.) is dissolved in ether (5 c.c.), treated with 2 c.c. of a (cold) reagent composed of 98% sulphuric acid (80 g.), 95% alcohol (10 g.), and water (10 g.). A few min. after vigorous shaking there appears in presence of sesamé oil a green (or greenish-yellow) colour and a green fluorescence in the acid layer. A. A. ELDRIDGE.

**Apparent change of volume of castor oil and Baku lubricating oil at low temperatures and high pressures, caused by increase of viscosity and consequent stoppage of narrow tubes.** G. TAMMANN and A. PAPE (Z. anorg. Chem., 1931, 197, 90—92).—The abnormal secondary pressure changes observed when the oils are forced under pressure through capillary tubes are a result of an increase of viscosity.

H. F. GILLBE.

**Oil of *Wrightia annamensis*, Dubard and Eberhardt: an oil similar to castor oil.** L. MARGAILLAN (Compt. rend., 1931, 192, 373—374).—The oil, obtained from the seeds of *W. annamensis* by extraction with various solvents, has the following constants:  $d_{25}^{25}$  0.966,  $n_D^{25}$  1.480, saponif. value 184, iodine value (Hübl) 85, acetyl value 127, free acids (as oleic) 2.7%, unsaponifiable matter 1%. The variation in viscosity with temperature is similar to that of castor oil. The chief fatty acid in the oil is a hydroxyoleic acid, probably identical with ricinoleic acid; a small amount of a hydroxylinoleic acid is also present. The seeds contain

water (5.8%), fatty material (36.1%), nitrogenous substances (29.1%), cellulose (5.1%), ash (4.3%), and nitrogen-free extractives (19.6%). H. BURTON.

**Vegetable oils of the Union of S.S.R. VII. Oils of the *Compositæ*.** S. IVANOV (Chem. Umschau, 1931, 38, 53—55; cf. B., 1931, 258).—Chemical data (fragmentary) for some 30 oils from seeds of species of the *Compositæ* are collected, many of which have been examined for the first time by the author. The edible oil (30—45%) from seeds of the "Kangal" thistle (*Silybum Marianum*, Gertn.; cf. Micheev, Subtropiki, 1930, No. 1—2) had acid value 1.49, saponif. value 177.05 [?], iodine value 114.68. [With L. E. KOMAROVA.] The pale yellow odourless oil (16.17%) from seeds of *Onopordon acantum* from Turkestan resembled sunflower seed oil and had acid value 3.1—3.59, saponif. value 183.97—184.90, iodine value 136.28—136.74; this oil should be producible commercially. E. LEWKOWITSCH.

**Examination of textile oils.** R. B. TRUSLER (Oil & Fat Ind., 1931, 8, 51—57, 77, 103—106, 109; cf. Textile World, 1930, Mar. 8).—The Mackey test is too susceptible to small differences in manipulation, and, moreover, gives very little information concerning the oxidisability of oils at low temperatures, e.g., 25—50°. In a new test (described in full detail) 25 g. of the oil are shaken with 100 c.c. of oxygen for 1 hr. at the required temperature, and the unconsumed oxygen is measured directly. Curves of oxygen absorption-temperature are plotted and illustrate well differences in oxidisability of similar oils. The results are comparable with the curves obtained by the Mackey test representing oxidation at higher temperatures. The presence of free fatty acids renders an oil more susceptible to oxidation (cf. Aspegren, B., 1929, 217).

E. LEWKOWITSCH.

**Analysis of Turkey-red oils and their products** (Collegium, 1931, 21—27).—A simplified scheme of analysis, as drawn up by the German Oils and Fats Commission ("Wizöff"), is given.

**Lubricating oils.**—See II. Soya beans.—See III. Fat-impregnated wool.—See VI. Vitamins in margarine.—See XIX.

See also A., April, 424, Effect of ultra-violet light on emulsions. 484, Oxidative degradation of  $C_{13}H_{14}$  from Congo copal oil. 507, Fatty acids of cat's kidney. 524, Vitamin-A and cod-liver oil. Natural fats and vitamin-A. 531, [Vegetable oils and] vitamin-D. 535, Formation of oil in plants. Laurel fats.

#### PATENTS.

**Steam-boiling apparatus for treatment of materials such as the meat, blubber, and bones of whale.** A. SOMMERMEYER (B.P. 343,699, 21.1.30).—The interior surface of the continuously acting rotatory drum used is provided with a number of lugs or projections to assist disintegration of the treated material.

E. LEWKOWITSCH.

**Washing soaps.** E. L. TYSON (B.P. 343,598, 26.11.29).—Household or laundry soap is dissolved in water, and turpentine, saltpetre, ammonia, and paraffin oil are added; if desired, some of the water in the mixture

may be evaporated off and the soap moulded, powdered, or flaked.

II. ROYAL-DAWSON.

**Extraction of oil from cottonseed.** R. O. BOYKIN, Assr. to N. R. VAIL (U.S.P. 1,775,154, 9.9.30. Appl., 8.12.25).—The albuminous or glutinous matter in the ground seed, which tends to hinder filtration of the oil-solvent extract, is coagulated by heat or by agents such as calcium hydroxide (1—2%), strontia water, brine, or acetic acid, prior to extraction. Benzene is preferably used as solvent and is evaporated from the finely-distributed oil solution by steam under high vacuum, rendering subsequent deodorisation unnecessary. Details of plant for continuous extraction are given.

E. LEWKOWITSCH.

**Purification of ergosterol.** C. E. BILLS, Assr. to MEAD JOHNSON & Co. (U.S.P. 1,775,548, 9.9.30. Appl., 15.4.29).—Crude ergosterol is recrystallised from a mixture of acetone and ether (preferably in the proportions 3:1 by vol.).

E. LEWKOWITSCH.

**Manufacture of thermoplastic products from fatty oils.** IMPERIAL CHEM. INDUSTRIES, LTD., and H. M. BUNBURY (B.P. 343,099, 8.8.29).—Unsaturated fatty oils are polymerised by heating at or above 200° in a stream of an inert gas (preferably under reduced pressure), and about 5—10% of a soap (sodium, magnesium, etc.) of a higher (preferably saturated) fatty acid is dissolved in the viscous product. The thermoplastic product may be vulcanised by any usual method and used as rubber substitute, or compounded with solvents etc. for the production of varnishes or lacquers.

E. LEWKOWITSCH.

**Manufacture of sulphurised fatty-oil products and of emulsions therefrom.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, and R. B. F. F. CLARKE (B.P. 343,533, 14.10.29).—Highly sulphurised oils (cf. B.P. 343,099; preceding abstract) are rendered readily emulsifiable by incorporating a small proportion of a substance consisting of or containing tannic acid, gallic acid, or tannins (e.g., quebracho, mimosa extract, etc.), before, during, or after the vulcanisation.

E. LEWKOWITSCH.

**Manufacture of esters of sulphonated fatty acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,989, 2.9.29).—Sulphonated unsaturated fatty acids (e.g., sulphonated oleic or ricinoleic acids, are esterified with an ester of an aliphatic or aromatic-aliphatic alcohol with an inorganic acid (e.g., benzyl chloride) or an arylsulphonic acid (e.g., methyl *p*-toluenesulphonate). The use of catalysts is mentioned in the provisional specification.

E. LEWKOWITSCH.

**Manufacture of sulpho-derivatives of higher fatty acids.** I. G. FARBENIND. A.-G., G. KALISCHER, and K. KELLER (B.P. 343,071, 7.11.29. Addn. to B.P. 342,761; B., 1931, 450).—The prior process is carried out using as starting materials the water-soluble salts of the halogenated fatty acids; less sulphite is then required. The more halogen atoms and hydroxyl groups are present in the molecule of the starting material the milder may be the sulphonation conditions.

E. LEWKOWITSCH.

**Refining of fats. Sulphonic acids.**—See II. Detergent.—See VII. Vitamin-D.—See XX.

**XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.**

**Composition of turpentine.** B. A. ARBUSOV (J. Appl. Chem., Russia, 1930, 3, 867—873).—A commercial turpentine,  $d_{4}^{15}$  0.8660,  $\alpha_D^{15} + 22.53^\circ$ , contained  $d$ - $\alpha$ -pinene 58.3,  $d$ - $\Delta^3$ -carene 20.5, dipentene and  $d$ -limonene 10.6, terpinolene with traces of  $\alpha$ -terpinene 3.0, alcohols 6.0, sesquiterpenes 1.0, residue 1.5%.

**CHEMICAL ABSTRACTS.**

**Iodine value of turpentine oils.** V. KUBELKA and S. ŽURAVLEV (Chem. Listy, 1931, 25, 124—128).—The various hydrocarbons present in different fractions of turpentine oil cannot be determined from the iodine value. If it be assumed that this value (Hanus) is a measure of the number of ethylenic linkings, it appears that the degree of unsaturation of the different fractions is approximately constant. Oils 18 months old possess substantially the same iodine value as freshly distilled oils.

R. TRUSZKOWSKI.

**Painting of [cement] façades.** H. WOLFF (Farbe u. Lack, 1931, 99—100).—A general discussion is given on the wide range of paints that find use on cement façades, and the destructive influences to which they are subjected, e.g., water (solvent and mechanical action), lime, and mould formation. It is pointed out that the complete neutralisation of lime in cement is a more lengthy process than is generally recognised, and the case of a 10-year-old plaster of high lime content in places is instanced. The action of *Aspergillus niger* on a white oil paint is illustrated and described. The old encaustic treatment of surfaces, i.e., application of wax preparations by heat, has recently been revived, but is generally too costly. Substitutes for this process are offered by proprietary solutions of wax in organic solvents, or water emulsions of wax containing organic solvents as stabilisers; the degree of penetration of such products is important, however, since it is desirable to prevent absorption of water without rendering it impossible for the surface to "breathe."

S. S. WOOLF.

**Plasticity of paints.** F. H. RHODES and W. J. JEBENS (J. Physical Chem., 1931, 35, 383—404).—The plasticity of paints made from linseed oil and zinc oxide, white lead, aluminium powder, iron oxide, or white lead-zinc oxide mixtures has been investigated by the method of Rhodes and Wells (B., 1930, 109). The addition of water to paints prepared with an oil of low acid value increases the yield value and lowers the mobility, the extent of the change depending on the type and the concentration of pigment. In general, the change in mobility appears to vary with the ease of wetting of the pigment by water. With a paint of zinc oxide in neutral linseed oil, the addition of sodium oleate or calcium oleate increases the yield value and decreases the mobility, sodium oleate having the greater effect; the joint effect of water and soap is qualitatively the same and is additive. Free fatty acids prepared from linseed oil markedly increase the consistency of paints prepared from the oil and dry zinc oxide, but small additions of water counteract a part of this increase; the effect is most pronounced with 1% of water and relatively high acid values. With paints of linseed oil and white lead, the effect of free fatty acids is relatively small; with an acid value of 2.40 or more, the mobility is at a maximum

with 0.5% of water and the yield value increases slightly with an increase in the water content. Anhydrous zinc sulphate has practically no effect on the plasticity of zinc oxide paints. In such a paint the increased consistency due to free fatty acid can be offset by heating at 150° for 30 min. or by the addition of 2 pts. of calcium oleate.

L. S. THEOBALD.

**Adhesion in the painting and in the glueing of wood.** F. L. BROWNE (Ind. Eng. Chem., 1931, 23, 290—293).—The adhesion between wood and glue has been studied by various observers from the aspects of strength of wood joints and microscopical examination of joint sections, and a summary of this work is given. The distinction between "strong" and "starved" wood joints is discussed and illustrated, the behaviour of glue tendrils in wood cavities being more significant than deep penetration. Two kinds of adhesion, specific and mechanical, are postulated, and it is considered that the influence of the former has been neglected. Paint adhesion is treated as analogous to glue adhesion, and the widely held opinion that priming paints must penetrate wood deeply is declared unsound, since the filtering off of paint vehicles by absorption is to be regarded as undesirable. Paint coatings probably adhere "specifically" when young, but later the adhesion becomes mechanical only, when the lumina in summerwood may prove too small for adequate anchorage.

S. S. WOOLF.

**Ethylcellulose dope.** I, II. T. ARAKI and T. NAGAMOTE (J. Soc. Chem. Ind., Japan, 1930, 33, 382 B, 382—383 B).—I. Solutions of ethylcellulose in benzene or toluene are turbid, but the addition of 3.5—37.5% of alcohol renders them clear. Petroleum may be used as a diluent for the dope, and tricresyl and triphenyl phosphates as plasticisers, but not castor oil. A dope consisting of ethylcellulose (12 g.), benzyl alcohol (2 g.), benzene (60 c.c.), and methyl alcohol (13 c.c.) is suitable as an undercoat for aeroplane fabric; plasticisers and pigments should be added for coverings or pigmented dopes.

II. No appreciable change occurs on storing ethylcellulose dope solution for 1½ years. The ethylcellulose film (tensile strength 5 kg./mm.<sup>2</sup>) is slightly more inflammable than the acetylcellulose, but less than the nitrocellulose film, whilst fabric doped with ethylcellulose is less loosened by atmospheric moisture than that treated with acetylcellulose. Exposure tests show that the clear dope is not suitable for use alone, but gives better results when pigments or aluminium powder and plasticisers are added.

F. R. ENNOS.

**Dry distillation of tolu balsam.** J. DUPONT and J. J. GUERLAIN (Compt. rend., 1930, 191, 716—717).—Appreciable amounts of guaiacol, 3-methoxy-*p*-cresol, and probably 4-hydroxy-3-methoxyethylbenzene are obtained during the dry distillation of tolu balsam; the same compounds have been extracted by Béhal and Choay (A., 1894, i, 575) from wood-tar creosote.

H. BURTON.

**Preparation of synthetic resins from alkali-lignin.** M. PHILLIPS and H. D. WEIHE (Ind. Eng. Chem., 1931, 23, 286—287).—Lignin, isolated from corn cobs (cf. A., 1929, 1168), was condensed with furfuraldehyde (in the presence of hydrochloric acid, sodium

carbonate, pyridine, and lime, respectively, as catalysts) and with a series of aromatic amines, and the optimum temperature, time of heating, and concentrations for the preparation of resins were investigated for each reaction. The resins are fusible and readily soluble in mixtures of organic solvents. Their applications as wood stains and as binders in the preparation of compressed paper boards are indicated. S. S. WOOLF.

#### Adhesives.—See XV.

See also A., April, 420, Adsorbents for dyes. 422, Matter in the film state. 446, Titanium white. 491, Xanthophyll of yellow pansy. 492, Polyene pigments of azafran root. Turpentine of *Pinus sylvestris*.

#### PATENTS.

**Refining of [mineral] pigments.** H. GROSSMAN, Assr. to W. S. PRITCHARD (U.S.P. 1,774,510, 2.9.30. Appl., 2.2.27).—An aqueous suspension of the pigment, produced by grinding and deflocculating the raw material in sulphite-cellulose waste liquor, diluting with water, and decanting off the suspension after impurities have settled out, is flocculated by the addition of an electrolyte and the pigment is filtered off, dried, and ground. L. A. COLES.

**Electrical conductive varnishes.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 343,282, 21.1.30. Ger., 25.1.29).—Insulating varnishes are mixed with a conductor, e.g., carbon or metal powder, and lithopone. S. S. WOOLF.

**Manufacture of artificial masses.** SOC. CHEM. IND. IN BASLE (B.P. 342,730, 6.11.29. Switz., 6.11.28. Addn. to B.P. 284,589; B., 1928, 826).—The infusible condensation product described in the prior patent is compressed in the presence of a fusible aldehyde condensation product as fluxing agent. S. S. WOOLF.

**Manufacture of artificial masses [aldehyde-amine resins].** SOC. CHEM. IND. IN BASLE (B.P. 342,767, 7.11.29. Switz., 7.11.28).—A Schiff's base is treated with an acid, the acid is removed, and the mass is washed free from electrolytes, dried, powdered, and hardened under pressure in presence of formaldehyde or other aldehydic hardening agent. The aldehyde may be added at the same time as the acid. At 150°/180 atm. translucent or transparent products are obtained. C. HOLLINS.

**Manufacture of artificial masses [hardening of aldehyde-amine resins].** SOC. CHEM. IND. IN BASLE (B.P. 342,723, 4.11.29. Switz., 2.11.28. Addn. to B.P. 284,589 and 342,325—6; B., 1928, 826; 1931, 405).—The fusible resins of the prior patents are hardened by incorporation of a thermo-hardening aldehyde condensation product, e.g., phenol-formaldehyde resole. C. HOLLINS.

**Manufacture of solutions of infusible amine-aldehyde condensation products.** SOC. CHEM. IND. IN BASLE (B.P. 343,031, 11.11.29. Switz., 10.11.28).—Infusible products of this type are treated with a chlorohydrin and, if desired, a swelling agent, e.g., cyclohexanone, ethyl lactate, chloroform. The dissolution may be accelerated by heating, and further solvents,

diluents, plasticisers, dyes, fillers, pigments, etc. may be added. S. S. WOOLF.

**Manufacture of colourless and transparent phenol resin.** M. NAKAMURA, Assr. to SANKYO KABUSHIKI KAISHA (U.S.P. 1,775,135, 9.9.30. Appl., 24.6.26. Jap., 2.9.25).—Phenol and formaldehyde are condensed in the presence of an acid catalyst (hydrochloric acid) and 25–80% of a urea-formaldehyde condensation product, the mixture being dried and hardened at 80–130°. L. A. COLES.

**[Bituminised felt-linoleum] floor coverings and the like.** D. L. IRWIN and R. E. TIPPINS (B.P. 345,386—7, 14.1.30).

**Plasticisers.**—See III. **Cellulose ester solutions.** **Softener for cellulose derivatives.** **Laminated materials.**—See V. **Insulating fabrics.**—See VI. **Compound glass.**—See VIII. **Thermoplastic products.**—See XII. **Adhesive.**—See XV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Separation of solvent from rubber and calculation of the energy change involved.** P. STAMBERGER (Kautschuk, 1931, 7, 68–70).—A mathematical treatment of the energy changes in the separation of solvent from dissolved rubber by means of a semi-permeable membrane or by evaporation. D. F. TWISS.

**Sulphur in rubber.**—See VII.

See also A., April, 492, Constitution of caoutchouc.

#### PATENTS.

**Manufacture of refined gutta-percha, balata, and the like.** W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 343,510, 14.11.29).—The low-grade constituents of deteriorated or inferior guttas are removed by treating the latter with a solvent which will dissolve only the former. Hot benzene (17–25°) and light petroleum (32–40°) are suitable solvents for the purpose. An alternative method is to dissolve the gutta completely in the solvent, and to cool the solution until crystallisation occurs, after which the mixture is warmed to within the effective temperature range, e.g., to 19° for benzene, and the purified gutta is removed from the mother-liquor. If the gutta contains resins, it may be desirable to remove these before carrying out the above purification process. D. F. TWISS.

**Formation of rubber strips from latex.** GOOD-YEAR TIRE & RUBBER CO., Assees. of C. R. PARK (B.P. 343,315, 17.2.30. U.S., 14.5.29).—A relatively thick, continuous layer of material, such as the tread for a motor tyre, is produced by extruding a number of thin layers of compounded latex into a bath of coagulant, e.g., acetic acid or acetone, and pressing them together in a superposed relation so as to yield a homogeneous strip. D. F. TWISS.

**Manufacture of continuous lengths of plastic materials.** SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 343,434, 8.11.29).—In the manufacture of continuous lengths of plastic material, such as rubber thread or tubes, particularly from latex, the shaped material is borne away, as produced, on or near the

surface of a liquid which moves continuously at such a speed relative to that of the extruded material as to produce a tractional effect thereon. D. F. TWISS.

**Production of aqueous emulsions of rubber, balata, or gutta-percha.** W. B. PRATT, Assr. to DISPERSIONS PROCESS, INC. (U.S.P. 1,775,569, 9.9.30. Appl. 2.12.22).—Rubber is dissolved in a volatile solvent such as benzene, together with a saponifiable substance such as oleic acid, and an aqueous solution of a saponifying agent, *e.g.*, ammonia or a suitable amine, is gradually added with constant agitation but without heating, until the rubber solution becomes the disperse phase. The rubber solvent can then be removed by evaporation under reduced pressure, the final product being an aqueous dispersion containing at least 25% of substantially undepolymerised rubber.

D. F. TWISS.

**Production of rubber products [containing artificial silk].** H. SUTER (B.P. 343,617, 29.11.29. Ger., 28.12.28).—Textile-reinforced rubber products, such as belting or pneumatic tyres, are produced by embedding in the rubber an artificial silk, *e.g.*, non-desulphurised viscose, of high tensile strength (at least 200 g. and preferably above 300 g. per 100 denier), of extensibility below 10% and preferably below 6%, and of a separate thread titre below 2 deniers and preferably below 1 denier. The wet strength of the artificial silk should be at least 100 g. and preferably above 200 g. per 100 deniers.

D. F. TWISS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., E. A. MURPHY, and R. G. JAMES (B.P. 343,921—2, 25.11.29).—(A) Aqueous dispersions of natural or artificial rubber, which may have been vulcanised, are mixed with aqueous emulsions of albumins, particularly blood-albumin, in the presence of a substance tending to inhibit the coagulation of the albumin, *e.g.*, ammonia. Such mixtures, containing 25—75% of albumin on the dry solids, when evaporated to dryness, preferably below 100°, yield a tough, semi-transparent, gutta-like residue capable of being softened by moderate heat. (B) The aqueous mixtures prepared as in (A) are converted into crumb-like masses, *e.g.*, by addition of a coagulant such as aluminium sulphate, which can be dried rapidly and consolidated by pressure.

D. F. TWISS.

**Manufacture of articles of or containing rubber or similar material.** DUNLOP RUBBER CO., LTD., E. A. MURPHY, R. G. JAMES, and D. F. TWISS (B.P. 343,532, 23.9.29).—Supports, *e.g.*, in the form of moulds, are coated with an acidified aqueous dispersion of rubber and are thereafter coated with a rubber dispersion of the normal alkaline reaction. The prior coating effects the coagulation of the superposed dispersion. The coagulation of the second coating may be assisted by the external application of another coating of acidified dispersion or of other coagulant.

D. F. TWISS.

**Coating of solid surfaces with rubber or the like.** DUNLOP RUBBER CO., LTD., D. F. TWISS, A. A. ROUND, and E. A. MURPHY (B.P. 343,531, 23.9.29).—A bonding agent for the attachment of rubber to impermeable rigid surfaces, such as metal, is prepared by the

action of sulphuric acid or sulphonc acids on gutta-percha or balata.

D. F. TWISS.

**Vulcanisation of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 344,265, 11.4.30. U.S., 12.4.29).—Many vulcanisation accelerators are markedly activated by traces of hydrogen sulphide or of labile sulphur such as arise during vulcanisation from the interaction of sulphur and non-caoutchouc constituents of rubber. Cadmium oxide and hydroxide are able to combine with hydrogen sulphide or labile sulphur, and so are able to prevent premature vulcanisation or scorching, particularly when using accelerators of the thiuram disulphide or aldehyde-amine classes or carbon disulphide derivatives of the latter. [Stat. ref.]

D. F. TWISS.

**Age-resisting rubber and rubber compound.** H. A. MORTON (B.P. 315,661, 15.7.29. U.S., 14.7.28).—2:3-Dihydro- and tetrahydro-glyoxalines, unsubstituted or carrying a hydrocarbon group in 2-position, or in any or all of the positions, are added to rubber compositions. Specific examples are the 2:4:5-triphenyl- and -trifuryl-2:3-dihydroglyoxalines, and the 1:3-di- $\alpha$ -naphthyl-, 1:3-diphenyl-2-propyl-, 2-phenyl-1:3-di-*p*-tolyl-, 1:3-diphenyl-2-propenyl-, 1:3-di-*p*-tolyl-2-methyl-, 2-thiol-1-phenyl-3-xylyl-, and 2-phenyl-1:3-di- $\beta$ -naphthyl-4-methyl-tetrahydroglyoxalines.

C. HOLLINS.

**Treatment [retarding the deterioration] of rubber.** NAUGATUCK CHEM. CO., Assees. of C. COLEMAN (B.P. 344,174, 7.2.30. U.S., 15.2.29).—A diamino-diphenylmethane or the formaldehyde condensation product of such a compound is applied as an antioxidant for rubber. Directions are given for the production of 4:4'-diaminodiphenylmethane.

D. F. TWISS.

**Viscous hydrocarbons etc.**—See II. **Thiazole compounds.**—See III. **Insulating compositions.**—See XI. **Thermoplastic products.**—See XII.

## XV.—LEATHER; GLUE.

**Bacteriology of red stains on salted raw hides.** W. HAUSAM (Collegium, 1931, 12—16; cf. B., 1930, 732).—The "*Proteus*" isolated by Stather and Liebscher (B., 1929, 991) consists of a number of different organisms none of which was a *Proteus*. The delicate rose colour of the earlier original cultures was shown to arise from *B. prodigiosum* (Ehrenberg), frequently occurring in sea-water, and capable of liquefying gelatin. Another gelatin-liquefying organism similar to *B. punctatum*, and three non-gelatin-liquefying organisms, viz., *B. coli*, varieties of *B. coli* resembling the paratyphoid group, and an organism of the *B. putidum* (*B. fluorescens non liquefaciens*) group, have been isolated.

D. WOODROFFE.

**Physico-chemical differences of hide powder by sex.** T. TADOKORO and K. YOSHIMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 37—38 B).—Hide powder, refined by extraction by the Wilson-Kern method, was prepared from (b) bull and (c) cow skins. In neutral solutions of salts the acid-producing power of (b) was the greater (cf. muscle fibres of the male). The absorptive power of (c) for iodine is greater, but for tannin is less than that of (b); the difference is well marked in the curves for the logarithmic equations. The authors suggest that

the female hide contains a protein molecule differing from that of the male, *e.g.*, by a greater iminazole ring content. In neutral salt solutions crude hide powder from the female showed considerably greater swelling power than that from the male (maximum in both cases in 0.5–0.2*N*-solutions): this difference was not observable with the refined powders.

E. LEWKOWITSCH.

**Determination of the copper contents of tanning extracts.** Report of a Committee of the Society of Leather Trades' Chemists. D. BURTON (J. Soc. Leather Trades' Chem., 1931, 15, 99–106).—Four methods for determining traces of copper have been tested, but the following is found to be the most satisfactory. 50 G. of the extract are dried at 100°, ashed at a low temperature, the ash completely moistened with concentrated sulphuric acid, avoiding excess, warmed to expel chlorides, cooled, 10 c.c. of 2*N*-sulphuric acid are added, and the residue is dissolved by warming. The solution is diluted to 100 c.c., and the copper determined by electrolysis overnight. Olive-green stains on sole leather and heavy deposits of sludge from tan liquors in the early stages of tanning have been traced to the copper in an extract; in one case, *viz.*, that of a liquid chestnut extract, less than 0.02% Cu was found.

D. WOODROFFE.

**Determination of water in vegetable[-tanned] leather.** R. F. INNES and J. G. M. COSTE (J. Soc. Leather Trades' Chem., 1931, 15, 126–136).—Official methods of determining water in leather have been examined. Samples of heavily greased leather both before and after degreasing were heated in an air oven at 110° for 24 hrs. and the losses in weight noted at intervals. Constant weight was attained only after 24 hrs. Lower results were given by heating in a vacuum oven at 98° and under 25 mm. pressure. Grease and sulphur were deposited on the walls of the ovens. Crust-tanned sheep leathers continued to lose weight, even after heating for 6 hrs. daily for 21 days. Moisture in many leathers cannot be determined with certainty by oven methods.

D. WOODROFFE.

**Fastness of pigment finishes [for leather] to rubbing.** M. C. LAMB and R. DENYER (J. Soc. Leather Trades' Chem., 1931, 15, 107–113).—The pigment should be finely ground in a ball mill, using soluble oil, sulphonated oil, or, preferably, soap solution as medium, and the product kept in the wet condition until mixed with the binding agents. The product should be mixed with a mixture of solutions of casein and glue as the binding agents, glycerin as the softening agent, and phenol or nitrobenzene as antiseptic. The dyed leather should be sprayed with a very light coat of the undiluted product, allowed to dry until tacky, and sprayed with a 1:1 mixture of formaldehyde and water. The addition of a tung oil emulsion to the ordinary commercial pigment finishes is also suggested for increasing their fastness to rubbing.

D. WOODROFFE.

**Collagen fibre.** F. HALLA and R. TANDLER (Z. physikal. Chem., 1931, B, 12, 89–92; cf. Herrmann, Gerngross, and Abitz, A., 1931, 27).—Stretched, raw collagen fibre from the skin of *Delphinus brevirostris* and stretched, chrome-tanned collagen fibre from the skin

of *Gadus morrhua* give the same X-ray diagram as stretched gelatin. No fibre diagram is obtained, however, from fibres from the latter fish, which have been treated with organic tans and stretched after drying.

R. CUTHILL.

**Wood glues and their control: use of adhesive films for wood.** O. GERNGROSS (Z. angew. Chem., 1931, 44, 231–237).—A transparent paper 0.01 cm. thick, impregnated with cresol-formaldehyde and an adhesive (casein, blood-albumin, or hide or bone glues) is placed between the pieces of wood, which are then subjected to heat and pressure for 10–45 min., whereby the adhesive undergoes the "bakelite" reaction. These papers, known as "Tego films" (T. Goldschmidt A.-G.) have been used in the manufacture of 3-ply woods, and the shear and tensile strengths of the latter determined, both dry and after 24 or 48 hrs.' immersion in water, respectively. The strength of birch and pine woods was not affected by the high temperature and pressure of the press. The shear strength of ply woods united by Tego films complied with the specifications of the German Aircraft Research Station, *viz.*, 20 kg./cm.<sup>2</sup> (dry) and 10 kg./cm.<sup>2</sup> (wet). When the water content of the woods was 2–5%, the pressure applied not less than 10 kg./cm.<sup>2</sup>, and the temperature below 130°, 3-ply woods were produced which were characterised by high elasticity and flexibility. The use of acetylcellulose films for cementing the plys rendered the products less susceptible to the moisture content of the wood.

D. WOODROFFE.

**Defects in raw hides and their resulting effects on the finished leather.** F. STATHER (Collegium, 1931, 3–12).—See B., 1931, 408.

**Glueing of wood.**—See XIII. Tannery wastes.—See XXIII.

See also A., April, 456, Glass electrode [for tan liquors]. 493, Condensation of catechin tannin. 503, Heat-coagulable protein from gelatin. 521, Action of enzymes in leather chemistry.

#### PATENTS.

**Manufacture of tanning materials [from lignite etc.].** A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 344,015, 26.11.29).—The products obtained according to B.P. 284,670 and 323,781 (B., 1929, 505; 1930, 311) are lixiviated with water or dilute acids after complete or almost complete dehydration, *e.g.*, with air, at temperatures below 100°. D. WOODROFFE.

**Treatment of mineral-tanned leather.** R. SAJITZ and E. THIEL (CHEM. FABR. POTT & Co.) (B.P. 317,834, 23.8.29. Ger., 23.8.28).—Mineral-tanned leather is thoroughly soaked, subsequently to and separately from the tanning process, with solutions of neutral alkali salts of simple and condensed aromatic sulphonic acids, with or without a carbohydrate, solvents, a sulphonated oil, and a soap; it can then be dried out, stored, and subsequently "wet-back" when necessary.

D. WOODROFFE.

**Treatment of leather.** W. H. ALLEN, Assr. to G. E. MAURER (U.S.P. 1,774,626, 2.9.30. Appl., 5.8.27).—Tanned skins are impregnated with 10–30% solutions of calcium chloride or other inert salt capable of

absorbing water, dried until the leather has shrunk sufficiently, the salt washed out, and the leather finished.

D. WOODROFFE.

**Manufacture of artificial leather.** (MISS) A. ENGEL (B.P. 342,908, 13.2.30. Fr., 13.2.29).—Leather scraps are oxidised, e.g., with ozone, before or during trituration, stabilised latex, basic dyes, a mordant (sumac extract), and an emulsifying agent (blown fish oil) are added to the disintegrated mass, and the product is made into sheets or "board."

D. WOODROFFE.

**Manufacture of gelatin.** E. ATHERTON (B.P. 344,303, 26.5.30).—Sheet gelatin is heated suddenly at 170–537° (e.g., 482°), whereby it becomes puffed, and is then broken or ground into flakes or powder; the product is subsequently allowed to absorb moisture from a sterilised atmosphere.

D. WOODROFFE.

**Adhesive material.** COMP. CENTRALE DES EMERIS ET PRODUITS À POLIR (B.P. 343,970, 24.10.29. Fr., 25.10.28).—A mixture of drying oil (1000 pts.) and not more than 25% of asphalt or bitumen (40 pts.) is heated with earths (umber 15 pts., green earth 10 pts., ultramarine 50 pts.). Alternatively, the drying oil is heated to 135–150°, the earths (or other usual oxidising substances) are added, the product is heated to about 245° and mixed, the asphalt or bitumen then added and the temperature raised to about 260–270°. When the desired degree of viscosity has been reached the product is cooled, hardened by heat, ozone, or ultra-violet light, diluted with a volatile solvent, and used for applying abrasive grains to a suitable backing, the product being unaffected by water, or for waterproofing textiles, paper, etc.

D. WOODROFFE.

**Machines for steeping pelts.** VEREIN. HUTSTOFFWERKE BLOCH & HIRSCH, C. F. DONNER GES.M.B.H. (B.P. 345,433, 15.2.30. Ger., 22.3.29).

**Imitation leather.**—See V.

## XVI.—AGRICULTURE.

**Acid and alkali soils in China.** T. Y. TANG (Lingman Sci., 1930, 9, 195–197).—A discussion.

CHEMICAL ABSTRACTS.

**Soil excrescences.** II. H. PUCHNER (Kolloid-Z., 1931, 54, 87–92; cf. A., 1917, i, 832).—The forms taken by salts on efflorescence from various soils are described.

E. S. HEDGES.

**Effect of lime on the texture of soil.** W. REIMER (Pflanzenbau, 1930, 4, 251–270; Chem. Zentr., 1931, i, 344).—Neither in mineral nor in humic soils does addition of lime or calcium carbonate cause marked changes of hygroscopicity or heat of moistening. The indirect action, through promotion of bacterial decomposition of organic matter, may consist of an increase or decrease of the soil surface.

A. A. ELDRIDGE.

**Action of silicic acid [in soils].** O. LEMMERMANN (Z. Ver. deut. Zucker-Ind., 1930, 80, 956–966).—Objection is taken to a number of statements concerning the author's work and opinions in a recent paper by Krüger and Wimmer (B., 1931, 37). J. H. LANE.

**Effect of frost on the elimination of carbon dioxide from soil.** G. HEYN (Pflanzenbau, 1929, 1, 671–

724; Chem. Zentr., 1931, i, 344).—Frost appears to have no specific physiological effect on soil bacteria; hence the increased elimination of carbon dioxide caused by frost is attributed to mechanical cracking increasing the surface exposed to oxygen.

A. A. ELDRIDGE.

**Determination of adsorbed bases in soils containing carbonates.** J. KANIWETZ (Verlag Zentrallab. Agric. Chem. Kiev, 1928; Proc. Internat. Soc. Soil Sci., 1930, 5, 110–112; cf. B., 1930, 578).—Comparative examination of the Hissink and Gedroiz methods for determining adsorbed bases is described. Hissink's method is modified by collecting 8 successive  $\frac{1}{2}$ -litre portions of neutral salt extract, to obtain greater accuracy with soils containing carbonates. The quantity of adsorbed bases as determined by titration methods always exceeds that by the modified Hissink process. From soils containing carbonates 0.05N-hydrochloric acid (Gedroiz) removes not only adsorbed sodium and potassium, but also other forms of these bases. There is no definite relationship between the results of the two methods examined. If, however, in the Gedroiz method, the adsorbed calcium is taken as the difference between the calcium content of 0.2N- and 0.05N-hydrochloric acid extracts, the results approach those obtained by leaching with sodium chloride solution after decomposition of the carbonates.

A. G. POLLARD.

**Detection of fluorine in plants and soils.** P. RECKENDORFER (Mikrochem., 1931, 9, 126–131).—Methods of detection of small quantities of fluorine are reviewed. The procedure recommended for the examination of plants consists in ashing the material in the Berthelot bomb and examining the ash microchemically by the molybdate-benzidine reaction of Feigl and Krumbholz (A., 1929, 783).

E. S. HEDGES.

**Soil analyses and phosphoric acid requirement.** A. NĚMEC (Pflanzenbau, 1930, 4, 229–246; Chem. Zentr., 1931, i, 345).—The determination of citrate-soluble phosphoric acid alone is not a certain means of ascertaining the value of phosphatic fertilisers; colorimetric determination of water-soluble phosphate also gives results of uncertain value. Soils which contain 40–50 (or more) mg. of iron or at least 12 mg. of soluble silica per litre do not give yields responsive to superphosphate fertilisation. A correlation of 96.3% between chemical analysis and field tests is obtainable when the iron and silica contents are considered. The ferric phosphate present in soil approaches  $\text{FePO}_4 \cdot \text{Fe}(\text{OH})_3$  in composition.

A. A. ELDRIDGE.

**Organic soil substance; quantity, and significance as nitrogen reserve.** VINCENT, HERVIAUX, and GAUDIN (Ann. Sci. Agron. Franç., 1930, 47, 654–671; Chem. Zentr., 1931, i, 344).—The yield of alkali-soluble humus depends on the concentration, temperature, and time of extraction. Cold extracts reduce permanganate the most readily. Alkali-insoluble humus contains more organic nitrogen than does alkali-soluble humus.

A. A. ELDRIDGE.

**Field experiments with fertilisers.** XIII. Flax. L. L. BALASHEV. XIV. S. T. ANTOSHIN (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 69, 5–46; No. 71, 17–166).—XIII. Flax takes from the soil only 33% of its



nutrient requirements in the first 45 days of its vegetative period, and continues to absorb nutrients throughout its vegetative life. It is sensitive to increase in concentration of the soil solution. Heavy applications of lime adversely affect the seeds; sodium chloride and phosphorus improves the quality of fibre. Thomas slag is more efficient than acid phosphate.

XIV. A review of experiments with rye, potatoes, oats, lupins, sugar beet, and wheat on chernozem and degraded chernozem soils in the Government of Kiev.

CHEMICAL ABSTRACTS.

**Flax and mineral fertilisers.** A. S. MITROFANOV (Udobr. Urozhai, 1930, 2, 86—93).—A complete fertiliser is preferable. Nitrogen is more effective in combination with phosphorus; heavy applications diminish the yield and quality of the fibre, but potash reduces the injury. At  $p_H$  4.5 heavy application of lime had no effect.

CHEMICAL ABSTRACTS.

**Response of various plants to a complete fertiliser.** F. A. SHWARTZ (Udobr. Urozhai, 1930, 2, 102—104).—Experiments with spring wheat, barley, oats, vetch and oats, flax, and potatoes are described.

CHEMICAL ABSTRACTS.

**Nitrogen availability [of fertilisers].** H. D. HASKINS (Amer. Fertiliser, 1931, 74, [6], 19—24).—The efficiencies of a number of processed, low-grade, organic nitrogen fertilisers are compared in pot cultures. The relative availability of the total and water-insoluble nitrogenous constituents are recorded. In other trials additions of varying quantities of manganese sulphate to soil did not increase the crop yields nor their total nitrogen contents.

A. G. POLLARD.

**Perspective of nitrogen fertilisers in the northern part of Ukrainian chernozem.** S. P. KULZHINSKI (Udobr. Urozhai, 1930, 2, 168—175).—Chile saltpetre is superior to sodium nitrate or ammonium sulphate for potatoes and sugar beet.

CHEMICAL ABSTRACTS.

**Forms and rate of application of nitrogenous fertilisers for oats.** A. L. VELIKOLEPOV (Udobr. Urozhai, 1930, 2, 93—96).—Nitrogen is preferably applied, as sodium nitrate, ammonium sulphate, or calcium cyanamide, with lime, or as ammonium sulphate with lime and phosphorus. High applications of sodium nitrate favour the availability of phosphorus; they also increase the yield of oat straw.

CHEMICAL ABSTRACTS.

**Soil nitrates as a guide to the nitrogen needs of vegetable crops.** H. D. BROWN (Ohio Agric. Exp. Sta. Bimonthly Bull., 1931, No. 149, 55—58).—Changes in the nitrate content of field soils under varying conditions of cropping are satisfactorily determined by a modified diphenylamine test, and utilised to determine the nitrogen requirement of crops.

A. G. POLLARD.

**Pot experiments with phosphates [as fertilisers].** H. D. HASKINS (Amer. Fertiliser, 1931, 74, [8], 20—23).—The availability of numerous phosphatic fertilisers is compared in pot trials with rape. The availability of most fertilisers appeared higher when based on the phosphate removed by the crop, than when total dry-matter yields were considered. Differences in the two series of values were not of the same order for all fertilisers.

A. G. POLLARD.

**Response of winter wheat to phosphorus in the form of raw phosphates.** F. V. CHIRIKOV (Udobr. Urozhai, 1930, 2, 96—98).—On degraded chernozem 90 kg. of  $P_2O_5$  as raw phosphate was equal to 45 kg. as superphosphate.

CHEMICAL ABSTRACTS.

**Can superphosphate increase the solubility of soil potash?** C. DREYSPRING and K. BRINKMANN (Superphosphate, 1931, 4, 69—75).—The increased solubility of potash in soil following applications of superphosphate is due to chemical changes and is attributable to the action of free phosphoric acid and, to a smaller extent, to the gypsum in the fertiliser.

A. G. POLLARD.

**Influence of gypsum on the availability [in soils] of phosphoric acid from raw phosphate.** A. G. MIKHALOVSKI (Udobr. Urozhai, 1930, 2, 99—102).—In pot experiments with millet a double, but not a triple, application of raw phosphate was as effective as a single application of soluble phosphate. Sulphur (2 g. per 3.5 kg. of soil) or gypsum renders raw phosphate soluble.

CHEMICAL ABSTRACTS.

**Causes of the beneficial effect of brown coal on the growth of crops.** II. A. KISSEL (Brennstoff-Chem., 1931, 12, 101—107. Cf. B., 1930, 877).—The fertilising action of brown-coal dust is due in the first place to an improvement in the physical nature of the soil. This applies, however, only to the first year of such cultivation. In the following year a further increased yield is observed due to changes in the chemical composition of the soil. The action of organic fertilisers in modifying the structure of the soil is attributed to the formation of water-soluble humic acids, which are subsequently precipitated as insoluble salts in the fine fissures of the soil. The mechanism of the process is discussed in detail. Experiments on the use of brown coal and preparations containing humins have shown that plants can utilise directly some of the carbon compounds in the fertiliser.

A. B. MANNING.

**Soil fertilisation for sugar beet.** J. TYSON and M. M. MCCOOL (Michigan Agric. Exp. Sta. Spec. Bull., 1930, No. 205, 31 pp.).—Field trials indicate the most satisfactory fertiliser mixtures to contain nitrogen, phosphoric acid, and potash in the ratios 1:4:1 or 1:4:2. On highly organic soils early application of soluble nitrogenous fertilisers is necessary. The use of sodium nitrate just before sowing was as effective as the same amount applied in successive portions in later stages.

A. G. POLLARD.

**Composition of sugar beet in various crop rotations.** A. M. NADEZH DIN (Udobr. Urozhai, 1930, 2, 29—34).—The preceding crop (clover, peas, maize, clean fallow) markedly affects the composition and top: root ratios.

CHEMICAL ABSTRACTS.

**Production of anthocyan in young grain plants and its value as an indication of quality.** G. GASSNER and W. STRAIB (Pflanzenbau, 1930, 4, 169—195; Chem. Zentr., 1931, i, 298).—In respect of the presence of anthocyan the cereals fall into two groups: in barley and oats the coloration is too weak to be used as an indication of quality, whilst rye and wheat possess

a red colour of which the intensity varies with the kind. With rye the comparison is preferably effected in weak illumination.

A. A. ELDRIDGE.

**Manurial experiments with rice.** III. L. LORD (Trop. Agric., 1931, 76, 70—83).—The value of green manures and of various compounded ammonium phosphates is demonstrated.

A. G. POLLARD.

**Effectiveness of various chemical methods in combating insect pests and diseases which attack plants.** G. D. UGRYUMOV and I. I. TRAUT (Udobr. Urozhai, 1930, 2, 45—53).—The relative effectiveness of treatment with various substances of smut, rodents, insects, and soil has been studied.

CHEMICAL ABSTRACTS.

**Incompatibility of lime with fluosilicates [in insecticides].** R. H. CARTER (J. Econ. Entom., 1931, 24, 263—268).—Interaction between slaked lime and the fluosilicates of barium, sodium, and potassium is examined. Small additions of lime reduce the acidity of the fluosilicate solutions. The alkalinity of solutions treated with excess of lime, although considerable, is less than the theoretical value based on the formation of free hydroxides and the precipitation of calcium fluoride.

A. G. POLLARD.

**Physical and chemical properties of commercial arsenical insecticides.** II. **Magnesium arsenate.** F. E. DEARBORN (J. Econ. Entom., 1930, 23, 758—764; cf. B., 1930, 961).—Commercial samples of magnesium arsenate contain a crystalline substance, probably  $Mg_3(AsO_4)_3 \cdot MgO \cdot yH_2O$ . The material is inferior to lead arsenate as an insecticide.

A. G. POLLARD.

**Calcium arsenate as a universal preparation against injurious insects.** N. SAKHAROV (Udobr. Urozhai, 1930, 2, 114—115).—Good results were obtained with mustard and sugar beet.

CHEMICAL ABSTRACTS.

**Nicotine tannate and lead arsenate [insecticides].** R. S. FILMER (J. Econ. Entom., 1931, 24, 277—283).—Nicotine tannate is as toxic as lead arsenate to codling moth, but lacks adhesion and persistence. A loss of 60—70% of nicotine from the tannate spray occurred within 10 days of its application.

A. G. POLLARD.

**Use of manganese in vegetable greenhouses.** I. C. HOFFMAN (Ohio Agric. Exp. Sta. Bimonthly Bull., 1931, No. 149, 58—62).—Cases of manganese chlorosis in greenhouse soils are recorded, together with symptoms, effects, and corrective measures.

A. G. POLLARD.

**Effect of soap on the toxicity of a pyrethrum product known as "Red Arrow."** A. E. BADERTSCHER (J. Econ. Entom., 1931, 24, 268—277).—Dilution of the pyrethrum preparation with water containing 0.4% of the potassium soap of coconut fatty acids increased its efficiency nearly fourfold. The surface tension of the spray fluid was reduced (to approx. 30 dynes) sufficiently to allow easy penetration of the respiratory organs of the insect. No appreciable loss of toxicity in such pyrethrum-soap mixtures occurred during 3 years' storage.

A. G. POLLARD.

**Insecticidal method for determination of kerosene extracts of pyrethrum.** H. H. RICHARDSON (J. Econ.

Entom., 1931, 24, 97—105).—Suitable apparatus and technique are described. The speed of paralysis of insects increases with the concentration of pyrethrum used and is a better criterion of efficiency than the percentage mortality.

A. G. POLLARD.

**Changes in toxicity of rotenone in solution and in suspension.** W. M. DAVIDSON and H. A. JONES (J. Econ. Entom., 1931, 24, 257—262).—During storage there is a loss of toxicity of rotenone in pyridine solution, in acetone solution containing tannic acid, and in aqueous suspensions prepared from fresh acetone or alcohol stock solutions. No loss occurred in pure acetone or alcohol solutions or in the dried material. The yellow decomposition product obtained from pyridine solutions of rotenone was much less toxic to insects and fish.

A. G. POLLARD.

**Preparations containing rotenone as insecticides.** I. **Aqueous suspensions.** H. A. JONES and W. M. DAVIDSON (J. Econ. Entom., 1931, 24, 244—257).—Permanent suspensions containing 0.5—5.0 g. of rotenone per litre are obtained by diluting with water solutions of rotenone in pyridine or acetone to which tannic acid is added as a protective colloid. Alternatively, mixtures of dry (precipitated) rotenone with protective agents (e.g., gums, saponin, or bentonite) may be mixed to a paste with water and subsequently diluted as required. Tannic acid is unsuitable for this method of preparation. Alkaline conditions favour the permanence of rotenone suspensions, but there is risk of decomposition of the material. Permanent suspensions are difficult to prepare with very hard water.

A. G. POLLARD.

**Comparison of rotenone, nicotine, and pyrethrum [insecticides].** M. M. DARLEY (J. Econ. Entom., 1931, 24, 111—115).—In comparative tests with a number of insects, the order of toxicity of rotenone, nicotine, and pyrethrum varied with insect type. A specificity of action of rotenone on red spider is suggested.

A. G. POLLARD.

**Nicotine activators [for insecticides].** E. P. FELT and S. W. BROMLEY (J. Econ. Entom., 1931, 24, 105—111).—Comparative tests with various activators (oils, soaps, etc.) are recorded. The numerous factors affecting the efficiency of nicotine sprays are examined and discussed.

A. G. POLLARD.

**Factors influencing inoculation experiments with *Azotobacter*.** P. L. GAINES (Kansas Agric. Exp. Sta. Tech. Bull., 1930, No. 26, 66 pp.).—The natural distribution of *Azotobacter* in soils is closely associated with, if not actually dependent on, soil reaction. The organisms cannot exist in soils of  $pH < 6.0$ , and the rate of their disappearance from such soils after inoculation increases with increasing acidity. Hydrogen ions appear to act as direct toxic agents towards the organisms in acid soils.

A. G. POLLARD.

**Organic solvents for aiding the removal of spray residue from waxy or oil-covered fruit.** R. H. ROBINSON (J. Econ. Entom., 1931, 24, 119—125).—Addition of alcohol, benzene, acetone, or kerosene to the hydrochloric acid solution used for removing arsenical residues from sprayed fruit increased its efficiency. The

use of kerosene is the most practicable, a 2% mixture causing no injury to the fruit. A. G. POLLARD.

**Tolerance of beans to sprays and dusts for the Mexican bean beetle.** H. C. HUCKETT (J. Econ. Entom., 1931, 24, 200—204).—Among arsenical sprays examined, magnesium arsenate was far less injurious to plants than were the lead or calcium compounds. Admixture with Bordeaux mixture reduced the injurious properties of arsenate sprays. Barium fluosilicate compared favourably with magnesium arsenate. In dusting mixtures similar results were obtained. Cryolite was inferior to barium fluosilicate. Use of copper-lime preparations as diluents for dry sprays was as satisfactory as hydrated lime, except in the case of magnesium arsenate. A. G. POLLARD.

**Effect of summer oil sprays on apple trees.** J. P. GINSBURG (J. Econ. Entom., 1931, 24, 283—290).—Of numerous oils examined, sperm oil was the least injurious to apple and peach foliage. A. G. POLLARD.

**New spray procedures [for apples].** P. J. PARROTT F. Z. HARTZELL, H. GLASGOW, and S. W. HARMAN (J. Econ. Entom., 1931, 24, 297—302).—A number of modifications in spraying practice are suggested. A. G. POLLARD.

**[Agricultural] spray-residue removal.** L. R. STREETER, P. J. CHAPMAN, and G. W. PIERCE (J. Econ. Entom., 1931, 24, 240—244).—Current processes are discussed. The use of dilute hydrochloric acid is generally satisfactory, but there is need for a machine combining washing and wiping operations. A. G. POLLARD.

**Formation and decomposition of humus in stable manure and soil.** W. SAUERLANDT (Pflanzenbau, 1929, 2, 434—471; Chem. Zentr., 1931, i, 344).

**Fleece analysis.**—See V. **Phosphates [as fertilisers].** Calcium arsenate.—See VII. **Derris root.** Pyrethrum extract.—See XX.

See also A., April, 459, **Mineralisation of humus nitrogen in low-moor soils.** 460, **[Armenian] soils.** 533, **Nitrogen relation in the soya bean.** Transport in the cotton plant. 537, **Lodging of cereals.**

#### PATENTS.

**Manufacture of fertilisers.** SOC. D'ETUDES SCIENTIF. ET D'ENTERPRISES INDUSTRI., Assees. of E. VOITURON (B.P. 342,931, 8.3.30. Ger., 9.3.29).—The mother-liquor containing sodium and potassium chlorides from a previous batch in the process is treated successively with phosphoric acid and ammonia (1:2 mols.), so that microcosmic salt crystallises on cooling, and with a solution containing sodium and potassium chlorides (sylvinite), to precipitate, on further cooling, a mixture of potassium and ammonium chlorides ("potazote"). The crystals are removed separately at each stage. L. A. COLES.

**Production of nitrogenous compounds from substances of vegetable origin.** H. WADE. From N.V. MAATS. TOT EXPLOITATIE VAN VEREDELINGS-PROCEDES (B.P. 342,758, 5.11.29).—Material containing little or no nitrogen is heated with aqueous ammonia under pressure; e.g., wood sugar is heated at 120—150°

under 2—5 atm. to yield a partial substitute for albuminous fodder, or insoluble material (cellulose, lignin, peat, coal, etc.) is heated at 150—340°/5—150 atm.

L. A. COLES.

**Cellulosic pulp.**—See V. **Calcium nitrate double salts.**—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Behaviour of "sugar-soluble" silicic acid in pure sugar solutions and beet juices of different alkali-alkalinities (natural alkalinities).** O. SPENGLER and A. TRÄGEL (Z. Ver. deut. Zucker-Ind., 1930, 80, 847—852; cf. B., 1930, 260).—15% Sucrose solutions containing sodium hydroxide equivalent to 0.01, 0.03, 0.06, 0.08, and 0.1% CaO, heated for 10 min. at 80—85° with 2% of lime containing "sugar-soluble" silica (yielding 9 mg. SiO<sub>2</sub> per 100 c.c. by the method described in B., 1930, 860), and then carbonated to an alkalinity of 0.07—0.09% CaO and filtered, contained 0.2, 0.4, 0.7, 4.2, and 10 mg. SiO<sub>2</sub>, respectively, per 100 c.c. After further treatment with 0.5% of similar lime, and carbonation to an alkalinity of 0.015% CaO, the filtered solutions contained 0.5, 3.2, 5.0, 6.0, and 10 mg. SiO<sub>2</sub> per 100 c.c. Similar liming and carbonation applied to a beet juice of practical natural alkalinity equivalent to 0.031% CaO (cf. Spengler and Brendel, B., 1928, 421) yielded first-carbonation juice containing 2 mg. SiO<sub>2</sub> per 100 c.c., and second-carbonation juice containing 8 mg. SiO<sub>2</sub> per 100 c.c. and 0.007% total CaO. By raising the natural alkalinity of the original juice to 0.053% CaO by addition of sodium hydroxide, and by lowering it to 0.012% by addition of hydrochloric acid, final juices were obtained from the second carbonation, containing in the former case 0.001% total CaO and 9 mg. SiO<sub>2</sub> per 100 c.c., and in the latter case only 1 mg. SiO<sub>2</sub> per 100 c.c., but 0.03% of total lime. Juices which owe most of their alkalinity to lime, as is the case after the first carbonation, retain much less silica in solution than those in which the alkalinity is mainly due to alkali carbonates, as should be the case after the second carbonation. To eliminate calcium salts as completely as possible without introducing harmful amounts of silicic acid into the juice, it is necessary to employ lime free from "sugar-soluble" silica, especially prior to the second carbonation. The proportion of "sugar-soluble" silica in lime is independent of the temperature employed in the lime kilns. J. H. LANE.

**Decomposition of alkaline sugar solutions at high temperatures.** I. O. SPENGLER, E. LANDT, and J. OST (Z. Ver. deut. Zucker-Ind., 1930, 80, 751—770).—An investigation of the coloration, and fall in polarisation and alkalinity, of 15, 30, and 50% sucrose solutions containing 0.15, 0.25, 0.4, and 0.5% of lime, when heated in a closed iron vessel at precisely controlled temperatures of 110°, 120°, 130°, and 140° for periods of 20—90 min. For any given solution at constant temperature the fall in polarisation was roughly proportional to the time. The alkalinity also diminished and many of the solutions became neutral or even acid within the maximum experimental period; in these cases the rate of development of colour increased notably

as neutrality was attained, but beyond this stage the solutions became lighter again. For solutions of given sugar content, at a given temperature, the higher the initial alkalinity, the more rapid was the fall in polarisation and in alkalinity; the rate of coloration was almost independent of the alkalinity so long as the solutions remained decidedly alkaline, but the colour maximum associated with the attainment of neutrality occurred later and was the more intense the higher the initial alkalinity. For a given initial alkalinity and a given temperature, solutions of high sugar content darkened more rapidly and suffered a somewhat greater fall in polarisation and alkalinity in a given time than more dilute solutions; the percentage of the total sugar destroyed in a given time was apparently greater in dilute than in concentrated solutions. For solutions of given initial alkalinity and sugar content, at different temperatures within the range investigated, a rise of  $10^{\circ}$  was found to treble the rates of fall of polarisation and alkalinity, and also the rate of coloration so long as the solutions remained alkaline. J. H. LANE.

**Precipitation of lime by sulphurous acid in saccharine solutions.** E. SAILLARD (Compt. rend., 1931, 192, 178—180).—When purification of sugar-beet juice is effected by combined carbonatation and sulphitation, sulphur dioxide is passed into a solution containing the free hydroxides of calcium, potassium, and sodium. To determine the course of the resulting reaction, sulphur dioxide was passed into solutions containing varying amounts of sugar and of calcium and sodium hydroxides. Under these circumstances both calcium and sodium sulphites are formed in spite of the very slight solubility of the former. Increase in the proportion of sodium hydroxide causes increase in that of sodium sulphite; with equal equivalents of the two hydroxides increase in the amount of either sugar or sulphur dioxide causes increased formation of calcium sulphite. C. A. SILBERRAD.

**Calcium carbonate hydrate [in sugar carbonatation].** V. V. YANOVSKI and G. M. BUNICH (J. Appl. Chem., Russia, 1930, 4, 265—273).—If a carbonated sugar-lime solution, after removal of excess of carbon dioxide, is kept in a stoppered vessel at  $10^{\circ}$  for 20—30 hrs., calcium carbonate hexahydrate, monoclinic,  $d$  1.808, separates; it is readily converted into (probably) vaterite,  $d$  2.6205. Irregularities in sugar manufacture are probably due to improper saturation preventing conversion into the insoluble form of calcium carbonate.

CHEMICAL ABSTRACTS.

**Catalytic action in the coloration of alkaline sugar solutions on heating.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1930, 80, 673—689).—Solutions containing 100 g. of sucrose, 2 g. of sodium carbonate, and 1 mg. of ferrous sulphate in 100 g. of water, heated for 2 hrs. in a brine bath, developed nearly twice as much colour as in absence of the iron salt. The further increase of colour with 10 mg. was less than that due to the first mg. The colour developed in absence of added iron was probably due to traces of this metal in the original materials; it was found to be considerably less in presence of 1 mg. of potassium cyanide, which is known to inhibit the catalytic action of

iron, and also in presence of 1—2 g. of sodium sulphite or pyrophosphate. The decomposition of sucrose on prolonged heating in alkaline solutions, as measured by fall in polarisation, is more or less independent of the development of colour (cf. B., 1930, 259), but it also is probably of catalytic nature, judging by the effect of potassium cyanide, although the data on this point are not conclusive. J. H. LANE.

**Relation between the ash content of raw [beet] sugars and the electric conductivity of their solutions at different concentrations and in presence of acid.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1930, 80, 853—865).—Ash determinations on 60 samples of German raw beet sugars confirmed the authors' previous conclusion (B., 1928, 422) that, in the electrometric method, 5% solutions give much more uniform agreement with the incineration method than is given by 26% solutions. Comparison of the conductivity of 70 samples, in 5% solutions, with the increase of conductivity produced by addition of acid, as proposed by Zerban, showed such irregularities as to exclude the application to German sugars of a correction formula like that found satisfactory with American sugars. J. H. LANE.

**Viscosity of sugar solutions.** O. SPENGLER and E. LANDT (Z. Ver. deut. Zucker-Ind., 1930, 80, 523—547).—The principles of viscosimetry are explained and several types of viscosimeters described. Data on the viscosity of pure sucrose solutions by previous workers are reproduced, and new determinations by means of the Vogel-Ossag capillary viscosimeter are described. Results for solutions of 0—70° Brix at  $20^{\circ}$  agree well with those obtained by Bennett and Nees (B., 1930, 260). Determinations were also made of the densities and viscosities of solutions obtained by dissolving 2.5, 5, and 10% of various salts in saturated (67%) sucrose solution. The viscosity of the latter was greatly increased by alkali hydroxides and carbonates, much less affected by neutral salts, and considerably reduced by ammonium chloride. J. H. LANE.

**Storage of raw [beet] sugars with special reference to affining qualities.** O. SPENGLER and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1930, 80, 690—709).—Samples of two raw beet sugars (polarisation 95.3 and 97.19, moisture 1.70 and 1.20%) were exposed for weeks to air of constant relative humidities, 30, 50, 70, and 90%, at constant temperatures of  $15^{\circ}$  and  $25^{\circ}$ . It is concluded that raw beet sugars containing up to 2% of moisture will retain their moisture content and affining qualities substantially unchanged at any ordinary temperature, provided the R.H. of the air is maintained between 50 and 60% of saturation. In air of lower R.H. they will lose moisture and yield darker sugars on affination, whilst in moister air they will absorb moisture and yield lighter affination products. Both the sugars investigated suffered an appreciable fall in alkalinity to phenolphthalein within 3 weeks in presence of air having R.H. in excess of 60%, and under the same conditions invert sugar was formed in one of them. The optimum R.H. for the storage of white sugar was found to be about 60%. J. H. LANE.

**Polyfructoses and their determination. II. Determination of trifructose anhydride for the evaluation of the rye-meal content of flours.** C. I. KRUISHEER (Rec. trav. chim., 1931, 50, 153—163; cf. B., 1930, 1043).—Trifructose anhydride (trifructosan) and sucrose may be determined in cereals by the following method. The meal (12.5 g.) is shaken with 50 c.c. of 70% alcohol for 1 hr., filtered, and 25 c.c. of the filtrate are mixed with 5 c.c. of *N*-sodium hydroxide in 70% alcohol. After 1 day the mixture is centrifuged, the supernatant liquor decanted, the precipitate washed twice with 2 c.c. of 70% alcohol, dissolved in 10 c.c. of warm water, and neutralised with 0.25*N*-sulphuric acid (methyl-orange). The solution is hydrolysed with 15 c.c. of water and 2.5 c.c. of 9.5*N*-hydrochloric acid for 10 min. at 68—70°, cooled, neutralised, and diluted to 50 c.c. In 10 c.c. of this solution the total reducing power is determined by School's method (B., 1929, 336) and calculated as percentage of the original substance ( $R_2$ ). For determination of the fructose content 20 c.c. of the invert solution are treated with 2.5 c.c. of 4*N*-sodium hydroxide and 8 c.c. of iodine solution in the manner previously described (B., 1929, 533), but only half the quantities are used in this case; the solution is diluted to 50 c.c., the reducing power determined in 25 c.c., and the percentage ( $F_2$ ) of fructose in the original substance calculated. The trifructose anhydride content ( $T$ ) is then calculated from the equation  $T = 0.90(2F_2 - R_2)$ . By this method the trifructose anhydride content of samples of bolted rye flour varies from 1.5 to 2.0%, and in wheat flour from 0.1 to 0.3%. The accuracy of the method for the determination of rye-flour contamination in wheat flour does not exceed 10%, owing to the varying content of trifructose anhydride in various samples of each flour. Examination of unripe cereals confirms the view that unripe wheat grains have a higher polyfructose content. J. W. BAKER.

**Viscosimeter.**—See I. **Sugar-beet soils.**—See XVI. **[Sugars from] rice straw.**—See XVIII. **Dextrose in milk.**—See XIX.

See also A., April, 422, **Decomposition of starch and dextrin.** 522, **Selective fermentation of dextrose-lævulose mixtures.** 535, **Gum tragacanth.** 538, **Volumetric determination of sugars.**

#### PATENT.

**Nitrogenous substances.**—See XVI.

### XVIII.—FERMENTATION INDUSTRIES.

**Constituents of dried yeast and yeast extracts. Application in human nutrition.** S. G. WILLIMOTT and F. WOKES (Lancet, 1928, 215, 668—673).—Any deficiency of vitamin-*B* in white flour can be remedied by the correct addition of dried yeast. About 80% of the vitamin-*B* content of fresh yeast can be extracted by heating at 98° with a saline solution for several hours. The addition of dried yeast to bread is recommended, an amount equivalent to 2—4% of the flour being sufficient to furnish more vitamin-*B* than is present in whole-wheat flour. Potato starch may contain sufficient vitamin-*B* to vitiate an assay; rice starch appears

to be free from this contamination. Data of the percentage composition and vitamin-*B* content of various yeast products are tabulated. L. S. THEOBALD.

**Fermentation of rice straw by *B. acetoethylicus*.** V. N. PATWARDHAN (J. Indian Chem. Soc., 1930, 7, 531—536).—The yields of reducing sugar obtained by hydrolysis of Bangalore rice straw are proportionately increased by increase of the concentration of the sulphuric acid used from 2% up to at least 6%, these results being at variance with those of Thaysen ("Fuel for Motor Transport," H.M. Stationery Office, 1927) for Burma rice straw. *B. acetoethylicus* may be trained to ferment pentoses (such as are obtained by processes similar to the above) at concentrations up to 7%, but the yields of alcohol and acetone, which are somewhat variable (cf. Thaysen and Galloway, B., 1929, 272), generally decrease continuously as the concentrations of sugar are increased above 3%. Change of the nutrient mixture from ammonium phosphate and peptone to combinations of ammonium nitrate, disodium hydrogen phosphate, and yeast water does not materially affect the yields. The presence of small quantities of hexoses, e.g., dextrose, facilitates the growth of the bacterium and fermentation of the pentoses. H. A. PIGGOTT.

**Extractives of whiskey.** W. PARTRIDGE (Analyst, 1931, 56, 177—178).—Of 44 samples examined, the total solids of 29 exceeded 0.15% wt./vol. (the limit usually mentioned in the literature), 4 samples of which gave 0.41—0.53%. Sherry appears to be a safe and profitable adulterant. T. MCLACHLAN.

**Treatment of wine with ozonised air.** P. MARSAIS (Rev. Viticult., 1930, 73, 311—314; Chem. Zentr., 1931, i, 375).—The alcohol, volatile acid, and non-volatile acid content was scarcely affected; the sulphur dioxide diminished. White wine gave a tannin-iron precipitate. The effect of the treatment on the taste is described. A. A. ELDRIDGE.

**Addition of ammonium salts to vinegar.** C. A. MITCHELL (Analyst, 1931, 56, 178—179).—These salts are added to spurious vinegars to give the nitrogen figure obtained from a normal malt vinegar. Less than one tenth of the total nitrogen should be present as ammoniacal nitrogen, whilst one fifth to one tenth should be precipitable on saturation with zinc sulphate. T. MCLACHLAN.

**Sterilisation of enzymes. Ferments in honey.**—See XIX.

See also A., April, 522, **Selective fermentation of dextrose-lævulose mixtures.** **Rôle of iron and copper in growth and metabolism of yeast.**

#### PATENT.

**Water-soluble vitamin.**—See XIX.

### XIX.—FOODS.

**Sterilisation of flour and of enzymes in powder form.** A. J. J. VANDE VELDE (Bull. Acad. roy. Belg., 1930, [v], 16, 585—591).—The carbon disulphide treatment previously developed for the sterilisation of flour (cf. B., 1910, 1129) has been applied to amylase and

pepsinase. By treating with pure carbon disulphide in Petri dishes at ordinary temperatures and subsequently removing the last traces by a current of dry sterile air, the enzymes were obtained in a sterile form without alteration to their hydrolytic powers. H. J. DOWDEN.

**Determination of starch in flour by diastase-acid hydrolysis.** B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1931, 14, 112—116).—Modifications have been made in the official A.O.A.C. diastase-acid hydrolysis method (cf. B., 1927, 90). Sugar and fat are extracted by solvents, followed by centrifuging; the residue is submitted to pepsin digestion in acid solution. It is neutralised, treated with malt extract for 20 min. at 65°, and then hydrolysed with hydrochloric acid; the sugar is determined in the normal manner. E. B. HUGHES.

**Baking quality of wheat.** F. SCHNELLE (Pflanzenbau, 1929, 1, 471—555; Chem. Zentr., 1931, i, 377).—Comparative tests with 33 varieties were made, the results being correlated with the season, time of ripening, geographical source, and nitrogen content.

A. A. ELDRIDGE.

**Rôle of milk constituents in breadmaking.** L. A. ALLEN and J. BELL (J. Roy. Tech. Coll., Glasgow, 1931, 2, 550—563).—Experiments were made on the use of separated milk, dried and separated milk, whey, and albumin-free whey in bread dough (4 lb. of strong spring and 1.25 lb. of soft winter wheat flours, sugar 0.5 oz.; lard 0.5 oz., salt 1.5 oz., yeast 2 oz., water 1500 c.c.). The increase in volume during fermentation was stimulated by albumin-free whey, but was slightly depressed by separated milk, and to a greater extent by whey. The theory that albumin is a determining and adverse factor in volume increase was confirmed from the depressant effect obtained on addition of a suspension of egg-albumin to an albumin-free control dough, and from the appreciable increase in volume obtained if the albumin was coagulated by boiling the milk under reflux for 15 min. before mixing. Dried (pressure-spray system) separated milk lowers the loaf volume, unless it has been preheated, when the effect is largely counter-balanced; lactose or rennin has no influence, but small amounts of lactic acid effect an improvement. Whey, boiled whey, and albumin-free whey increase, and raw or boiled separated milk decrease, the rate of gas evolution. This may be explained in terms of a balance between the stimulating effects of phosphates and the inhibitory effects of albumin (*vide infra*) and of casein. In all cases the colour, texture, and flavour of the final loaf were improved by addition of whole milk, separated milk, or whey, the first being superior in this respect. The increase in volume of fermenting dough is best measured by placing the standard whisked dough (strong spring wheat flour 225 g., yeast 22.5 g., salt 9 g., water 250 c.c.) in graduated cylinders immersed in water at 29°.

J. GRANT.

**Direct determination of available carbon dioxide in baking powder.** M. R. COE (J. Assoc. Off. Agric. Chem., 1931, 14, 99—101).—The A.O.A.C. gasometric method (*ibid.*, 1923, 6, 453) is modified to give a direct determination of the available carbon dioxide by using a 5% solution of ammonium sulphate as the reacting

liquid, heating the reaction flask to boiling to evolve all gas, and then cooling it to room temperature.

E. B. HUGHES.

**Dispersoid-chemical study of milk. III. Separation of cream.** A. SCHNECK [with E. MUTH] (Milchwirt. Forsch., 1930, 10, 1—29; Chem. Zentr., 1930, ii, 1154; cf. A., 1930, 1204).—The mechanism of the process is discussed. The speed diminishes at first quickly and then slowly; it is unaffected by movement of the milk, and hence by agglomeration of the fat. The separability of milk heated at 61° is greater than that of unheated milk; the effect of various temperatures was examined.

A. A. ELDRIDGE.

**Small-scale research on spray-drying of milk.** A. W. SCOTT (J. Roy. Tech. Coll., Glasgow, 1931, 2, 456—460).—The engineering problems associated with spray-drying of milk are briefly reviewed, and a detailed description is given of an experimental plant. The principal feature is the low-pressure atomiser in which the milk under a pressure of about 4 lb./in.<sup>2</sup> issues vertically upwards from a nozzle 0.005 in. in diam. in an annular chamber, while air at 4—6 lb./in.<sup>2</sup> is directed by means of a curved cap so as to impinge tangentially on the issuing jet. The atomised milk meets a downward stream of hot air in a chamber of tinned sheeting, and passes out at the bottom through filter-bags which collect the milk, recondensation of water vapour being avoided by ensuring an outlet temperature of at least 55°. Separated milk gave a fine powder with an average solubility of 99.5%.

J. GRANT.

**Determination of milk proteins. III. Proposed modified method for casein.** G. M. MOIR (Analyst, 1931, 56, 147—149; cf. B., 1931, 413).—The casein is precipitated from warm diluted milk at  $p_H$  4.6 by the successive addition of acetic acid and sodium acetate, filtered, and the nitrogen determined by the Kjeldahl method. The result is returned as casein-nitrogen on account of uncertainty of the structure of the protein molecule.

T. McLACHLAN.

**Determination of the f.p. of milk.** A. HEIDUSCHKA and A. KERN (Milchwirt. Forsch., 1930, 10, 165—172; Chem. Zentr., 1930, ii, 1153).—Requirements for unanimity are discussed. Watering of milk is more readily detected by means of the f.p. than by means of the density of the serum.

A. A. ELDRIDGE.

**Surface tension of milk.** W. MOHR and C. BROCKMANN (Milchwirt. Forsch., 1930, 10, 72—96; Chem. Zentr., 1930, ii, 1153).—Semi-dynamic (drop method) values are always higher than, but generally parallel with, static values; the surface tension falls with increasing fat content, owing to concentration of capillary active substances at the fat globules. The depression differs for solid and liquid fats. Structural phenomena and film formation account for anomalies with skim milk. Pasteurisation, boiling, sterilising, ageing, freezing, and addition of formaldehyde are practically without effect on the surface tension. Increased air content increases and souring diminishes the value.

A. A. ELDRIDGE.

**Indications of dextrose in milk.** C. H. WHITNAH (J. Amer. Chem. Soc., 1931, 53, 300—304).—Small amounts of dextrose in milk are determined by clarifying

the milk with mercuric nitrate, measuring the rotatory power of the solution, removing the dextrose by fermentation with yeast, and again measuring the rotatory power. Application of the method to normal milk indicates the presence of 0—0.35% of dextrose.

H. BURTON.

**Microscopical examination of milk for tubercle bacilli.** D. R. WOOD (Analyst, 1931, 56, 179—180).—From a simple calculation it is shown that, without concentration, the test is about 500 times less sensitive than the biological test, and is limited to the detection of not less than 10—500 per c.c., or, in the case of milks with less than 2.5 c.c. deposit per 100 c.c., 10—50 bacilli per c.c.

T. McLACHLAN.

**Vitamin content of margarine.** K. H. COWARD (Lancet, 1928, 215, 726—727).—Data showing the vitamin content of "Viking," "Silver Tray," "Welcome," and "Gold-Chain" margarine are recorded. The margarines are equal to best summer butter in their vitamin-A and -D contents and show more constant values than the latter. The vitamin-D content averaged 1.25 units per g.

L. S. THEOBALD.

**Vitamin-C content of commercially canned sauerkraut; observations on its vitamin-A content.** B. CLOW, H. T. PARSONS, and I. STEVENSON (J. Agric. Res., 1930, 41, 51—64).—The vitamin-C content of six commercial brands of sauerkraut varied within wide limits, protection against scurvy of guinea-pigs being afforded at levels of 2½, 5, 7½ g. and over of the various makes. A satisfactory technique for the determination of vitamin-A was not achieved.

E. HOLMES.

**Use of acetaldehyde in the storage of fruit.** S. A. TROUT and R. G. TOMKINS (J. Coun. Sci. Ind. Res., Australia, 1931, 4, 6—11).—The sound condition of stored fruit may be prolonged in an atmosphere containing small proportions (1 in 250—1000 vols.) of acetaldehyde. The margin of safety between concentrations necessary to prevent mould growth and those causing injury to fruit are small. Dipping the fruit in solutions of acetaldehyde was unsuccessful.

A. G. POLLARD.

**Corrosion by fruit preserves.** H. SERGER (Konserven-Ind., 1930, 17, 621—622; Chem. Zentr., 1931, i, 540).—The *P.D. (b)* at 22° between a tantalum dish containing an infusion of the fruit and a zinc plate, and that (*a*) using 0.1*N*-oxalic acid are measured; then  $d = 10b/a$ . Values of *d*, the corrosiveness, ranged from 3.93 to 14.43.

A. A. ELDRIDGE.

**Determination of pectins.** A. MENLITZ (Konserven-Ind., 1930, 17, 624—626, 640—645, 654—657, 671—673; Chem. Zentr., 1931, i, 378).—In the determination of methoxyl by Zeisel's method the error is 2.5%, by Fellenberg's method 2.8%, and by a simplification of the latter 4.6%. For Lüers and Lochmüller's breaking test the error is 10%. Fiedler's method at  $p_H$  3.0 and nearly equal pectin concentration gives reproducible values. The ash, as well as the methoxyl, content should be determined. The amount of calcium pectate in the precipitate obtained with alcohol characterises the origin (apple 60—70%, citrus 90—95%). In Lüers and

Lochmüller's tension test mercury is used instead of shot.

A. A. ELDRIDGE.

**Behaviour of the diastatic ferments in honey on heating.** H. W. DE BOER (Chem. Weekblad, 1930, 27, 646—648).—No destruction of the ferments is observed below 60°. The number falls by one unit for every 7 hrs. heating at 65°, for every 4—5 hrs. at 70°, for every 2—2½ hrs. at 75°, for every 10 min. at 80°, and for every 45 sec. at 95°. The rate of fall diminishes with time, the last traces disappearing only slowly.

S. I. LEVY.

**Colour of meat.** A. A. BENEDICT (Proc. Iowa Acad. Sci., 1929, 36, 301—302).—Change in the colour of meat is prevented by placing the sample between glass plates immediately after cutting. The intensities of the light diffusely reflected from various cuts were compared spectrophotometrically in the region 500—700 mμ.

CHEMICAL ABSTRACTS.

**Removal of the astringent taste of coffee by treatment of the raw beans with ozone *in vacuo*.** H. JORDT (Chem.-Ztg., 1931, 55, 161).—The raw beans are placed in a chamber which is evacuated to 10 mm., and ozonised air saturated with moisture is then introduced until the inner and outer pressures are the same. In this way the ozone is able to penetrate through the cellular tissue to the interior of the beans; a pretreatment with a suitable solvent to remove the external wax layer is also advantageous. After several hours' treatment the beans are removed from the chamber and roasted as usual.

F. R. ENNOS.

**Parchment packing.**—See V. Solubilities of metals in milk.—See X. Determination of fat. Indian ghee.—See XII. Anthocyan [in cereals].—See XVI. Rye meal in flour.—See XVII. Dried yeast etc.—See XVIII.

See also A., April, 498, Trigonelline [from coffee beans]. 508, Determination of sterol and ergosterol in cow's milk. 529—531, Vitamins. 531, Antiscorbutic potency of apples. 537, Proteins of Indian foodstuffs.

PATENTS.

**Treatment of cereal flour or dough.** BRIT. ARKADY CO., LTD., and H. HEWITT (B.P. 342,697 and 343,677, [A] 2.11.29, [B] 7.1.30).—(A) Leguminous flour is added to the dough in the proportion of 2% or less of the wheat flour, and the exposure of the dough to the air during mixing causes subsequent bleaching. Persulphates, bromates, iodates, or organic peroxides may be added. (B) The leguminous flour is added as an extract prepared by soaking in water, filtering, and concentrating the filtrate *in vacuo* at 50°.

E. B. HUGHES.

**Preparation of bread.** G. CHAUDET (B.P. 342,657, 29.10.29. Switz., 10.6.29).—A solution of iron and calcium bicarbonates, calcium and sodium chlorides, magnesia, and lactic acid in mineral water is added to the dough to replace the yeast wholly or in part.

H. ROYAL-DAWSON.

**Baking powder.** A. H. FISKE, Assr. to RUMFORD CHEM. WORKS (U.S.P. 1,775,037, 2.9.30. Appl., 2.3.28).—Improvers, of the type used in flour for bread-making,



including persulphates, perborates, and peroxides, are added to baking powder and the proportion of the acidic reagent is increased so as to leave a neutral residue after reaction is complete. E. B. HUGHES.

**Electrothermal treatment of cheese.** C. H. PARSONS and W. D. RICHARDSON, Assrs. to SWIFT & Co. (U.S.P. 1,774,610, 2.9.30. Appl., 29.6.27).—Emulsified cheese is pasteurised by forcing the product between suitably arranged electrodes in a cylindrical vessel. It is claimed that uniform heating is thus obtained.

E. B. HUGHES.

**Manufacture or treatment of coffee.** W. E. CLIFTON (B.P. 342,778, 8.11.29).—The sealing of the volatile constituents and essential oils of coffee is achieved by grinding the beans in an edible, water-soluble, viscous liquid (*e.g.*, sugar solutions, condensed milk). An apparatus is described. E. B. HUGHES.

**Concentrating the water-soluble vitamin of a material containing such vitamin.** A. B. O. NORR-BIN, and AKTIEB. ASTRA APOTEKARNAS KEMISKA FABR. (B.P. 343,086, 13.11.29).—In a dilute acid extract of a vitamin-containing material, *e.g.*, yeast, is dissolved at least one substance which, on neutralisation of the solution, forms a compound which is precipitated, carrying the vitamin with it. *E.g.*, calcium carbonate is dissolved in a dilute acetic acid extract of yeast and a sugar is added to the solution. On neutralisation with alkali, the sugar-calcium compound is precipitated; this is separated and stirred with sufficient dilute sulphuric acid to transform all the calcium into calcium sulphate. Alcohol is then added to the mixture and the dilute alcoholic extract is separated and concentrated to give an emulsion which contains the vitamin.

E. H. SHARPLES.

**Freezing of food products.** A. E. WHITE. From FROSTED FOODS CO., INC. (B.P. 345,618, 18.12.29).

**Preparing animal meats for the market.** W. W. TRIGGS. From SWIFT & Co. (B.P. 345,328—9, 16.12.29).

**Nitrosylsulphuric acid.**—See VII. **Nitrogenous substances.**—See XVI. **Vitamin-D.**—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Genuine *Derris* root containing no rotenone.** R. C. ROARK (J. Econ. Entom., 1931, 24, 329—330).—Analyses of numerous samples of *Derris* root show rotenone contents ranging from 0 to 5.5% and total ether extracts of 5—23%. No relationship exists between the two values and a sample containing 23% of ether extract and no rotenone is recorded.

A. G. POLLARD.

**Determination of the active principles of pyrethrum extract.** H. TATU (Parfumerie Mod., 1930, 54, 607—615; Chem. Zentr., 1930, ii, 3840).—Evaluation by means of Fehling's solution is unsuitable for commercial tests, since the colour change is uncertain, small variations in procedure affect the results, and constancy of the relation between reducing power and toxicity has not been established. Gravimetric determination of pyrethrin as semicarbazone is difficult. Only

a determination of relative toxicity gives a measure of the value. A. A. ELDRIDGE.

**Evaluation of cantharides.** J. O. RIV (Anal. Fis. Quim., 1931, 29, 164—169).—For the determination of cantharidin in cantharides the powdered material (7.5 g.) is extracted with 30 c.c. of acetone and 0.6 g. of hydrochloric acid for 2 hrs., and the filtered and evaporated extract is washed four times with a mixture of 10 c.c. of light petroleum, saturated with hydrochloric acid and cantharidin, with 6 c.c. of ethyl alcohol. Further purification may be effected by recrystallisation from acetone and repeated washing. The product has m.p. 216°. Specimens of cantharides derived from *Lytta vesicatoria*, *Zonabris phalerata*, and *Epicauta dubia*, from various localities, contained from 0.060 to 1.825% of cantharidin. Critical comparison of various methods for the determination shows that, of the official methods, only those of the German and Russian Pharmacopœias yield concordant and satisfactory results.

H. F. GILLBE.

**Chrysophanic acid, chrysarobin, and sulphur.** R. HUERRE (J. Pharm. Chim., 1930, [viii], 12, 145—149).—To detect chrysarobin in chrysophanic acid 0.25—0.30 g. of the product is heated gently with 0.1 g. of sulphur until evolution of gas begins and for a few seconds longer. In the presence of 15% or more of chrysarobin a green coloration is produced. Addition of chloroform gives an intensely green solution. The reaction is given by the fractions of chrysarobin soluble and insoluble in potash solutions of at least 2% concentration, but not by the fraction soluble in 1% potash and reprecipitated from its potash solution by addition of sulphuric acid. The test is applicable to chrysarobin pomades.

W. J. BOYD.

**Oil of *Eucalyptus dives*.** K. STEPHAN and M. DÜKER (J. pr. Chem., 1931, [ii], 129, 145—150).—Two samples of the oil ( $d_{20}^{20}$  0.9194, 0.8936;  $n_D^{20}$  1.4858, 1.4808;  $\alpha$  -41.44°, -56.8°) were mixed and the unsaponifiable matter (90%) was separated and fractionally distilled under reduced pressure. From the oxygen-containing fractions, b.p. 72—210°/12—14 mm., piperitone, terpinen-4-ol, and an acid, probably  $C_{18}H_{34}O_3$ , m.p. 109—112°, were isolated. The piperitone, readily separated as its compound with sodium hydrogen sulphite, was readily oxidised to thymol by ferric chloride and aqueous acetic acid. Other oxidising agents, with the exception of potassium permanganate in neutral solution which gave diosphenol, m.p. 81°, gave negative results.

H. A. PIGGOTT.

**Japanese peppermint oils.** V, VI. **Constituents of the high-boiling fractions I, II.** Y. SHINOSAKI and T. NAGASAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 420 B, 421 B; cf. B., 1930, 264).—The high-boiling fractions consist mainly of esters and other compounds boiling above 240° and possessing a characteristic odour. I. The following alcohols were recovered:  $\Delta^8$ -hexenol ( $\alpha$ -naphthylurethane, m.p. 70—71°; silver phthalic ester, m.p. 125—126°) yielding *n*-hexyl alcohol on catalytic hydrogenation (the acetate absorbed 1—2 mols. of hydrogen); and two sesquiterpene alcohols,  $C_{15}H_{26}O$ , having, respectively, b.p./4 mm. 129—130°, 134—136°;

$d_{4}^{20}$  0.9716, 0.9726;  $n_D^{20}$  1.5070, 1.5079;  $\alpha_D^{15}$   $-4.0^\circ$ ,  $+5.22^\circ$ .

II. Fractionation of the methyl esters of the acids yielded formic, acetic, isovaleric, *n*-hexoic, pelargonic (?), and (probably) higher saturated acids.

E. LEWKOWITSCH.

Tolu balsam.—See XIII.

See also A., April, 424, Colloidal lead phosphate. 428, Sol and coacervate of ichthyocoll. 479, *iso*-Eugenol from clove oil. 483, Local anæsthetics. 487, Synthesis of antiseptic derivatives of indan-1:3-dione. Essential oil of *Backhousia angustifolia*. 492, Linalool from coriander oil. 498, Alkaloids of *Anabasis aphylla*. 498—9, Nicotine and its derivatives. Lupin alkaloids. 500, *Strychnos* alkaloids. 501, Codeine. Phenarsazine derivatives. 502, Derivatives of phenylboric acid. 504, Determination of cryogenin. Micro-detection of alkaloids. 517, Preparation of hydroxypyruvaldehyde. 518, Gaseous higher hydrocarbon narcotics. 529—531, Vitamins. 536, Phytochemistry of Oregon plants. Leonurine.

#### PATENTS.

Skin-protective creams. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. FREDERICKSON (B.P. 342,947, 26.3.30. U.S., 27.3.29).—An emulsified mixture of soap 19, gum arabic 4, purified wool fat 2, glycerin 1, and water 74 pts. is claimed. H. ROYAL-DAWSON.

Manufacture of stable acridine salt solutions. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,690, 1.11.29).—Solutions of 3:7-diaminoacridine salts or of 3:7-diamino-5-methylacridinium salts are stabilised for injection by addition of a reducing agent, e.g., sodium hydrogen sulphite, sodium hyposulphite, acetone-bisulphite, before or after effecting dissolution.

C. HOLLINS.

Manufacture of stable therapeutically valuable solutions containing antimony. I. G. FARBENIND. A.-G. (B.P. 342,375, 5.11.29. Ger., 6.11.28).—Polyhydric alcohols having 4 or more hydroxyl groups are added to stabilise aqueous solutions of organic salts of antimony or amine salts of antimonious acid, e.g., the antimony solutions of B.P. 309,184 (B., 1930, 967).

C. HOLLINS.

Purification of extracts of sexual hormone-like-acting substances. SCHERING-KAHLBAUM A.-G. (B.P. 343,779, 14.3.30. Ger., 15.3.29).—The crude extracts are treated with methyl alcohol and the solution is extracted several times with light petroleum, the methyl alcohol being continuously further diluted with water between each successive extraction. The hormone remains in the aqueous methyl alcohol, which is evaporated.

E. H. SHARPLES.

Preparation of material containing vitamin-D. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 343,528, 14.9.29).—Ergosterol is submitted to controlled radiation by ultra-violet light from which rays having wave-lengths of  $< 270$  m $\mu$  and  $> 300$  m $\mu$  have been removed by filtration. E. H. SHARPLES.

Water-soluble vitamin.—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Latent photographic image. R. HILSCH and R. W. POHL (Nachr. Ges. Wiss. Göttingen, 1930, 176—178; Chem. Zentr., 1930, ii, 3890).—In the silver bromide layers of the dry photographic plate the nuclei of the latent image, on account of their small concentration, are not detectable optically. In an exposed silver bromide crystal several mm. thick, however, the absorption band of the latent image has a maximum at 690 m $\mu$ .

A. A. ELDRIDGE.

Photography on copper. T. J. BAKER (Nature, 1930, 126, 279; cf. Smithells, B., 1930, 883).—Photosensitive plates are best prepared by thinly coating copper or brass with electrolytic copper and immersing for a few seconds in a boiling solution of cupric chloride.

L. S. THEOBALD.

Action of azo components on treated fibres.—See VI.

See also A., April, 427, Silver migration in partly swollen gelatin layers. 428, Hydrolysis during washing of gelatin-silver nitrate system. 442, Photochemical polariser. Photometry. 443, Action of short electromagnetic waves on plates.

#### PATENTS.

[Double-image prism] colour-testing apparatus [for colour photography]. F. J. CHESHIRE (B.P. 345,868, 6.5.30).

Cellulose derivatives. Films etc.—See V.

## XXII.—EXPLOSIVES; MATCHES.

Variation of *sp.dt* with charging density for different types of [explosive] powders. H. MURAOUR and G. AUNIS (Compt. rend., 1931, 192, 90—92).—Muraour's method (A., 1930, 1530) shows that between maximum pressures of 300—2500 kg. the reciprocal of *sp.dt* is approximately a linear function of the reciprocal of the maximum pressure for (1) 20% centralite prepared with a mixture of 66 pts. of guncotton powder and 25 of nitroglycerin; (2) 9% centralite prepared similarly; (3) 50% each of guncotton powder and nitroglycerin; (4) nitrocellulose powder B plates. With mixtures (1), (2), and (4) only, the law was followed for maximum pressures down to 150 kg. The percentage variation of *sp.dt* is unaffected by the diam. (2—3 mm.) of the cylindrical stick of mixture, except for mixture (3) at low charging densities. The greatest reduction in area was obtained with (1), which has the lowest explosion temperature, and in all cases the area for 2000 kg./cm.<sup>2</sup> falls to 50% of its value for a maximum pressure of 150—200 kg./cm.<sup>2</sup> The reductions in area obtained with low charging densities are not accounted for by the influence of the action of the crusher, or of the cooling of the walls.

J. GRANT.

Laws of combustion of colloidal [explosive] powders containing vaseline. H. MURAOUR and G. AUNIS (Compt. rend., 1931, 192, 418—421. Cf. A., 1930, 1530; B., 1931, 224).—Curves relating the reciprocals of *sp.dt* and of *p* are given for an English cordite containing nitrocellulose and nitroglycerin with 5% of vaseline, and for a similar powder save that the 5% of

vaseline is replaced by a mixture of 4% of vaseline, 4% of centralite (*s*-diphenyldiethylurea), and 1% of diphenylamine. These curves show a very small diminution of  $f_p \cdot dl$  with diminution in density of loading, and consequently at all densities of loading a velocity of combustion nearly proportional to the pressure, thus differing essentially from other powders at low densities of loading. The authors' theory of combustion of powders (cf. A., 1931, 317) applies to such vaseline-containing powders, probably because vaseline, being insoluble in nitroglycerin and not gelatinising nitrocellulose, reacts more slowly than centralite.

C. A. SILBERRAD.

#### PATENT.

**Production of detonating caps.** W. ESCHBACH and W. FRIEDERICH (B.P. 343,485, 15.11.29. Ger., 15.1.29).—The sensitiveness to shock and friction of nitropenterythritol, for use as a detonator composition, is reduced by mixing with it a phlegmatising agent, *e.g.*, beeswax or paraffin wax, or a "gelatine" consisting of nitroglycerin and nitrocellulose.

W. J. WRIGHT.

### XXIII.—SANITATION; WATER PURIFICATION.

**Influence of chlorine on sewage sludge digestion.** C. E. KEEFER and H. KRATZ (Sewage Works' J., 1930, 3, 338—346).—In two cases the speed of digestion of sedimentation solids was markedly, and in two cases slightly, increased; in two cases there was a slight decrease and in one little difference. In four tests the chlorinated solids produced more total gas and in five more methane. The  $p_H$  of the chlorinated solids does not fall as rapidly as that of unchlorinated solids.

#### CHEMICAL ABSTRACTS.

**Poisonous nature of amyl nitrite.** O. GERHARDT (Chem.-Ztg., 1931, 55, 128).—A note stressing the need for adequate ventilation of rooms in which amyl nitrite is used. Its physiological effects are briefly described.

H. F. GILLBE.

**Sterilisation of water by metals.** F. DIENERT and P. ETRILLARD (Compt. rend., 1931, 192, 185—187).—Water treated as described by Lakhovsky (cf. B., 1929, 578) with silver or zinc showed marked diminution in bacterial content after three days, which was followed by an increase, very rapid in the case of silver. Sand metallised with silver by Kayser's method is more effective, removing pathogenic germs from practically any water which passes through it for a few minutes. Such water (containing less than 0.003 mg. of silver per litre) possesses bactericidal properties which are transferred to ordinary sand when filtered through it.

C. A. SILBERRAD.

**Treatment of tannery wastes.** REPORT OF THE TANNERY WASTE DISPOSAL COMMITTEE OF PENNSYLVANIA (J. Amer. Leather Chem. Assoc., 1931, 26, 70—110).—The waste lime, tan, acid- and alkaline-bleach liquors, together with one half of the wash waters, were pumped into large tanks 15 ft. above the ground, left overnight to settle, and mixed with the settled liquor from the mixture of the waste soak liquors and the remaining wash waters. The mixture was (a) chemically coagulated with sulphuric acid and 18 grains of alum per gal. in a precipitation tank, through which it was passed very slowly to the outfall and thence into

the sewer or stream, or (b) sprinkled continuously over the surface of a wooden trickling filter divided into eight compartments, each of which was separately underdrained and filled to a depth of 6 ft. with pieces of coke (0.75—1.50 in.) or hogwood. The wastes were sprinkled continuously over the surface of the filter by revolving arms over a 24-hr. period with a short rest for adjustment each morning. The effluent from this primary filtration was passed to long, shallow, earthenware basins arranged in groups, then through a second filter filled with 6 ft. of hogwood, and finally through small settling basins. Chemical coagulation with alum, lime, or lime mixed with alum or copperas was applied at the point of discharge from either or both filters, and settlement then effected in the basins. The floor of the settling tanks were fitted with sludge valves at 4-ft. intervals to permit of sludge removal without disturbing the settlement. The sludge was dried in under-drained beds (100 × 12 ft.) consisting of a 12-in. layer of screened coarse cinders topped by a 2-in. layer of smaller cinders and topped again by a 2—4-in. layer of fine cinders. Details of equipment, cost, and efficacy are given.

D. WOODROFFE.

**Determination of manganese in potable waters.**

E. BARTOW and H. THOMPSON (Proc. Iowa Acad. Sci., 1929, 36, 245—250).—No relationship was found between the manganese content and the alkalinity or the chloride or iron content. The periodate method of determination of manganese is as accurate as and more rapid than the persulphate or bismuthate method.

#### CHEMICAL ABSTRACTS.

See A., April, 538, **Determination of oxygen in seawater and fresh water.**

#### PATENTS.

**Purifying aqueous liquids, particularly waste water from factories.** METALLGES. A.-G. (B.P. 344,282, 1.5.30. Addn. to B.P. 305,657; B., 1930, 442).—The recovery of phenolic and other substances absorbed by the activated charcoal is facilitated and risk of damage minimised by substituting solvent extraction for steam scavenging. It is claimed that a more regular and efficient removal is obtained if the solvent is introduced while the absorbent is still covered with waste water, instead of previously permitting it to drain, and the temperature is maintained at 50—80°. The final traces of solvent may be removed by steam.

C. JEPSON.

**[Biological] treatment of waste water.** O. MOHR (B.P. 343,426, 4.10.29).—The efficiency of biological processes using compressed air may be increased by introducing into the aeration tank a horizontal partition, in one or more sections, so arranged as to retain air on the underside and produce an air-cushion having a pressure equal to the combined pressure of the atmosphere and that due to the hydrostatic head, and thus increasing the rate of desolution of the oxygen. Surplus air may be released either by using partitions of a permeable or semi-permeable material or by means of pipes preferably discharging in countercurrent to the inflow. An aeration chamber utilising this device may be placed with advantage midway along the settlement channel of a double-storey tank.

C. JEPSON.

**Carbon monoxide in gaseous mixtures.**—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JUNE 5 and 12, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Applications of the phthaleinoscope.** P. BRUÈRE (Ann. Falsif., 1930, 23, 532—534).—Further applications to those already described (A., 1930, 731) are to the determination of the alkalinity of liquid manures and the acid value of oils. E. B. HUGHES.

**Continuous control of salt content of boiler feed-water by means of visual conductivity tests.** E. ROTHER and G. JANDER (Z. angew. Chem., 1930, 43, 952—954).—A form of conductivity apparatus, which permits the continuous reading of electrolytic concentration of solutions over a wide range, is described. It is particularly useful in determining the salt content of boiler feed-water, in which case the concentration range which can be examined corresponds to 0.50—50.0 g. Cl/litre. E. S. HEDGES.

**Nomogram for use in gas analysis.**—See II. **Concentration by submerged combustion.**—See VII. **Testing of metallic materials.**—See X.

#### PATENTS.

**Open-hearth furnace.** S. NAISMITH (U.S.P. 1,778,505, 14.10.30. Appl., 31.7.26).—A reverberatory furnace is constructed with the whole of one side wall in the form of doors without even supporting columns to obstruct access. B. M. VENABLES.

**Recuperative soaking pit.** G. R. McDERMOTT, Assr. to CHAPMAN-STEIN FURNACE Co. (U.S.P. 1,777,125, 30.9.30. Appl., 4.4.28).—The main exhaust ports from the pit are in the same wall as the inlets, so that the bulk of the gases take a horse-shoe course; to enable the furthest portion of the pit to be equally heated, auxiliary ports are placed in the opposite wall and the gases from these are led through passages to a point in the recuperator which is subjected to greater suction than the main inlet to the recuperator, i.e., to a point nearer the chimney. B. M. VENABLES.

**Rotary kiln.** R. W. P. HORN and J. H. NISSLEY (U.S.P. 1,779,626, 28.10.30. Appl., 5.12.28).—The claims refer to means for the outlet of gases and inlet of material at the upper end of the kiln. The end of the kiln is closed except for a minor aperture through which the material is blown by compressed air. The gases are exhausted through two apertures in the cylindrical wall which, though at opposite ends of a diameter, are not opposite longitudinally and are provided with surrounding walls within the kiln to prevent exit of material. The compressed air admitted with the feed completes the combustion of partly burnt fuel. B. M. VENABLES.

**Sintering machine and the like.** E. CUDDIHY (U.S.P. 1,777,398, 7.10.30. Appl., 5.6.28).—A method

of lubricating the sliding pallets of a sintering machine is described, the oil being delivered by compressed-air ejectors. B. M. VENABLES.

**Device for the distribution of charges in a shaft furnace.** W. W. TRIGGS. From Soc. ANON. AWANS-FRANÇOIS (B.P. 345,121, 11.4.30).—Adjustable inclined blades are placed beneath the bell of the charging hopper. B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. P. COULSON (B.P. 345,583, 16.6.30. U.S., 14.6.29).—In a boiler of the type described in B.P. 319,220 (B., 1931, 139), a device is inserted in the top end of each Field tube which causes a substantial separation of liquid and vapour while they are still within the tube; in this way the bulk of the liquid is made to circulate rapidly within the tube without leaving it. B. M. VENABLES.

**Heat exchange.** C. B. GRADY, Assr. to METROPOLITAN ENG. CORP. (U.S.P. 1,779,538, 28.10.30. Appl., 24.1.27).—An exchanger suitable for transferring heat from a hot fluid, e.g., gases of combustion, simultaneously to a liquid, e.g., feed water, and to a gas, e.g., combustion air, comprises a number of vertical, double-concentric tubes, the hot fluid flowing downwards through the annular spaces, the liquid upwards through the cores, and the gas transversely and zig-zag upwards outside the tubes. B. M. VENABLES.

**Heat-exchange apparatus.** F. HEATHER (B.P. 345,279, 17.9.29 and 11.3.30).—A form of baffle suitable for insertion into the passages of a heat exchanger, to improve the contact of the fluid with the walls, is described. B. M. VENABLES.

**Apparatus for heating and cooling fluids.** R. MORTON & Co., LTD., and P. ROBINSON (B.P. 344,687, 29.3.30).—In a heat exchanger of the filter-press type, the dividing walls between the fluids are formed of deeply corrugated thin plates. Sealing means are described. B. M. VENABLES.

**Tunnel drying ovens.** J. G. OLSSON and F. I. E. STENFORS (B.P. 345,531, 17.4.30).—A tunnel kiln is provided with at least two tracks with heaters are situated between them. The main circulations are transverse and are produced by individual fans driven by a common shaft, the flows being reversible by flaps. A fresh-air inlet is provided at one end and moist air is withdrawn by independent fans. B. M. VENABLES.

**Drying apparatus.** H. HAAS (U.S.P. 1,778,318, 14.10.30. Appl., 21.11.28).—In an apparatus of the type in which the material is supported on a pervious conveyor and subjected to currents of air circulated by

\* The remainder of this set of Abstracts will appear in next week's issue.

low-pressure fans, the material is first loosened up by smaller quantities of high-pressure air applied underneath the conveyor. B. M. VENABLES.

**Drying apparatus.** J. D. ULLGREN, ASST. to AKTIEBOLAGET KARLSTADS MEKANISKA VERKSTAD (U.S.P. 1,779,571, 28.10.30. Appl., 17.12.28. Swed., 10.11.27).—A bowl is covered by a hood within which is a conical baffle upon which the material is fed and under which hot gases are admitted. The material is moved from the centre to the circumference of the bowl by scrapers running on the bottom; the angle of the scraper blades can be varied by altering the height of their supporting arms. From the hood depend one or more circular baffles so that vapours may be exhausted from two or more zones at different temperatures.

B. M. VENABLES.

**Drying apparatus.** G. W. RILEY, and G. SCOTT & SON (LONDON), LTD. (B.P. 345,302, 20.12.29).—Material such as powdered milk is produced by evaporation and grinding without removing it from a vacuum. The evaporation may be effected on a belt conveyor interleaved with heating plates and enclosed in a casing which is connected to a vacuum pump through a condenser. The flaked milk drops off the conveyor into other casings containing the grinding mills and connected direct to the same vacuum pump. B. M. VENABLES.

**Dryer.** F. A. and G. P. JADIN (U.S.P. 1,777,493, 7.10.30. Appl., 10.2.30).—The goods to be dried are contained in a stationary, cylindrical casing with perforated bottom; heat is applied to a solid bottom below it, and provision is made for catching condensate dripping from the cold roof. B. M. VENABLES.

**Desiccating apparatus.** J. O. B. COULLING and P. N. RYLANDER, ASSTS. to OTATO CORP. (U.S.P. 1,778,310, 14.10.30. Appl., 5.12.29).—A spray-drying chamber having a roof of concrete or other rigid material is provided with an inner false roof which is constructed of flexible sheets to permit expansion and contraction. The centre part is rigidly held to the outer roof and supports the spraying device and apertures for admission of drying air. B. M. VENABLES.

**Drying of grass, peat, and other divided materials.** N. TESTRUP, O. SODERLUND, and TECHNOCHEMICAL LABORATORIES, LTD. (B.P. 344,762, 1.11.29, 17.1.30, and 16.7.30).—The material is conveyed through a sinuous conduit in a current of heated air or gases of combustion. The speed is so high (10–25 m./sec.) that an initial temperature of 800–1100° may be used without harm to the material. The conduit is designed to prevent the formation of pockets of stationary material. B. M. VENABLES.

**Temperature-regulating means.** A. A. STOW and E. H. P. JONES (B.P. 344,481, 28.11.29).—A thermostat comprising concentric tubes alternately of different metals operates, through doubly magnifying levers, electric switches which control heating resistors directly or through relays. The outer tube of the thermostat is perforated to permit direct access of the hot gases to the inner tubes. B. M. VENABLES.

**Heat-insulating casings for pipe conduits.** H. SCHNELL (B.P. 344,882, 5.12.29).—Electrical heating

elements are placed in grooves in the insulating material and adjacent to the surface of the pipe so that the steam or other vapour may be maintained in superheated condition. The jacket for the heating element may be made of mica, hard porcelain, solidified water-glass, and/or oiled paper. B. M. VENABLES.

**Refrigerating medium.** H. MANNHARDT, ASST. to W. MANNHARDT (U.S.P. 1,778,033, 14.10.30. Appl., 8.8.29).—The use of sulphur hexafluoride is claimed.

L. A. COLES.

**Machine for treating materials. [Disintegrator.]** F. KUTASZEWICZ (U.S.P. 1,777,205, 30.9.30. Appl., 15.3.27. Ger., 1.2.26).—The rotor of a disintegrator acts mainly as a fan, its blades being shorter axially than the casing so that air currents can return towards the axis at the ends. Disintegration is effected by the material swirling through pockets formed in the circumferential wall, these being pear-shaped and inclined towards the material flung by the fan; the openings are at the small end. B. M. VENABLES.

**Crushing mill.** H. E. WHIPPLE (U.S.P. 1,777,061, 30.9.30. Appl., 29.10.28).—A rotating cylinder contains a number of loose, hollow, open-ended crushing rollers of decreasing diameter and placed one within the other. The material enters and leaves through opposite ends of the mill, and screens may be provided at the latter end. B. M. VENABLES.

**[Crushing or pulverising] mill.** E. H. DIMLER, ASST. to BETHLEHEM FOUNDRY & MACHINE CO. (U.S.P. 1,777,605, 7.10.30. Appl., 10.8.27).—The crushing surfaces are formed of material of the stellite type, viz., an alloy of cobalt with another member of the chromium group, e.g., tungsten, preferably applied as a welded-on lining. B. M. VENABLES.

**Grinding or crushing mills.** F. E. MARCY (B.P. 345,192, 7.1.30).—A lining for a ball mill is constructed of rolled material; the plates are wavy or corrugated, the part of the plate on the slope of the corrugations that wears most being made thicker than the rest, but any axial section is of uniform thickness.

B. M. VENABLES.

**Grinding, crushing, and pulverising machinery.** G. CLARK (B.P. 344,890, 12.12.29).—A toothed rotor effects disintegration of the material in three stages, viz., against a plain or ribbed surface well clear of the rotor; against the ends and sides of curved channels intercalating with the teeth; and against grid bars.

B. M. VENABLES.

**Grinding mills.** E. BARTHELMLESS (B.P. 345,388, 14.1.30).—A rotor produces high-speed circular currents of air between two concentric rings and in addition drives mullers, rolls, or other positive crushing elements on the inside of the inner ring. The rotating air current effects the final grinding, and conveys away the finely-crushed material without the use of another fan; partly ground material drops out of the air current into the path of the positive crushers. B. M. VENABLES.

**Pulveriser.** C. E. BRAINARD, ASST. to WESTERN FOUNDRY CO. (U.S.P. 1,776,411, 23.9.30. Appl., 2.8.28).—The machine comprises a rotating shell containing loose, segmentally divided grinding rollers alternating with

distributing rings of bladed form. Charging and discharging is effected through hollow trunnions.

B. M. VENABLES.

**Pulveriser.** C. A. DREISBACH, ASST. to STANDARD EQUIPMENT CO., INC. (U.S.P. 1,776,654, 23.9.30. Appl., 27.2.28).—A pair of crushing rolls, preferably fluted, are mounted so that the upper one can rise and fall and both are surrounded by a rotating drum which is provided with screening and lifting devices, and dips into a bath of water; the driving shaft and arms for the rolls are inserted through large openings in the ends of the drum.

B. M. VENABLES.

**Pulveriser.** W. W. PETTIBONE (U.S.P. 1,778,564, 14.10.30. Appl., 19.7.29).—In a disintegrator comprising a beater chamber, an inlet chamber for additional air, and one or more fan chambers in series around the same shaft, the air-inlet ports are provided with deflectors which divert coarse particles from the outer part of the whirling currents to positions near the axis, whence they re-enter the grinding chamber.

B. M. VENABLES.

**Pulverisers.** SOC. FRANÇ. DE CONSTRUCTIONS MÉCANIQUES ANC. ETABL. CAIL (B.P. 345,534, 22.4.30. Fr., 24.4.29).—A ball or pebble mill has the ground material removed by a current of air which is admitted to the lower part of the grinding mass through ports which are actually covered by it. The fixed air-inlet pipe inserted through the trunnion communicates only with ports that happen to lead to that part of the mass that has the most kinetic energy, and the air stream carries the feed material with it.

B. M. VENABLES.

**Pulverising apparatus.** CHAUFFE ET COMBUSTION, SOC. ANON. (B.P. 345,465, 5.3.30. Belg., 6.3.29).—A conical grinding element is gyrated in a conical casing in such a manner that its axis generates a cone; a spherical portion attached round its apex works in a corresponding spherical recess in the casing, and the point of oscillation lies on or near the spherical surface.

B. M. VENABLES.

**Screening plant.** V. REYNOLDS and J. MASEFIELD (B.P. 344,673, 13.3.30).—The use of rubber bands on the tyres and/or rollers of a rotary screen is claimed.

B. M. VENABLES.

**Separation of dry materials.** C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 343,946 and 344,802—4, [A] 19.8.29, [B—D] 28.8.29).—Devices in which a bed of the mixed materials is kept fluid by air currents flowing upwards through a pervious support are described. In (A) a number of fingers are provided upstanding from the deck of a reciprocating table or dependent from a grid above it, the fingers in either case partaking of the movement of the table and extending right through the bed of material. In (B) the device comprises an inclined conveyor, or rakes running over an inclined surface. The feed is near the top of the slope, the heavy material being dragged upwards and the light running downwards by gravity and being discharged over a weir at the foot of the slope. In (C) a substantially level trough has two runs of a single conveyor or of two separate conveyors running through it at different levels and moving the light and heavy material to opposite ends. In (D) a cylindrical tank is provided with a

pervious diaphragm above which are rotated arms, curved in opposite directions in different strata and delivering the lighter material to the circumference and the heavier to a central outlet; the feed is also central at a height which is adjustable above the edge of the tank.

B. M. VENABLES.

**Separation of dry materials.** I. L. BRAMWELL, and BIRTLEY CO., LTD. (B.P. 345,275, 10.9.29).—A pneumatic shaking table is supplied with a steady current of air which is rendered pulsating as it passes through the deck by forming the bottom of the air chamber as a bellows and reciprocating it in a vertical direction simultaneously with the horizontal reciprocation of the whole table.

B. M. VENABLES.

**Centrifugal machines for separating solids and liquids.** F. DUPUIS, and ATELIERS J. HANREZ SOC. ANON. (B.P. 344,658, 3.3.30).—A method of continuous discharge of solids is described. It involves raking by worms in two stages, in the second stage of which the rakes moving at centrifugal speed work adjacent to a stationary surface; the gearing also operates at high speed.

B. M. VENABLES.

**Separation of solids from liquids.** M. P. REYNOLDS, ASST. to W. S. TYLER CO. (U.S.P. 1,775,830, 16.9.30. Appl., 7.1.27).—Sugar cane or similar material containing liquid amenable to such treatment is crushed to liberate the juice, the solids are removed by vibratory screens, and the residue is re-crushed to remove further liquor.

B. M. VENABLES.

**Apparatus for conveying powdered materials in pipes.** I. E. and E. LANHOFFER, ASSTS. to FULLER CO. (U.S.P. 1,777,043, 30.9.30. Appl., 29.10.25. Fr., 24.9.25).—When powdered material is conveyed in a stream of compressed air, expansion of the air may so disperse the powder that it settles out; to avoid this the conveying conduit is gradually contracted in the direction of flow. Means for charging the powder and air into the conduit are described.

B. M. VENABLES.

**Agitation of materials.** I. T. THORNTON and J. H. CROWELL, ASSTS. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,778,049, 14.10.30. Appl., 4.6.28).—A mixer is described which shears the material away from the wall, changes the peripheral movement to a radial one, and gives a downward motion to the centre of the mass.

B. M. VENABLES.

**Inclined mixers comprising a bowl-like rotating drum.** F. MOSER (B.P. 345,169, 12.6.30. Ger., 15.6.29). The fixed axle-pin is extended within the bowl and on it is placed a deflecting plate which catches material dropping from near the closed end of the bowl and deflects it towards the open end.

B. M. VENABLES.

**Centrifugal separators and methods of operating same.** H. W. FAWCETT (B.P. 344,464, 9.9.29, 16.10.29, and 4.7.30).—In a centrifuge adapted to the continuous separation of solids from liquids, the waste of lighter liquid is prevented and the discharge of solids aided by the use of a carrier liquid of intermediate density. This is supplied to an outer zone partitioned off from the separating zone except for passages opposite the apertures for discharge of solids; the outer zone is vented over a weir which is adjustable, and the carrier liquid

is so balanced against the lighter liquid that the dividing surface comes within the transfer ports between the inner and outer zones.  
B. M. VENABLES.

**Centrifugal machines for separating milk or liquids of different density.** ECREMEUSES MELOTTE SOC. ANON. (B.P. 344,710, 29.4.30. Belg., 28.5.29).—An elastic restraint for the shaft of a centrifuge is described.  
B. M. VENABLES.

**Production of conical discs for centrifugal separators.** BERGEDORFER EISENWERK A.-G. ASTRA-WERKE (B.P. 344,473, 4.11.29. Ger., 3.11.28).—Methods of manufacture of the discs are described, involving spinning so that the conical part is thinner than are the flanges.  
B. M. VENABLES.

**Device for separating light liquids.** W. LINN-MANN, JUN. (B.P. 344,604, 21.1.30).—The tangential inlet of a settling vessel is continued right round the circumference in the form of a gutter open to the interior of the vessel.  
B. M. VENABLES.

**Filters.** H. S. HELE-SHAW, W. J. RIDGWAY, and W. N. WILLIAMS (B.P. 345,251, 14.11.29).—The elements of an edge-filter are formed of wire-gauze sheets having porous granular material attached to their edges by the natural roughness, assisted, if desired, by interleaving them with discs of solid sheet of rather larger diameter.  
B. M. VENABLES.

**Filters.** METALLGES. A.-G., G. MÜLLER, and E. SIEBERT (B.P. 344,502, 5.12.29).—In a filter of the type comprising a mass of active or other material restrained between perforated diaphragms, provision is made to permit any gas that may accumulate on the inlet side to pass to the outlet side through a small controllable by-pass, the incoming liquid being guided so as to sweep towards the entrance of the by-pass.  
B. M. VENABLES.

**Filter device.** R. M. THOMPSON (U.S.P. 1,778,342, 14.10.30. Appl., 3.1.21).—The material is filtered through two pervious conveyors running together which may approach, recede, and approach each other. During one or more of the recessions a washing or extracting liquid may be applied to the partly pressed material, and gutters are provided to catch separate filtrates so that, *e.g.*, in the pressing of fish or fish offal, the oils may be collected separately from the glue stock.  
B. M. VENABLES.

**Thickening filters.** J. B. VERNAY (B.P. 345,383 and Addn. B.P. 346,078, [A] 10.1.30, [B] 3.2.30. Fr., [A] 10.1.29, [B] 18.5.29).—In (A) the filter comprises a number of hollow, rotating, cylindrical elements entirely submerged in a trough of the prefilter, the whole of each element being subjected either to a vacuum for filtering or to a fluid under pressure (preferably air) to dislodge the thickened mud. In (B), in order to reduce the amount of air that has to be exhausted, to re-establish the vacuum, and to enable wet vacuum pumps alone to be used, a hollow hermetically sealed spacer is placed within each element, leaving only a small annular space for filtrate or air.  
B. M. VENABLES.

**Mechanical filters.** S. J. RICKS and C. E. DOUGLAS (B.P. 345,514, 4.4.30. Addn. to B.P. 322,148; B., 1930, 126).—A less expensive form of the filter is

described, in which the stirring and cleaning apparatus is applied only to the upper part of the sand.

B. M. VENABLES.

**Filtration process and apparatus.** C. T. CABRERA, ASSR. to ELECTRO DIALYZER CORP. (U.S.P. 1,776,883, 30.9.30. Appl., 1.6.28).—The liquid, *e.g.*, sewage or waste liquors from pulp mills, canning plant, etc., is passed upwardly through a chamber, or preferably through a number of chambers in parallel, having open top and bottom and containing a mass of filtering material such as stranded metal which is compacted to any desired extent by a screw and pressure plate at the top and may be flushed through closable openings in the sides of the chamber; provision is made for rapidly changing the lower section independently of the upper part.  
B. M. VENABLES.

**Apparatus for dialysing liquids.** O. M. URBAIN, ASSR. to OHIO SANITARY ENG. CORP. (U.S.P. 1,777,057, 30.9.30. Appl., 13.7.28).—The apparatus is suitable for tests on putrescible matter which may take several days. The membrane is in the form of a shallow tray that floats on the dialysing water contained in a closed vessel. The water is constantly renewed from a closed reservoir; the air, which must be admitted to permit outflow of water, is freed from oxygen by an absorbent such as alkaline pyrogallate and the dissolved oxygen is removed from the water before or during use.  
B. M. VENABLES.

**Distillation apparatus.** D. T. WILLIAMS, ASSR. to PETROLEUM DERIVATIVES, INC., OF MAINE (U.S.P. 1,777,950, 7.10.30. Appl., 12.7.24).—The apparatus comprises a number of straight inclined tubes arranged zig-zag in a setting for heating by gases of combustion. Vapour outlets are provided at the return bends down one side of the setting and, as the apparatus is specially intended for the continuous distillation of mineral oils under high vacuum, provision is made for breaking up foam, comprising inner tubes, spaced from the walls of the outer tubes, having closed upper ends and perforations on the top. The spacer in the annular space at the lower end of any tube is arranged to permit flow of liquid through the annular space, but to confine vapour to the core. The feed is at the uppermost end, and the first tube serves only as a preheater. There may be a number of vertical rows of tubes in parallel in the same setting.  
B. M. VENABLES.

**Distillation apparatus.** D. T. WILLIAMS (U.S.P. 1,778,177, 14.10.30. Appl., 20.11.25).—In a distillation system, especially for mineral oils, the still and condenser are maintained at a higher vacuum than is the receiver, the difference being obtained by placing the last-named at a lower level.  
B. M. VENABLES.

**Fractionating tower.** J. C. BLACK (U.S.P. 1,776,061, 16.9.30. Appl., 10.11.26).—A system for the fractionation of hydrocarbons or other liquids is described. Above a main fractionating tower is an analyser producing reflux liquid and the final vapour. Below the main part of the tower are two other compartments heated by steam coils and provided with bubble trays producing final liquid fractions.  
B. M. VENABLES.

**Fractionating column.** R. E. WILSON and W. H. BAHLKE, ASSRS. to STANDARD OIL CO. (U.S.P. 1,778,475,



14.10.30. Appl., 6.8.27).—A form of bubbling tray is described in which the pool of liquid is held at a definite depth. B. M. VENABLES.

**Dephlegmator or bubble towers.** H. MOORE, and J. B. JOHNSON & Co. (REFINERY ENGINEERS), LTD. (B.P. 345,565, 19.5.30).—In a bubble tower, the liquid is caused to flow in a circular direction round the trays, flow across the centre being prevented by a large bubble cap. The downflow port is in the form of a radial opening protected on one side by a wall, the plate next below being rotated slightly with respect to the one above. B. M. VENABLES.

**Bubble cap for gas and liquid contact apparatus.** C. M. ALEXANDER (U.S.P. 1,777,869, 7.10.30. Appl., 18.1.27).—The cap is shaped like a hollow gear wheel in plan view, and has slot-shaped gas outlets in both the flanks and ends of the teeth. B. M. VENABLES.

**Bubble caps.** J. E. KOBERNIK (U.S.P. 1,776,032—3, 16.9.30. Appl., 5.1.28).—In (A) the inner part of a duplex cap has a closed top and the outlet for the gases is through slots in the lower part of the vertical cylindrical wall; the bubbles are caught under an attached annular cap with serrated lower edges and perforations in the top. In (B) the cap comprises a perforated dished top supported by a pair of uprights, and is formed out of a single sheet of metal. B. M. VENABLES.

**Heating and cooling of fluids and viscous liquids.** C. W. STANCLIFFE (B.P. 345,312, 20.9.29).—In apparatus of the type described in B.P. 286,757 (B., 1928, 352), the scrapers, rollers, or spiral springs are subdivided into comparatively short lengths so that they will effectively operate irrespective of slight untruth of the tubes. B. M. VENABLES.

**Apparatus for recovery of the energy transmitted to liquids in the conveyance of the same against high pressures.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 344,842, 30.8.29).—The pressure on an earlier batch of liquid is reduced, and that on new liquid raised, on opposite sides of a piston or diaphragm, the pump side being smaller than the motor side by the area of the piston rod; in cases where a diaphragm is employed, booster pumps may be used to make up friction losses. Hydraulic and electrical means are described for controlling the valves. [Stat. ref.] B. M. VENABLES.

**Apparatus for vaporisation of liquids.** F. C. CALVERT & Co., LTD., and B. LE N. FOSTER (B.P. 344,821, 7.12.29).—A liquid, such as disinfectant, is slowly dripped on to an electrical hot-plate, the resistance wire of which is insulated by means of magnesium metal (or alloy) which has been converted into hydroxide by heating in steam; claim is also made for the form of the apparatus. B. M. VENABLES.

**Concentrating and homogenising apparatus [for prefill].** E. HEIBIG, Assr. to FILTRES PHILIPPE (U.S.P. 1,778,596, 14.10.30. Appl., 14.3.29. Fr., 18.6.28).—A thickening filter for providing concentrated and thoroughly mixed feed for a rotary filter is described. The pulp dropping from vertical cylindrical vacuum elements is collected by a pair of rotating cylinders provided with vanes so that they act as a gear-wheel

pump and force the thick pulp to a further stirrer and conveyor. The cylinders not only mix and deliver the pulp, but also prevent the disturbance in the bottom of the vessel from reaching the filtering zone. B. M. VENABLES.

**Catalytic gas reactions.** DU PONT AMMONIA CORP., Assees. of J. A. ALMQUIST (B.P. 344,576, 31.12.29. U.S., 31.12.28).—In a process involving the use of a main catalyst and a purifier catalyst, the flow of gases is in the following order: in heat exchange with, and within the same pressure-resisting wall as, but not in direct contact with, the main catalyst; through the purifier catalyst in a separate pressure-resisting vessel; through a device to remove the converted impurity and to regulate the temperature; through the main catalyst to the place of recovery of the desired constituents. B. M. VENABLES.

**Apparatus for treatment of gases or mixtures of gases and vapours, in the presence of catalytic or purifying agents and for the reactivation of such agents.** COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 345,496 and Addns. B.P. 345,511 and 346,967, [A] 25.3.30, [B] 1.4.30, [C] 28.3.30. Fr., [A] 2.8.29, [B] 13.1.30, [C] 5.12.29).—In (A) the catalytic or purifying material is supported between perforated disc partitions in a cylindrical casing and the gases to be treated pass through it from end to end, but the gas for regeneration is admitted and exhausted through perforated tubes embedded in the mass; thus the gas passes transversely through it in a well-distributed manner. In (B) coils for heating or cooling are also embedded in the mass. In (C), the casing of the apparatus is formed as a parallelepiped having small width compared with length and height; the distributing roses for the regenerating gases are placed horizontally along the long sides with their apertures facing the walls, which may there be indented to act as reflectors. B. M. VENABLES.

**Apparatus for separating and recovering vapours and gases with adsorbent carbon.** O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,777,460, 7.10.30. Appl., 2.7.24. Renewed 21.12.27).—A vertical cylindrical casing is divided into three compartments by vertical foraminous walls: the outer compartments serve as inlet and outlet headers for the various reacting and regenerating fluids, and the inner contains the active carbon in which are embedded heating-cooling coils served from headers formed in the lower end of the shell. B. M. VENABLES.

**Separation of [acidic] gases.** GIRDLER CORP. (B.P. 344,786, 4.12.29. U.S., 4.12.28).—Gases such as carbon dioxide, sulphur dioxide, and/or hydrogen sulphide are absorbed by definite chemical combination in a liquid which is afterwards regenerated by heat. Suitable absorbents are mono- and di-ethylamine and "triethanolamine"; the last-named has the advantages that it is a viscous non-volatile liquid soluble in water and its salts are decomposed at about 100°. B. M. VENABLES.

**Gas washer.** G. W. VREELAND (U.S.P. 1,776,644, 23.9.30. Appl., 24.2.28).—The apparatus comprises a tower containing a number of trays and lifting-spraying

cones. Moisture eliminators may be placed in an exit passage wrapped round the tower. B. M. VENABLES.

**Gas scrubber.** A. G. MCKEE (U.S.P. 1,778,426, 14.10.30. Appl., 15.3.26).—The apparatus comprises a tower containing centrifugal devices which spray the liquid to the walls, from which it is collected and re-sprayed. B. M. VENABLES.

**Apparatus for treatment of industrial gases.** COMP. GÉN. DE CONSTRUCTION DE FOURS (B.P. 345,457, 1.3.30. Fr., 2.3.29).—The gas is caused to pass through either a ring of jets or a narrow annular orifice and then to impinge on a film of liquid spread centrifugally over the surface of a rotating dish; the flow of the gas is inwards to a central exhaust passage, and therefore countercurrent to the film of liquid, this film being also sufficiently strong to prevent puncturing by the jets of gas. The apparatus is suitable for the chemical as well as physical removal of a constituent or impurity of the gas. B. M. VENABLES.

**Centrifugal means for treating [separating dust from] gases.** W. W. TRIGGS. FROM INTERNAT. PRECIPITATION CO. (B.P. 344,421, 28.11.29).—The apparatus comprises a number of vortical separating chambers, of which the length is many times the diameter, operated in parallel and having common inlet, outlet, and dust-collecting chambers. The clean air outlets are  $\frac{1}{2}$ — $\frac{3}{4}$ , and the dust outlets are tapered down to  $\frac{1}{4}$ — $\frac{3}{8}$ , of the diameter of the largest part of the cyclone chambers. It is stated that a double vortex is formed, the outer one producing a downward force on the dust many times that of gravity, and the inner producing an upward flow of clean air which originates in the dust-settling chamber at the bottom. B. M. VENABLES.

**Gas pumps particularly for use in gas-analysis apparatus.** AKTIEBOLAGET CARBA, ASSCES. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 344,952, 14.12.29. Swed., 17.12.28).—A water-operated suction pump acting on the broken-column system is described. B. M. VENABLES.

**Apparatus for solidifying gases.** H. B. RUDD (B.P. 345,229, 14.11.29. U.S., 17.11.28).—Liquid carbon dioxide or other gas to be solidified is passed through a cooling coil and expansion jet into a chamber lined with a cartridge of hard rubber or other smooth non-conducting material. The gas produced by the expansion passes over the cooling coil, and when the chamber becomes full of solid the increase of pressure operates an automatic valve to cut off the supply of liquid. B. M. VENABLES.

**Lubrication of steam turbines.** G. W. BURKHARDT, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,778,447, 14.10.30. Appl., 16.3.27. Ger., 13.10.26).—The lubricant comprises a mixture of a refined mineral oil distillate with, e.g., 3% of high-molecular alcohols of wool grease. L. A. COLES.

**Hydraulic-brake fluid.** R. M. BAGLEY, ASSR. to R. M. HOLLINGSHEAD CO. (U.S.P. 1,779,460, 28.10.30. Appl., 8.3.29).—A mixture of equal volumes of ethylene glycol monoethyl ether and castor oil is claimed; part or all of the latter may be replaced by glycerin. A. R. POWELL.

**Charge for fire extinguishers.** C. A. THOMAS and C. A. HOCHWALT (U.S.P. 1,777,339, 7.10.30. Appl., 12.11.26).—A charge for anti-freeze fire extinguishers for use at temperatures down to  $-40^{\circ}$  comprises a solution containing 25% of sodium potassium carbonate, 36% of ethylene glycol, and 39% of water in one portion of the receptacle and a solution of chlorosulphonic or similar acid in the other. A. R. POWELL.

**Chemical thermoscope composition.** T. L. DAVIS, ASSR. to W. O. SNELLING (U.S.P. 1,777,353, 7.10.30. Appl., 19.12.28).—A solution of 1 g. of hexapyridine cobaltous cyanate in 100 c.c. of chloroform to which are added 16 c.c. of pyridine is pink below  $20^{\circ}$ , lilac at  $20$ — $30^{\circ}$ , violet at  $30$ — $40^{\circ}$ , and blue above  $40^{\circ}$ . With increasing amounts of pyridine the colour changes occur at higher temperatures, and with less pyridine at lower temperatures. A. R. POWELL.

**Reducing the atmospheric moisture content in confined spaces.** R. A. COCK (B.P. 345,358, 23.12.29).—A pad impregnated with a 1:3 solution of calcium chloride is used to prevent steaming of shop windows etc. B. M. VENABLES.

**Container for corrosive materials.** R. M. MEIKLEJOHN, ASSR. to GEN. CHEM. CO. (U.S.P. 1,778,532, 14.10.30. Appl., 24.8.27).—The invention relates to the closure of a lined tank. The non-corrodible lining, or a collar-like extension of it, is brought out through the bung hole and bent back over the edge of the mouth, which is, if necessary, reduced in thickness so that the lining lies below the roots of the screw thread if such be used. The closure is completed by a female cap lined with a disc of the same material, no additional gasket being provided. B. M. VENABLES.

**Polarimeter indicator.** L. MEYER (U.S.P. 1,777,216, 30.9.30. Appl., 11.2.29. Ger., 20.1.28).—The graduated drum is geared to the polarimeter proper by worm and wheel, and is provided with two scales each of which reads in both directions: one is graduated in plus and minus degrees of angle; the other, for use in urine analysis, is marked in percentage of sugar on the positive side and percentage of albumin on the negative. To prevent confusion as to positive and negative and to count complete revolutions of the worm or drum, a pointer is attached to the wheel or polarimeter proper. B. M. VENABLES.

**Continuously indicating and recording viscosimeters.** J. L. HODGSON and H. E. DALL (B.P. 345,210, 14.12.29).—A continuous sample of the liquid is brought to a standard temperature and caused to flow under a constant head through an orifice with turbulent (or mixed turbulent and laminar) flow into a vessel having a capillary outlet giving purely laminar flow. The amount of liquid accumulating in the second vessel under these conditions will be a measure of the head producing the discharge, and hence of the viscosity; the weight is determined by mounting the vessel on a scale beam. In each case the flow from the outlet is in a horizontal direction to avoid effects of momentum. Alternatively to the first vessel and orifice a pump having constant discharge may be used. B. M. VENABLES.

**Apparatus for aerating and/or mixing liquids.** W. N. JONES (B.P. 346,455, 13.1.30).—See U.S.P. 1,765,338; B., 1931, 140.

**Furnaces for cremating or incinerating.** K. LUDWIG (B.P. 344,946, 16.12.29).

**Filtering device for liquids under high pressure, particularly fuel oil for internal-combustion engines.** F. V. HAUGSTED (B.P. 344,932, 14.12.29).

**Tunnel kiln for charcoal.**—See II. **Filtration of suspended sediments.** Dry kiln.—See IX. **Furnace parts.**—See X. **Purification of gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Classification of Rumanian coals.** L. BLUM (Bul. Chim. Soc. Române, 1931, 31, 3—11).—Classification on the basis of geological and "chemical age" (*i.e.*, the extent of chemical transformation of the primal material) is discussed. Comparison of the elementary composition of Rumanian coals and lignites, the temperatures at which they evolve gases, and the amounts and composition of these gases, indicates that Lupeni coal is of a bituminous nature. Comparison of the effects of reagents (*e.g.*, potassium hydroxide, dilute nitric acid, mixtures of nitric and sulphuric acids) leads to similar conclusions. The contents of bitumen, lignin, humic acids, and cellulose in the coals and lignites were determined by extraction with benzene-alcohol mixture, sulphurous acid, ammonia, and Schweitzer's reagent, respectively; lignin and humic acids characteristic of lignite were absent in Lupeni coal. It is concluded that Lupeni coal should be classed as bituminous.

H. E. BLAYDEN.

**Influence of the constituents of Rumanian coals on the composition of the gases obtained by distillation to 500°.** I. BLUM (Bul. Chim. Soc. Române, 1931, 31, 13—19).—Four Rumanian coals were exhaustively extracted in succession with benzene-alcohol mixture and with benzene under pressure, to extract bitumens; with normal sulphurous acid to extract lignin; and with ammonia to extract humic acids. The untreated coals and the residues from each extraction were distilled up to 500° and the volume and composition of the gas evolved determined in each case. The coals richest in humic acids and lignin evolved the most gas, and the amounts of unsaturated hydrocarbons decreased on extraction of the bitumen. Carbon dioxide was evolved in least quantities from the final extraction residue, which also evolved large amounts of methane; but carbon dioxide and monoxide were evolved in large amounts from the residues rich in lignin and humic acids. Bitumen could be extracted from the ligneous coals by use of benzene-alcohol mixtures alone.

H. E. BLAYDEN.

**Low-temperature carbonisation of Donetz coals.** A. P. SCHACHNO and I. B. RAROPORT (Brennstoff-Chem., 1931, 12, 121—122; cf. B., 1930, 42).—Six coals of varying rank from the Donetz region have been carbonised at 200°, 350°, 425°, and 500° in the aluminium assay apparatus. A flaming gas coal (moisture 3.5%, ash 11.4%, volatile matter 43.1%) proved most suitable for low-temperature carbonisation; its semi-coke was little hygroscopic, easily combustible, relatively hard,

and stable on storage. Distillation of this coal at 500° yielded 14.1% of tar and about 8% of gas.

A. B. MANNING.

**Lignite III. Analysis of lignite from Kankyohokudo, Korea.** R. ODA (J. Soc. Chem. Ind., Japan, 1930, 33, 398—399 B).

**Lignite. IV. Low-temperature distillation of lignite, humic acid, lignin, and cellulose.** R. ODA (J. Soc. Chem. Ind., Japan, 1930, 33, 441—444 B; cf. B., 1930, 973).—Tables and curves are given showing the evolution of gas, analysis of gas, and the proportion of coke, tar, water, and gas obtained in the distillation between 0° and 500° of lignite (mediaeval lignin, cf. B., 1930, 973), humic acid I prepared from the foregoing, humic acid II prepared from dextrose by Conrad's method, lignin isolated from *Picea ajanensis* according to Willstätter, and cotton-paper.

L. J. HOOLEY.

**Destructive hydrogenation in bomb-polymerisation in cracking.** S. A. KISS (Ind. Eng. Chem., 1931, 23, 315—318; cf. B., 1930, 175).—A theoretical discussion of the results obtained by Waterman and Perquin (B., 1925, 746).

C. B. MARSON.

**[Carbonisation of coal and dehydration of gas.]** H. C. SMITH (Gas J., 1931, 193, 787—794).—Results obtained by carbonisation of cleaned coal, and by gasification of the resulting low-ash coke, are briefly described. Working data and costs are also given for a gas-dehydration plant dealing with 3 million cub. ft. per day.

R. H. GRIFFITH.

**Partial dehydration of town gas.** R. L. GREAVES (Gas J., 1931, 193, 797—799).—A review of the arguments for and against drying of town gas. Labour and capital costs are given for a plant working with calcium chloride and dealing with 100,000 cub. ft. per hr.

R. H. GRIFFITH.

**Catalytic reduction of carbon monoxide to methane in mixtures of carbon monoxide and hydrogen and in illuminating gas, in presence of nickel-asbestos, whereby carbon dioxide, heavy hydrocarbons, and oxygen are removed.** H. A. J. PIETERS (Chem. Weekblad, 1931, 28, 209—210).—An experimental confirmation of the method of Larson and Whittaker (B., 1925, 400). The carbon monoxide content of carbon monoxide-hydrogen and carbon monoxide-hydrogen-methane-nitrogen mixtures may be accurately determined by hydrogenation. The results agree satisfactorily with those obtained by the ammoniacal cuprous chloride method. H. F. GILLBE.

**Nomogram for use in gas analysis.** J. SCHMIDT (Chem. Fabr., 1931, 137—138).—The nomogram is constructed to enable methane and hydrogen content to be read separately from the volume contraction and carbon dioxide absorbed after the combustion of an aliquot part of the gas sample with air. Small values such as may be obtained for carbon dioxide absorption after the combustion are multiplied by 10 or 100 in order to yield accurate results.

C. IRWIN.

**Aqueous liquors from low-temperature carbonisation of coal.** II. G. T. MORGAN and A. E. J. PETTET (J.S.C.I., 1931, 50, 72—74 T).—*o*-Cresol, *m*-cresol, homocatechol, isohomocatechol, resorcinol, and quinol

were isolated from the liquor produced at 625° from a Warwickshire coal, and in the same liquor determinations were made of the forms in which sulphur occurs. In a liquor obtained from Dalton Main coal at 450°, phenol, *o*-cresol, pyrocatechol, resorcinol, quinol, and acetic acid were identified. Comparisons are drawn between liquors produced from several coals at 625°, and the total phenolic content is shown to depend on the oxygen content of the coal used. In comparing liquor from high-temperature carbonisation with that from low-temperature carbonisation, it is found that the former has a lower total phenolic content, but a higher content of phenol itself.

**Determination of the paraffin content of asphalts.** W. von PIOTROWSKI and H. BURSTIN (Petroleum, 1931, 27, Asphalt, [3], 3—7).—The paraffin contents of three samples of asphalt (obtained by concentration of a paraffin-free mineral oil, by air-oxidation of the residues from the cracking of mineral oils, and by concentration of a paraffin-containing mineral oil, respectively) were determined in three independent laboratories by (a) the Engler-Holde, (b) the Marcusson, and (c) the Schwarz methods, details of which are given. A comparison of the results indicates that method (c) is unreliable on account of the disturbing influences of the high temperatures used, that method (b) is too complicated for technical purposes and gives results difficult to reproduce, and that method (a) is simple and rapid, and gives relatively good reproducibility of results. On the basis of the results, the Mineral Oil Section of the Polish Standardisation Committee have accepted the Engler-Holde method as standard. H. E. BLAYDEN.

**Cracking of Rangoon paraffin in vapour phase.** J. W. HEYMANS (J. Inst. Petroleum Tech., 1930, 16, 661—677).—Rangoon paraffin (av. mol. wt. 407) was cracked under vacuum at 450°, 600°, and 700°. The products were examined for quantity, mol. wt., bromine absorption, m.p., and refractive index. At 450°, 2.1 mols. per mol. of paraffin were obtained, 6 mols. at 600°, and 13 mols. at 700°. Splitting takes place at the end of the molecules and the yield of unsaturateds increases with rise of temperature. A certain amount of polymerisation takes place at the lower temperatures. At 700° the non-gaseous products are almost entirely unsaturated. T. A. SMITH.

**Cracking and condensation of hydrocarbons.** II. Acetylene. E. BERL and K. W. HOFMANN (Z. angew. Chem., 1931, 44, 259—261; cf. A., 1931, 596).—In presence of carbon as catalyst, acetylene polymerises at 600—700° to aromatic hydrocarbons, with high yield. Metallic catalysts, and those containing silica, stannic oxide, or calcium oxide, are unsatisfactory, whilst carbon catalysts of large active surface promote decomposition of the acetylene and formation of methane. The yield of liquid products of low b.p., suitable for use as fuel, is increased in presence of water vapour, a 98.8% conversion being obtained at 740° without appreciable loss of activity of the catalyst even after prolonged use. H. F. GILLBE.

**Determination of the gum content of cracked spirits.** W. LITTLEJOHN, W. H. THOMAS, and H. B. THOMPSON (J. Inst. Petroleum Tech., 1930, 16, 684—695).

—As the present methods were found to give discordant results for samples of the same spirit, the following standard method of carrying out the test has been developed. A hemispherical glass dish (wt. 20 g., diam. 7 cm., depth 3.5 cm.) is placed on a steam-bath with an arrangement for giving a standard current of air over the surface of the spirit. The sample (25 c.c.) is evaporated for 1—1½ hrs. If the sample yields more than 11 mg. of gum, the test is repeated with a fresh diluted sample which will give not more than this weight of gum. Gum-free spirit must be used for blending. Precise details are given in the original. T. A. SMITH.

**Lubricating fractions of Cabin Creek (W. Va.) petroleum. Action of solvents on the heavy constituents of petroleum.** H. M. SMITH (U.S. Bur. Mines, Tech. Paper, 1930, No. 477, 48 pp.).—Lubricating oil stock was prepared from the crude oil by distillation in steam after topping by natural gas, and removal of wax by cold-settling following addition of acetone-*sec*-butyl alcohol mixture. The lubricating stock was then repeatedly extracted with acetone at 37.8° until 60 fractions had been taken, and then a further 35 fractions were obtained at 50° as the solubility had fallen too low at the lower temperature for satisfactory extraction. A further six fractions were then extracted by ethyl acetate at 21.1°. The physical constants and empirical formula of each fraction were determined, and the fractions were further examined by fractionation with acetone and vacuum distillation. The tables of data show that the lubricants are roughly divided into two types by selective extraction with acetone. The more soluble portions have relatively high sp. gr. and C:H ratio, low mol. wt., and low or medium viscosity, the less soluble portions having the converse of these properties. For paraffin-base crude it is shown that sp. gr., refractive index, sulphur content, and carbon residues are dependent on  $x$  where  $C_nH_{2n+x}$  is the empirical formula of the oil. The other properties are functions of the molecular constitution.

[With F. W. LANE, I. H. NELSON, and J. M. DEVINE.] Extractions on 3 crude oils were carried out with acetone and with ethyl alcohol. The extraction tends to divide the oils into two types, that extracted first being the more naphthenic in its qualities. Distillation alone gives only a series of oils of increasing density and viscosity. Distillation is necessary, however, for fractionation after the initial separation into types has been made by means of solvents. T. A. SMITH.

**Desulphurisation of Tschusovskie Gorodki petroleum distillates, using silica gel.** G. I. TSCHUFAROV and M. R. LITOVTSCHENKO (J. Chem. Ind., Moscow, 1931, 8, 65—68).—Silica gel removes only mercaptans from petroleum distillates; by a combination of treatment with sulphuric acid and with this adsorbent 70% of the sulphur can be removed from this material. R. TRUSZKOWSKI.

**Determining the viscosity of oils by the Tausz falling-ball viscosimeter.** F. GEIGER (Petroleum, 1931, 27, 209—214).—After multiplying the time taken by the ball to fall, as measured by this instrument, by a factor, the result differs by 0—5% from that obtained by Ostwald's method. The figures obtained vary with

(a) the weight of the ball, (b) the oils used, and (c) temperature and the length of tube. Neither the Stokes nor the Ladenburg formula is sufficiently accurate, but these formulæ may be made more accurate by using them in conjunction with a factor. Experiments are still in progress to determine the viscosity of turbulent oils and liquids.

E. DOCTOR.

**Viscosity-temperature relations of lubricating oils.** W. L. BAILLIE (J. Inst. Petroleum Tech., 1930, 16, 643—660).—The equations  $\log A = m \log \theta + K$  and  $\log V = n \log \theta + C$ , due to Eckart (Day, "Handbook of the Petroleum Industry," 1, 390), in which  $A$  and  $V$  are the absolute and kinematic viscosities and  $m$ ,  $n$ , and  $C$  are constants, agree to within  $\pm 2.5\%$  with results obtained experimentally. The expression is a special case of a general expression due to Slotte. The values of the constants for different oils are given. The values of  $m$ ,  $n$ , and  $C$  increase with the viscosities of the oils,  $C$  being always positive and  $m$  and  $n$  negative. The expression  $\log (\bar{V}_1/\bar{V}_2) = \rho \log \theta_1/\theta_2$  is more convenient for determining the viscosity-temperature relations, where  $V$  may be either the absolute or kinematic viscosity and  $\rho$  denotes  $m$  or  $n$ , depending on the basis of calculation adopted;  $\theta$  is measured in °F. The relation of the time of efflux of an oil from a short-tube viscosimeter to its kinematic viscosity is given by the expression  $V = AT - B/T$ . Since  $V = \theta^n$ , where  $\log w = C$ , then  $AT - B/T = \theta^n$ . This equation gives the relation between efflux time and temperature and agrees with experiment. For the Redwood viscosimeter the values of  $A$  and  $B$  are 0.0026 and 1.715, respectively. The effects of inaccurate temperature and time measurements with the Redwood instrument are shown to be considerable with oils of low viscosity and with more viscous oils at the higher temperatures. A table is given showing the kinematic viscosities of oils and the Redwood efflux times.

T. A. SMITH.

**Detonation as affected by mineral lubricating oils.** R. O. KING and H. MOSS (Engineering, 1931, 131, 1—4).—The action of various typical constituents of mineral lubricating oil blends, including distillates of naphthenic, asphaltic, and paraffin-base crudes and a cylinder stock and spindle oil, on fuels improved in anti-knock value by the addition of "ethyl fluid" or benzol has been determined at normal and high induction temperatures. For a fuel the highest useful compression ratio (H.U.C.R.) of which has been increased by the addition of ethyl fluid, consisting of a 3:2 mixture (by vol.) of lead tetraethyl and ethylene dibromide, the detonation-inducing effect is greatest with the asphaltic-base oil, being represented by a fall of 0.75 compression ratio for 12% of oil in the fuel-oil mixture. The behaviour of the cylinder stock is exceptional and resembles that of rape and castor oils in having no deleterious effect on the detonation-delaying action of ethyl fluid. Raising the induction temperature results in the different oil effects, including that of the cylinder stock, becoming practically equal. Using a 50/50 petrol-benzol mixture, all the oils except the spindle oil were of nearly equal deleterious action, which was less at normal induction temperatures than when the fuel was given an equally high anti-knock value by

ethyl fluid, but on raising the induction temperature it became greater. At induction temperatures of 90° the oils destroy one third of the beneficial effect of benzol as against one quarter of that of ethyl fluid in similar circumstances. For a fuel consisting of cyclohexane alone the deleterious oil effect varied from one half to two thirds of that obtained for a 50/50 petrol-benzol mixture, and taking the combined oil and temperature effects at 50° induction temperature, the H.U.C.R. for the cyclohexane had fallen from 6.0 to 5.55 and for the petrol-benzol mixture to 5.10. With ordinary commercial petrols having a H.U.C.R. of 4.3—4.5 and containing up to 8% of oil in solution, the deleterious oil effect was barely measurable, even in one variety containing 20% of benzol. The deleterious effect becomes evident at higher compression ratios only.

H. S. GARLICK.

**Hydrocyanic acid.**—See VII. **Corrosion of iron by hydrogen cyanide.**—See X. **Carbolic powder.**—See XXIII.

#### PATENTS.

**Vertical retort with crossovers.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,779,958, 28.10.30. Appl., 26.8.24).—The alternate vertical chambers of a vertical retort battery are provided with a bridge block across the charge opening so that combustion gases pass up the side heating flues, through the bridge block, and down the opposite side flue. The coal is preheated by passage over the sides of the bridge block, and the flues work in conjunction with regenerators.

H. E. BLAYDEN.

**Coking oven retort.** J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,780,803, 4.11.30. Appl., 31.7.23. Renewed 27.3.30).—The heating walls of each oven of a battery contain vertical heating flues divided into groups (e.g., two), each group consisting of two sub-groups which are joined by horizontal flues and work in conjunction with separate inflow and outflow regenerators.

H. E. BLAYDEN.

**Heating of stills or coke ovens with hot gases.** CHEM.-TECHN. GES.M.B.H. (B.P. 344,920, 9.12.29. Ger., 7.12.28).—Stills or ovens which are heated by circulating hot gases in helical or circular paths are provided with a rotating propeller placed at a turning point in the path of the gas. Fresh heating gases are introduced before or after the propeller and are mixed with the circulating gas, and an amount of waste gas equal to the fuel gas introduced is by-passed to waste and preheats the fuel gas.

H. E. BLAYDEN.

**Tunnel kiln [for activating charcoal].** O. L. BARNEBEY and M. B. CHENEY (U.S.P. 1,778,747, 21.10.30. Appl., 21.2.25. Cf. U.S.P. 1,541,099; B., 1925, 581).—The kiln, through which the charcoal is conveyed on trays placed on movable carriages, is separated into a lower compartment (provided with coolers) in which the carriages run, and an upper compartment (containing the trays) which receives supplies of heating, cooling, and reactive fluid along its length so that the temperature gradient rises gradually from each end to the middle section of the kiln. Airtight vestibules are situated at each end for charging and discharging the kiln, and means are provided for atomising water into the exit end

of the kiln, and for regulating the return to the kiln of the exit gases. L. A. COLES.

**Carbonisation of carbonaceous material.** E. B. EDWARDS (B.P. 344,455, 6.12.29).—Carbonaceous material which is to be subjected to heat treatment is carried through an oven or furnace in a number of metallic containers. These containers are forced from one end of the furnace to the other, in contact with each other so that the material in them cannot move freely. If desired, the containers may be made to return through the hot zone by means of a cyclic path. Means are provided for filling each vessel and for removal of the semi-coke produced; interlocking devices may be employed to ensure perfect movement through the hot zone. R. H. GRIFFITH.

**Production of carbon for absorption and decolorising.** E. URBAIN, ASST. to URBAIN CORP. (U.S.P. 1,778,343, 14.10.30. Appl., 17.7.28. Fr., 20.7.27).—Carbon is activated by passing over it phosphorus vapour, with or without admixed steam, above 350° and washing out the absorbed reaction products.

H. E. BLAYDEN.

**Manufacture of practically dry carbon.** M. GOBERT (B.P. 345,237, 10.12.29).—Carbon black, obtained by the action of an electric arc on a hydrocarbon oil, is purified by heating to 350–400°, so that it is freed from water and unchanged oil. The necessary heat can be provided by gases formed in the cracking process; if necessary, unsaturated hydrocarbons in this gas may be previously removed and used as additional raw material for production of carbon. Similarly, liquid hydrocarbons driven off during purification may be condensed and returned to the arc.

R. H. GRIFFITH.

**Hydrogenation of carbonaceous materials.** IMPERIAL CHEM. INDUSTRIES, LTD., R. HOLROYD, and C. COCKRAM (B.P. 344,975, 28.12.29 and 23.10.30).—Coal is hydrogenated with the aid of massive catalysts (*e.g.*, a member of the copper sub-group or of group II, in the presence of metals of groups III–VII) in the form of strips or grids of the metals used, one of which may form a coating upon a base metal, *e.g.*, tin plate.

H. E. BLAYDEN.

**Conversion of liquid carbonaceous materials into more valuable products by destructive hydrogenation.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 345,282, 15.10.29).—High-boiling hydrocarbons or tars which contain considerable amounts of asphaltic materials are subjected to a preliminary separation before hydrogenation in the presence of a catalyst, in order to avoid troubles due to deposition of pitch. The treatment consists in heating, preferably in the presence of hydrogen under pressure, so that a distillate alone comes into contact with the catalyst; the residue withdrawn from the apparatus is mixed with fresh material for further use.

R. H. GRIFFITH.

**[Catalysts for] destructive hydrogenation.** H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 345,305, 20.12.29).—Catalysts for production of low-boiling hydrocarbons consist of nickel or cobalt, alloyed or coated with tin; alloys of nickel or cobalt with each

other or with iron can similarly be coated with tin. Such materials can be employed for working with oils in either the liquid or vapour phase. R. H. GRIFFITH.

**Manufacture of water-gas.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 345,858, 29.4.30. Addn. to B.P. 214,544; B., 1924, 549).—In the production of water-gas by treating a moving bed of granular fuel with steam, secondary air is admitted to the space immediately above the fuel bed, during the air blast, so that carbon monoxide and solid particles of fuel are completely burned before leaving the generator. For a vessel 3 m. in diam., and with a grate 7 m.<sup>2</sup> in area, 1400 m.<sup>3</sup> of primary air and 1600 m.<sup>3</sup> of secondary air are required per hr. The latter is supplied from 12 channels in the side walls, the openings in which are placed so that combustion cannot occur on the walls, but attains a maximum at a central point above the fuel bed. R. H. GRIFFITH.

**Manufacture of oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., ASSEES. of J. A. PERRY (B.P. 344,742, 19.6.30. U.S., 27.7.29).—For gasification of large quantities of heavy oils, in association with water-gas manufacture, modifications in plant are made to avoid blockage by carbon or pitch. Twin generators are provided, having a relatively large free space above the fuel bed, which serves as a primary cracking zone. Heat is stored in the fuel bed and in an adjacent superheater, during an air-blow through both generators in parallel; during the steam-run, oil is added to the top of one generator while steam passes in at the base. The resulting gas stream is divided, part of it going through the superheater and part through the second generator, where excessive cracking occurs; after an interval this process is reversed, steam and oil being fed to the second generator. Owing to the flexibility of the method, gas of almost any calorific value can be obtained, and a further modification may include back-run steaming through the superheater.

R. H. GRIFFITH.

**Production of hydrogen by decomposition [separation] under pressure of coke-oven gas.** PATENTVERWERTUNGS A.-G. "ALPINA" (B.P. 345,755, 8.2.30. Ger., 8.2.29).—The yield of hydrogen obtained by separation of coke-oven gas, by cooling under pressure after a preliminary washing with water under pressure, is materially increased by returning to the process the first gaseous fraction which escapes on releasing the pressure on the water used. This gas contains 30–35% of hydrogen.

R. H. GRIFFITH.

**Production of acetylene.** J. FERGUSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 342,771, 8.11.29).—Hydrocarbons other than methane (with or without methane) are passed with large excess of steam through a tube heated above 1000° at such a rate that the time of heating of the gases does not exceed 2 sec. *E.g.*, oil gas passed with 10 vols. of steam at 220 litres per hr. through a 5-mm. tube heated for 22 cm. of its length to 1250° gives 0.37 vol. of acetylene per vol. of oil gas. [Stat. ref.] C. HOLLINS.

**Conversion of methane into liquid hydrocarbons by thermal treatment.** N.V. SILICA EN OVENBOUW

MIL., and C. OTTO (B.P. 344,443, 27.11.29).—An apparatus for subjecting methane to a high temperature consists of a modified coke-oven setting, in which the gas is passed through channels in the brickwork, or in which the ovens themselves are used as contact chambers. Heating is provided by the combustion of gas in suitable adjoining chambers, and the process may be operated so that carbonisation of coal is carried out in the usual way, conversion of the methane obtained being achieved in another part of the same plant. Heat exchange between ingoing and outgoing gases increases the economy of the system, and means for condensation of any liquid products are provided. Hydrogen which is formed as a by-product may be added to that separated in the primary treatment of the carbonisation gases.

R. H. GRIFFITH.

**Gas calorimeters.** S. W. PARR, Assr. to BURGESS-PARR Co. (U.S.P. 1,779,371—2 and 1,778,958, 21.10.30. Appl., [A] 9.3.26, [B] 26.8.27, [C] 19.4.28).—(A) The liquid (e.g., water) used in the calorimeter is preheated to a constant temperature in a storage vessel, and the gas to be tested is heated to the same temperature by passage through heat-interchange coils immersed in the vessel. Equal volumes of gas and liquid, respectively, are made to flow alternately into each of two vessels, from which equal volumes of gas (at constant pressure) and liquid are alternately withdrawn through suitable valves, the gas being burned in a heat exchanger through which the liquid flows. The volumes of liquid and gas used may be measured. The water leaving this heat exchanger passes into a mixing chamber where a stream of air bubbles thoroughly mixes it. Means are provided for observing the temperature of the liquid entering the heat-exchange vessel and of the liquid leaving the mixing chamber, and the corresponding heat of combustion of the gas may be continuously recorded. (B) The liquid in use is continuously circulated from the heat exchanger and mixing chamber over cooling coils through which cold water flows and into the storage tank, where the gas to be tested is brought, by heat exchange, to the same temperature as the liquid. The air for combustion is heated to a constant temperature by passage through a vessel containing the preheated liquid, which, if water is used, ensures constant humidity of the air. (C) The cooling and heating sections of the calorimeter and valve mechanism are mounted between heat-insulating partitions to insulate them from each other and from the atmosphere. Improvements are claimed in the electro-mechanical control of the valves and measuring vessels.

H. E. BLAYDEN.

**Gas calorimeter.** J. P. FISHER, Assr. to BURGESS-PARR Co. (U.S.P. 1,779,373, 21.10.30. Appl., 9.3.28).—The gas and water are metered in fixed proportions to the calorimeter by means of a screw pump in connexion with inlet and outlet chambers containing the gas and water. Pressure regulators maintain the pressures in each chamber constant and equal, and the density of the outlet gas is maintained constant with varying temperature of the water.

H. E. BLAYDEN.

**[Automatically controlled] gas production.** H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,772,641, 12.8.30. Appl., 16.5.21).—Combustible gas is obtained

by passing an air blast through a bed of carbonaceous material at a high temperature; the blast is operated electrically and is controlled by the amount of gas present in a storage holder. Means are also provided, involving a thermostatic device heated by burning a portion of the gas, for passing to waste any low-grade gas that is made on restarting the plant.

R. H. GRIFFITH.

**Oxidation of natural gas.** E. H. BOOMER, Assr. to GOVERNORS OF THE UNIVERSITY OF ALBERTA (U.S.P. 1,776,771, 30.9.30. Appl., 23.7.28).—In order to obtain alcohols, aldehydes, etc. by direct oxidation of natural gas, the latter is mixed with oxygen equivalent to half the volume of hydrocarbons present in the gas. The mixture is compressed to 60–300 atm. and passed over a catalyst at 250–500°; suitable contact materials are zinc and lead chromates, or a salt of a metal of group II or III, together with a salt of a metal of group IV, VI, or VIII.

R. H. GRIFFITH.

**Gas purifier for gas works, coke-producing plants, and the like working with dry [purifier] mass.** J. CHATWIN. From THYSEN'SCHE GAS- u. WASSERWERKE G.M.B.H. (B.P. 345,880, 22.5.30).—The purifying material is supported in a number of double trays, mounted one above another in a vessel which is preferably of tower form, and through the centre of which passes a gas-distributing main having perforations at regular intervals. The crude gas enters the numerous small chambers by this path, penetrates upwards and downwards through the purifying material held by each double tray, and is carried into an annular space formed by the outer shell of the whole vessel. Renewal of the purifying material is carried out by withdrawing the trays, but sulphur can also be extracted, without dismantling, by treatment with carbon disulphide or a similar solvent.

R. H. GRIFFITH.

**Purification of gas from sulphur.** HUMPHREYS & GLASGOW, LTD., Assees. of W. H. FULWEILER and T. K. CLEVELAND (B.P. 345,067, 27.2.30. U.S., 17.5.29).—Organic sulphur compounds are removed from gases by eliminating the hydrogen sulphide in the usual manner and passing them over a zinc oxide catalyst (which may contain promoters such as chromium, vanadium, cerium, etc.) at 300–500° so as to convert the sulphur present into hydrogen sulphide, which is removed. Undecomposed organic sulphur compounds are eliminated by passing the gas through sulphuric acid.

H. E. BLAYDEN.

**Purification of exhaust gases.** L. VECCHIO (U.S.P. 1,775,876, 16.9.30. Appl., 12.12.25).—Waste gases from an internal-combustion engine are purified by contact with ammonia, aqueous alcohol, and a vegetable oil in successive compartments of a bubble-washer. Oily, acidic, or tarry particles are removed in the first stages, and any material that is mechanically suspended is finally held back.

R. H. GRIFFITH.

**Testing the inflammable condition of a mixture of air or oxygen with an inflammable gas or vapour.** N. J. THOMPSON (U.S.P. 1,779,569, 28.10.30. Appl., 14.9.26).—The inflammable properties of mixtures of air and gases or vapours are tested continuously by passing the mixture over a heated surface or suitable



catalyst and making the rise in temperature resulting from combustion to indicate (*e.g.*, by means of a thermocouple and suitable calibration) the heat developed in terms of a fraction of that produced by the combustion of mixtures of hydrocarbons at the lower explosive limit. The last-named amount of heat is stated to be the same for all hydrocarbons. The indicator may be adapted to control automatically the amount of air in the mixture tested.

H. E. BLAYDEN.

**Distillation of coal tar.** BARRETT Co., Assees. of G. T. GAMBRILL, JUN. (B.P. 345,654, 21.11.29. U.S., 30.11.28).—The lower-boiling constituents of a coal tar are continuously removed, to produce a pitch of any desired characteristics, by means of a hot stream of inert gas which is passed through the heated liquid. The necessary apparatus comprises a tube still, where the tar is heated, a vessel for ensuring contact with hot gases, condensers and heat exchangers for cooling the escaping vapours and for preheating the gases, and also means for circulating the permanent gases back to the contact vessel. It is also advantageous to preheat the crude tar, and to supply fresh material as pitch and distillate are withdrawn.

R. H. GRIFFITH.

**Bitumen and tar emulsions.** J. LEBEN, and ORMUL PRODUCTS, LTD. (B.P. 344,562, 18.12.29).—Bituminous materials are mixed with emulsifying agents in such a way that the product will give a good suspension when shaken with water. The agents employed for this purpose are fatty acids and/or resin acids, together with an alkali silicate and <9% of water. The amount of alkali present, beyond that required to neutralise the organic acids, must be 3–5 mols. per mol. of silicic acid.

R. H. GRIFFITH.

**Removal of tar acids and the like from oils.** SOUTH METROPOLITAN GAS Co., and H. STANIER (B.P. 344,614, 31.1.30).—Tar acids are extracted from mixtures which also contain hydrocarbons by treatment with an organic base which is practically insoluble in liquid hydrocarbons. This type of substance is obtained by the interaction of an alkylene oxide and ammonia, or in the form of its hydrochloride by treating an alkylene monochlorohydrin with ammonia; "triethanolamine" is particularly useful. The extract is treated with water, preferably in the presence of a weak acid such as a solution of carbon dioxide, for the separation of the tar acids, and further purification from small quantities of hydrocarbon is accomplished by distillation or by extraction with a petroleum oil. The triethanolamine is regenerated by hydrolysis of its salts.

R. H. GRIFFITH.

**Purification of water containing tar, such as water used in coke manufacture.** P. GRUHL (B.P. 345,579, 6.6.30).—Waste water produced during carbonisation of coal etc. is freed from suspended or dissolved tarry impurities by distillation and fractional condensation. For this purpose a boiler, holding the water to be treated, is connected to a tower filled with a packing of ceramic material or coke; the heavy carbonaceous substances carried by the steam are condensed more easily than is the water vapour, so that they return to the boiler and can be drawn off from time to time. Clean water is condensed at the top of the column, and, by

controlling the rate of heating, a satisfactory separation can always be obtained.

R. H. GRIFFITH.

**Purification of mineral oils, tars, their distillation products, and the like.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,738, 23.1.30).—Previous to the purification of oils by means of high-pressure hydrogenation, asphalts or resins, or substances which would polymerise under the conditions employed, are removed from the raw material by a pretreatment which consists in mixing the crude oil, at temperatures below 100°, with dilute sulphuric, acetic, oxalic, or malonic acid, or in employing polymerising catalysts such as sulphur, potassium bisulphite, zinc chloride, or aluminium chloride. At higher temperatures, preferably 350°, the same results may be achieved by using substances with a large superficial area such as silica gel, pumice, etc. Liquid or gaseous diluents can then be added to the material and the liquid or vapour obtained can be hydrogenated without any loss in activity of the catalyst due to the clogging of its surface.

R. H. GRIFFITH.

**Distillation of hydrocarbons.** BARRETT Co., Assees. of C. G. STUPP (B.P. 345,272, 18.12.29. U.S., 18.12.28).—A still for the treatment of tars, oils, etc. contains a large volume of the material to be distilled, in which a roller is immersed close to one wall of the vessel. The rapid rotation of this roller throws a film of oil against the side of the still and causes a constant fall of spray. Into the centre of this spray is passed a stream of hot gas, which supplies the heat for distillation and carries off the volatile products. At the same time, any chance of overheating of part of the still is precluded, so that no risk of coking can arise. The top of the still is preferably curved to assist in the thorough distribution of the spray; the apparatus can be worked continuously by adding fresh tar and withdrawing pitch.

R. H. GRIFFITH.

**Treating hydrocarbon oils.** C. W. WATSON, Assr. to TEXAS Co. (U.S.P. 1,780,286, 4.11.30. Appl., 1.7.27).—The oil is passed through a battery of series-connected stills provided with fractionating towers, where the oil is exposed to progressively rising temperatures. The vapours evolved from the first few of the stills of the series are condensed and collected, and divided into a smaller and a larger portion. The smaller portion is used as a reflux medium to those fractionating towers corresponding to the stills from which the vapours forming such fractions are obtained. The larger portion is introduced into heat-exchange with other hot products formed in the system and then passed through the first still in the series.

H. S. GARLICK.

**Treatment of [liquid] hydrocarbons.** C. J. FRANKFORTER, Assr. to FRANKFORTER OIL PROCESS, INC. (U.S.P. 1,780,873, 4.11.30. Appl., 27.8.29).—A gas containing hydrogen is activated by a preliminary passage over the exposed heated surface of a catalytic contact material situated in an external conduit, and then passed in a finely-divided condition in contact with an electrically heated contact surface into a hydrocarbon oil undergoing distillation in a cracking or other still. Most of the sulphur and sulphur compounds in the oil

are volatilised and can be separated from the gas by known means prior to its return to the system.

H. S. GARLICK.

**Treatment of [heavy] hydrocarbons.** E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,780,536, 4.11.30. Appl., 5.6.26. Fr., 9.4.26).—Heavy hydrocarbons, under slight pressure, are preheated by waste heat to approx. 200° and then passed through alternative atomisers into one of a pair of cracking chambers, maintained at 500—600°, containing a catalyst which aids decomposition and purification. Atomisation is effected by use of the residual gases of the process supplied under pressure. The resultant products are fractionally condensed and separated from the residual gases, which are collected; the heavy oil fractions are returned to the preheating reservoir. The coke deposited during cracking is burned from the inner surfaces of one of the cracking chambers by passing through a stream of air while cracking is proceeding in the alternative chamber, the heat generated by such combustion being employed to heat the residual gases.

H. S. GARLICK.

**Manufacture of products from hydrocarbons.** H. R. ROWLAND, Assr. to C. & C. DEVELOPING CO. (U.S.P. 1,779,402, 21.10.30. Appl., 21.11.23).—Oil vapours mixed with hydrogen are subjected at about 370° to a high-tension discharge (frequency 25—150 cycles per sec.). The vapours are then fractionated.

T. A. SMITH.

**Cracking of hydrocarbons.** E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,777,307, 7.10.30. Appl., 4.6.29).—Raw lubricating oil stock, mixed with caustic alkali, is brought in direct contact with the vapours from a vapour-phase cracking unit in a dephlegmator. The lubricating fraction is vaporised and recovered in a second dephlegmating tower as a refined distillate. Fractions too light to be included in the petrol are returned to the cracking plant from a third dephlegmating tower.

T. A. SMITH.

**Cracking of hydrocarbons.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. R. MADEL (B.P. 345,664, 24.12.29).—Tars, mineral oils, etc. are sprayed into a cracking chamber maintained at 650—750°, at a pressure not much above that in the chamber, by means of a jet of cracked gases from the process itself. The cracking chamber may be packed with a catalytic material, whilst the material to be treated may be subjected to a separate initial cracking in the liquid phase and may preferably be a middle oil from a destructive hydrogenation process.

H. S. GARLICK.

**Cracking of [petroleum] oils.** A. E. WHITE. From BETHLEHEM STEEL CO. (B.P. 345,005, 18.1.30).—The vapours from the petroleum still are fractionally condensed under pressure and the condensate is removed from the system, instead of returning it to the still. Remaining condensable vapours are condensed at atmospheric pressure. The first-named distillate may be further distilled to give high- and low-boiling fractions, the high-boiling fractions being returned to the petroleum still.

H. E. BLAYDEN.

**Hydrocarbon oil conversion.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,779,465, 28.10.30. Appl., 30.7.27).—Oil is passed through a

series of chambers in which it is brought in contact with the vapours from the preceding chamber and thus preheated. It is then passed through a cracking coil to a reaction chamber, from which vapours can be removed to the preheating chambers. The vapours from the chambers may be passed as desired through a vapour-phase cracking coil and returned to another chamber of the series.

T. A. SMITH.

**Recovery and purification of lubricating oils from mineral oils.** INDIAN REFINING CO. (B.P. 345,632, 16.9.29. U.S., 18.10.28).—A wax-bearing fraction obtained by distilling a wax-containing petroleum oil under reduced pressure is treated with acetone and the mixture allowed to separate. The upper acetone layer, containing most of the wax in solution, is drawn off. The lower layer is treated with a selective solvent having practically complete solvent action on the fraction at approx. 32° and substantially no solvent action on the solid components of the fraction at temperatures below -20°, e.g., approx. equal parts of acetone and benzol, in proportion greater than the volume of the wax-bearing fraction, and the resulting solution is cooled to below -17° in order to precipitate the wax. The dewaxed fraction is subsequently distilled (pressure < 1 atm.).

H. S. GARLICK.

**Breaking of petroleum emulsions.** M. DE GROOTE, L. T. MONSON, and (B, C) A. F. WIRTEL, Assrs. to W. S. BARNICKEL & CO. (U.S.P. 1,780,343—5, 4.11.30. Appl., 21.1.29).—Emulsions of water in petroleum are broken by agitation with (A) neutralised sulphite-cellulose waste liquor and dipropyltetrahydronaphthalene-sulphonic acid, (B) the ammonium salt of  $\alpha$ -methyl-naphthalenesulphonic acid or analogous compound which forms insoluble calcium and magnesium salts, or (C) a compound of the type  $XFRSO_3H$ , where X is a polycyclic aromatic nucleus, e.g., naphthalene, F is a detergent-forming group, e.g., hydroxystearic acid, and R is an alkyl (e.g., propyl) group attached to the nucleus.

A. R. POWELL.

**Manufacture of liquid fuels.** COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 345,864, 2.5.30. Fr., 15.5.29. Cf. B.P. 267,512; B., 1928, 470).—In the process described in the prior patent for obtaining low-boiling hydrocarbons by low-temperature carbonisation of coal, followed by catalytic hydrogenation of the product at ordinary pressures, a second hydrogenation of all the light oil obtained either by distillation, cracking, or primary hydrogenation is effected. The resulting spirit is suitable, without further refining, for use as a motor fuel.

R. H. GRIFFITH.

**Disposal of sulphuric acid materials [from petroleum refining].** C. HINDE (U.S.P. 1,776,070, 16.9.30. Appl., 3.12.28).—Waste sulphuric acid from petroleum refining is sprayed through a heated chamber in such a way that the water is evaporated in the cooler parts and the carbonaceous material burned to carbon dioxide and sulphur dioxide in the hotter parts; finally, in the hottest zones, the remaining sulphur trioxide is caused to dissociate into sulphur dioxide and oxygen. The gases may be passed to a sulphuric acid plant or discharged into the air from a high stack.

A. R. POWELL.

Filtering material [for purifying and bleaching mineral oils]. II. BLUMENBERG, JUN. (U.S.P. 1,777,546, 7.10.30. Appl., 10.8.27).—A finely-ground (20–100-mesh) mixture of 65 pts. of zinc sulphate crystals, 35 pts. of calcium hypochlorite, and 10–30 pts. of kieselguhr is claimed. A. R. POWELL.

Regulation of the gas supply to coke ovens. N.V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., G.M.B.H. (B.P. 346,254, 29.9.30. Ger., 28.9.29).

Heat exchanger. Drying of peat etc. Separation of dry materials. Distillation apparatus. Fractionation tower. Bubble caps. Treating gases. Gas scrubber. Turbine lubrication.—See I. Fibre board.—See V. Oiling of fibres.—See VI. Metal aminocarbonyls. Catalyst.—See VII. Water-proofing concrete.—See IX. Protection of pipes etc. Inhibitor for pickling baths.—See X. Spray oils. See XVI. Removing carbon monoxide from gases.—See XXIII.

### III.—ORGANIC INTERMEDIATES.

Synthesis of methyl alcohol, using copper, zinc oxide, and chromic oxide as catalysts. V. A. PLOTNIKOV, K. N. IVANOV, and D. A. POSPECHOV (J. Chem. Ind., Moscow, 1931, 8, 119–126).—The most active catalyst for the reduction of carbon monoxide by hydrogen contains 70 at.-% Cu, 27.5 mol.-% ZnO, and 2.5 mol.-% Cr<sub>2</sub>O<sub>3</sub>, whilst the greatest yields (98%) of liquid products (chiefly methyl alcohol) are obtained when the proportions 60 at.-% Cu, 35 mol.-% ZnO, and 5 mol.-% Cr<sub>2</sub>O<sub>3</sub> are employed. Increase in the hydrogen content of the reaction mixture above the theoretical amount necessary for the reaction  $\text{CO} + 2\text{H}_2 \rightleftharpoons \text{CH}_3\cdot\text{OH}$  leads to increase in the velocity of reaction and of the proportion of carbon monoxide converted into alcohol and methane, and to decrease in the amount converted into carbon dioxide. The optimal temperature is 265°; above 280° the yield falls owing to increased formation of water, carbon dioxide, and methane, whilst below 250° the velocity is very small. The addition of sodium silicate increases the stability of the catalyst, but decreases its activity.

R. TRUSZKOWSKI.

Detection and determination of methyl alcohol. L. SEMICHON and M. FLANZY (Ann. Falsif., 1931, 24, 80–87).—The present methods of determination based on oxidation to formaldehyde are unsatisfactory, owing to further oxidation to formic acid or even to carbon dioxide. A suggested improvement lies in the use of chromic acid to determine the oxygen equivalent of the solution. This amount of chromic acid is added to another part of the solution for the oxidation of the methyl alcohol to formaldehyde, which takes place to the extent of 32% under the conditions specified in the paper. The formaldehyde is characterised and may be determined colorimetrically by means of the red colour formed with phenylhydrazine and potassium ferri-cyanide in presence of hydrochloric acid.

E. B. HUGHES.

Tests for impurities in ether. II. Acetaldehyde. III. Acetone. G. MIDDLETON and F. C. HYMAS (Analyst, 1931, 56, 238–245).—II. Various

tests for aldehyde show the following sensitivity (parts per million): ammoniacal silver nitrate 1000, potassium hydroxide 40, diazobenzenesulphonic acid 3, Schiff's reagent with the addition of 0.1% of pyrogallol 2, 'Tollens' reagent 1, Nessler's reagent 1, Nessler's reagent diluted (1 : 4) 1. (Cf. B., 1928, 425.)

III. Reagents for acetone show: mercuric chloride and baryta or hydroxylamine and hypochlorite 100, sodium nitroprusside 20–25, acid mercuric sulphate 5, vanillin (Dutch pharmacopœia) 1, Nessler's reagent 0.5 (upper limit 20). Schiff's reagent, modified as above, is recommended as an official test for aldehyde, and the vanillin test is suggested for acetone, in order to combine specificity with sensitivity. T. McLACHLAN.

Volumetric determination of small quantities of formic acid in acetic acid. R. G. C. OLDEMAN (Pharm. Weekblad, 1931, 68, 379–380).—The use of 0.5*N*-alkali for titration of the hydrochloric acid liberated in the Fuchs method (A., 1929, 1323) is troublesome if less than 2.5% of formic acid is present, as the mercuric oxide formed at the end-point is difficult to observe and, further, it obscures the colour change of an indicator. If an excess of sodium chloride be added to the solution mercuric oxide does not separate, owing to complex formation, and by titration (phenolphthalein) 0.2% of formic acid may be determined.

H. F. GILLBE.

[Manufacture of] 1 : 3 : 5-trinitrobenzene ("benzite"). L. DESVERGNES (Chim. et Ind., 1931, 25, 3–17, 291–306).—1 : 3 : 5-Trinitrobenzene is most cheaply and conveniently manufactured by oxidation of 2 : 4 : 6-trinitrotoluene with chromic acid. Other methods studied are reduction of picryl chloride, nitration of *m*-dinitrobenzene, nitration of benzyl chloride. For the manufacture of picryl chloride 1-chloro-2 : 4-dinitrobenzene is dissolved in 2 pts. of 40% oleum and heated for 3 hrs. at 150° with 5.6 pts. of 100% sulphuric acid and 1.5 pts. of 48% nitric acid, the process being an improvement on the Griesheim method (G.P. 78,309); a similar yield and quality are obtained by the Frankland and Garner method (J.S.C.I., 1920, 39, 257 r). The picryl chloride is reduced to 1 : 3 : 5-trinitrobenzene (64% yield) by heating for 1 hr. with 0.32 pt. of copper powder in 3 pts. of 96% alcohol and for a further 0.5 hr. after adding 1 pt. of water. The crude product is crystallised from hot benzene; the mother-liquors contain 2 : 4-dinitro- and 2 : 4 : 6-trinitro-phenol. In Köhler's method (private communication) for the oxidation of trinitrotoluene to trinitrobenzoic acid, the starting material is dissolved in 16.6 pts. of 92% sulphuric acid and 1.58 pts. of powdered potassium dichromate are added in small portions during 3 hrs. at 40° with constant stirring. The washed trinitrobenzoic acid is dissolved in 2–2.2 pts. of water at 90°, and the solution, filtered at 70–75°, is boiled for 5–6 hrs., the trinitrobenzene being skimmed off the surface at intervals; the yield is 73% (calc. on the trinitrotoluene). Oxidation with nitric acid (1 pt. of trinitrotoluene in 6 pts. of 94% sulphuric acid, heated at 190° for 40 hrs. with 5 pts. of 90–92% nitric acid and 4 pts. of 94% sulphuric acid) is dangerous, since trinitrotoluene in presence of air decomposes violently at 209–210°

(Köhler, private communication; Verola, *ibid.*). *m*-Dinitrobenzene, nitrated with 4.5 pts. of 93% nitric acid in 9 pts. of 67% oleum at 80–120° for 8 hrs. gives 50% yield of 1:3:5-trinitrobenzene. A table of setting points for mixtures of di- and tri-nitrobenzenes is given. 1:3:5-Trinitrobenzene has at present no commercial application. Added in small amount (3%) to trinitrotoluene it has a plasticising effect, and the mixture, after stoving at 40–55°, may be compressed to *d* 1.60–1.63; detonation is complete. The solubilities of trinitrobenzene in water at 15° (0.028%), 50° (0.10%), and 100° (0.498%), and in various organic solvents at 17° and 50° [*e.g.*, alcohol (2.09% and 4.57%), pyridine (112.6% and 194.2%)] are determined. With pyridine it yields a compound, not melted at 240°.

C. HOLLINS.

**Mercury catalyst in nitration processes.** A. I. ZACHAROV (J. Chem. Ind., Moscow, 1931, 8, 30–37).—Yields of 60% of *p*-nitrophenol are obtained, using 3–6% nitric acid in the presence of mercuric nitrate (10% of the weight of phenol taken) and maintaining the temperature at –5°. No dinitrophenol is obtained, either in more concentrated acid or using more catalyst. 93% yields of nitrosalicylic acid, containing 80–82% of the *o*-isomeride, are obtained, using the same catalyst, 10–12% nitric acid, and maintaining the temperature below 45°, when the reaction is completed in 12–14 hrs. 85% yields of 1-nitro- $\beta$ -naphthol are similarly obtained, using 2% acid at 35°; the reaction time in this case is 8–9 hrs. 65–70% yields of 1:8-dinitronaphthalene are obtained by passing oxides of nitrogen at 20–25° through a solution in carbon tetrachloride of naphthalene, in the presence of mercuric nitrate. R. TRUSZKOWSKI.

**Bromo-potentiometric titration of  $\beta$ -naphthol-sulphonic acids in presence of each other. III. Mixtures containing oxy-Tobias acid, F acid, and trisulpho-acid.** J. S. HARLAND, S. D. FORRESTER, and D. BAIN (J.S.C.I., 1931, 50, 100–102  $\tau$ ).—Oxy-Tobias acid and F acid have been determined by bromine potentiometrically in 10*N*-sulphuric acid. Trisulpho-acid does not brominate under the same conditions. Those products to be found in sulphonation mixtures of  $\beta$ -naphthol which do not contain a sulphonic group in the 8-position can be determined in the presence of those which do contain a sulphonic group in the 8-position. F acid exists as the tetra-, tri-, and monohydrates and as the anhydrous form, the m.p. of which are given.

**Diphenyl : a starting point in organic synthesis.** G. T. MORGAN and L. P. WALLS (J.S.C.I., 1931, 50, 94  $\tau$ ).—Attention is directed to the possible utilisation in organic synthesis of the hydrocarbon, diphenyl, now available commercially at a moderate price. The following intermediates are obtainable from this source : *o*- and *p*-xenylamines, benzidine, carbazole, 2-amino-carbazole, phenanthridine and its homologues and derivatives.

**Lactic-acetic fermentation.**—See XVIII.

PATENTS.

**Catalytic processes. [Preparation of methyl alcohol].** BRIT. CELANESE, LTD., W. BADER, and E. B. THOMAS (B.P. 345,649, 18.9.29).—Metal oxides or

hydroxides, employed as catalysts, are prepared by electrolysis of the corresponding metal salt solutions, using an *E.M.F.* in excess of that required to deposit the metal. In the preparation of methyl alcohol from carbon monoxide and hydrogen, the catalyst may consist of zinc oxide or hydroxide, with or without the oxide or hydroxide of chromium. W. J. WRIGHT.

**Acetylene. Oxidation of natural gas. Sulphonated hydrocarbons.**—See II. **Metal aminocar-bonyls.**—See VII. **Products from fish-liver oils.**—See XII. **Anethole.**—See XIII. **Organic acids by fermentation.**—See XVIII.

## IV.—DYESTUFFS.

**Possible adulteration of saffron with annatto.** P. BRETIN, P. MANCEAU, and M. NAMDAR (J. Pharm. Chim., 1931, [viii], 13, 369–371).—The colouring matter of annatto may be separated from that of saffron by extraction with benzene, toluene, trichloroethylene, tetrachloroethane, or carbon disulphide, and can be detected in the extract by evaporation and addition of concentrated sulphuric acid to the residue. A greenish-blue coloration is formed. E. H. SHARPLES.

PATENTS.

**Manufacture of azo dyes for wool.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,640, 10.10.29).—*N*-Acylcyclohexylaminoarylamines (cf. B.P. 340,495; B., 1931, 334) are diazotised and coupled with a sulphonated or carboxylated coupling component. The acyl group may be aminobenzoyl (etc.), in which case disazo dyes are obtained; or the acyl group may be removed after coupling. Examples are dyes from the following *N*-acyl-*N*'-cyclohexyl-*p*-phenylenediamines; acetyl derivative  $\rightarrow$  Schäffer acid (orange), R-acid (orange), benzoyl-K-acid (red), tetrahydronaphthalene- $\beta$ -sulphonyl-H-acid (bluish-red), salicylic acid (red-yellow chromed), or H-acid (red; hydrolysed, greenish-blue); benzoyl derivative  $\rightarrow$  *o*-anisyl- $\gamma$ -acid (brown); 2:5-dichlorobenzoyl derivative  $\rightarrow$   $\beta$ -naphthol-8-sulphonic acid (yellow-orange) or 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow); *m*-aminobenzoyl derivative  $\rightarrow$  2 mols. of  $\beta$ -naphthol-8-sulphonic acid (orange).

C. HOLLINS.

**Manufacture of acid disazo dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,807, 19.11.29).—A 4:4'-diaminodiarlylmethane in which the methylene residue may carry an aryl or two methyl substituents is tetrazotised and coupled with 2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid or its monoarylsulphonate, or with 1 mol. of this acid or ester and 1 mol. of another component; the arylsulphonyl group may be introduced after coupling. Examples are: 4:4'-diamino- $\beta\beta$ -diphenylpropane  $\rightarrow$  2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid (red on wool; *p*-toluenesulphonated, bright yellowish-red); 4:4'-diamino-3:3'-dimethoxy- $\beta\beta$ -diphenylpropane  $\rightarrow$  1 mol. of *N*-*p*-toluenesulphonyl-H-acid and 1 mol. of 2:8-dihydroxynaphthalene-6-sulphonic acid (bluish-claret); 4:4'-diamino-di- or -tri-phenylmethane  $\rightarrow$  2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid (red; brighter and yellower on *p*-toluenesulphonylation). [Stat. ref.] C. HOLLINS.

**Colouring foodstuffs.**—See XIX.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Difference in physico-chemical properties of aqueous solutions of [silk] cocoons which show yellow and violet fluorescent colours under ultra-violet light.** H. KANEKO and M. MIYASAKA (Bull. Sericult. Silk Ind., Japan, 1931, 3, 1—3; cf. B., 1930, 857).—Measurements of the physical properties of aqueous solutions of sericin prepared from each type of cocoon shows that cocoons showing a yellow fluorescence have by far the better reeling properties. The effect of storage of the cocoons on the properties of the aqueous extract has also been examined.

R. CUTHILL.

**Cotton for nitration from native [Italian] conifers.** F. C. PALAZZO (Giorn. Chim. Ind. Appl., 1931, 13, 61—68).—Subjection of the crude cellulose of conifers, such as is used for paper-making, to chemical and physico-mechanical treatments readily applied industrially yields a product which contains a very high proportion of  $\alpha$ -cellulose and very little hemicellulosic material and is virtually equivalent to bleached cotton cellulose. The resemblance extends to the external physical characters and to the state of aggregation of the fibres, which are quite similar to those of linters and suggest the name "conifer cotton-waste." The behaviour of this material on nitration also corresponds closely with that of linters, the cellulose nitrate obtained being of the same titre and capable of stabilisation by the same procedure.

T. H. POPE.

**Highly nitrated cellulose.** G. DE BRUIN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 84).—Priority is claimed for the preparation of nitrocellulose containing 14% N. The product, which can be used in the manufacture of smokeless powder, is unaffected by boiling water, dilute caustic soda, and very dilute acids. Nitrocellulose containing 13.87% N loses only traces of nitrogen on boiling for 72 hrs. and is about 2% soluble in ether-alcohol. (Cf. Berl and Rueff, also Lenze and Rubens; A., 1931, 203; B., 1931, 368.)

A. RENFREW.

**Measurement of swelling criteria of wood pulp.** A. NOLL (Papier-Fabr., 1931, 29, 114—116).—The capillary rise of sodium hydroxide solutions of mercerising concentration is found to be complete after 10 min. The mean of the values for strips of pulp  $15 \times 180$  mm., cut in the "length" and "cross" directions, is determined at 20°. The swelling of discs of pulp is measured by means of an apparatus described. The discs are held under slight pressure between nickel plates, one plate being free to move as the pulp swells. The linear expansion is measured directly, being of the order of 250—500%. It is found that swelling is complete in 5 min. The amount of mercerising solution absorbed by the pulp is measured by draining the discs and weighing. Results are corrected for "density" or "bulk" of the pulp examined. The results of several determinations are given.

T. T. POTTS.

**Mechanism of the [paper pulp] beating process.** I, II. K. KANAMARU (J. Soc. Chem. Ind., Japan, 1931, 34, 39—50 B).—The mechanism of the beating process has been investigated by studying changes in the  $\zeta$ -potential and surface conductance of sulphite pulp

during beating. The relation between the former ( $\zeta$ ) and the time of beating,  $t$ , is expressed by the equation  $-d\zeta/dt = a(\zeta - \zeta_\infty)$ , where  $a$  is a constant depending on the conditions of beating, and  $\zeta_0$  and  $\zeta_\infty$  are the values of  $\zeta$  when  $t = 0$  and  $\infty$ , respectively. A similar relation between  $\zeta$  and  $t$  is found on immersing previously dried fibres in water; the velocity coefficient  $a$  is here determined by the conditions of immersion, and a theoretical significance is found for it on assuming that the reaction causing hydration of the cellulose micelles is monomolecular. The relation between the surface conductance,  $\kappa_F$ , at the cellulose-water interface and the time of beating,  $t$ , is given by  $d\kappa_F/dt = k(\kappa_F - \kappa_{F0})/t$ , where  $\kappa_{F0}$  is the value of  $\kappa_F$  when  $t = 0$ , and  $k$  is a constant characteristic of the sample used. When fibres are immersed in water without further treatment the conductance first increases to a maximum with time and then decreases. Surface conductance is valuable as a measure of the degree of beating and as a means of studying the beating mechanism. B. P. RIDGE.

Blood-albumin.—See XIX.

## PATENTS.

**Production of cellulose.** A. FRANZ, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,777,751, 7.10.30. Appl., 16.2.28. Ger., 5.8.26).—Vegetable material is treated below 80° with a concentrated solution of a chlorate (preferably sodium chlorate), together with a moderate quantity of a mineral acid and, where this acid is not hydrochloric acid, in the presence of a catalyst capable of producing hydrochloric acid from chloric acid (copper sulphate); the resulting pulp is washed with water.

F. R. ENNOS.

**Manufacture of  $\alpha$ -cellulose.** L. I. L. DURCHMAN (U.S.P. 1,780,347, 4.11.30. Appl., 8.5.29).—Wood chips are cooked with black and white sulphate liquor containing 0.8—1.0%  $\text{Na}_2\text{SO}_3$  (based on the dry wt. of wood), and the resulting pulp is boiled under pressure (1 atm.) with a solution of caustic soda and common salt, bleached, and treated at ordinary temperature for 3—6 hrs. with a 5—12% solution of sodium hydroxide. The latter is recovered for re-use and the pulp washed and treated with oxalic acid.

F. R. ENNOS.

**Manufacture of viscose.** S. WEIN, Assr. to N. RAFF (U.S.P. 1,779,521, 28.10.30. Appl., 1.3.24).—Cellulose is treated with zinc chloride solution to hydrate it without dissolution. After draining off the excess of solution the hydrated cellulose together with zinc chloride is treated with caustic soda and carbon disulphide to form cellulose xanthate, which is dissolved in alkali to yield viscose, the contaminating sulphur salts being precipitated as zinc salts which are removed by filtration.

F. R. ENNOS.

**Sealing material.** L. RADO (B.P. 344,469, 28.10.29).—Cellulose hydrate film, optionally in association with metal foil, paper, fabric, etc., is used in the manufacture of adhesive tape.

D. J. NORMAN.

**Production of foils from solutions of cellulose compounds, gelatin, etc.** WOLFF & Co. KOMM.-GES. AUF AKT., and M. KÖNIG (B.P. 345,179, 1.7.30. Ger., 22.7.29).—The film-forming solution, e.g., viscose, is fed on to a rotating cylinder, the lower part of which is

immersed in a coagulating bath, and the film is stripped off before the coagulating liquor has penetrated to the pouring base, so that during the whole operation the cylinder retains a coating of uncoagulated viscose.

D. J. NORMAN.

**Manufacture of foils from viscous solutions [containing cellulose, gelatin, etc.].** ZELLSTOFF-FABR. WALDHOF, and A. BERNSTEIN (B.P. 344,982, 2.1.30. Ger., 28.2.29).—The solution is fed into the gap between oppositely rotating rollers, and is then coagulated in the usual way.

D. J. NORMAN.

**Production of artificial filaments or threads.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 344,351, 26.11.29).—The output of the spinning machine is increased without additional apparatus, particularly pumps, by using with each pump a single disc-type jet with its spinning orifices arranged in groups so that two or more threads may be spun at the same time.

D. J. NORMAN.

**Manufacture of artificial fibres and films.** HEBERLEIN & Co. A.-G. (B.P. 345,811—2, 21.3.30. Ger., 21.3.29).—To prevent degradation of regenerated cellulose when precipitated by means of concentrated mineral acids, addition is made to the precipitating bath of (A) formaldehyde, or a condensation product, polymerisation product, or a derivative which yields formaldehyde in the bath; or (B) a pyridine base or a salt thereof which is soluble in the concentrated acid, no stretch being applied to the product. F. R. ENNOS.

**Manufacture of artificial silk.** W. HARRISON (B.P. 344,873—4, 8.11.29).—(A) To improve their tensile strength, threads of regenerated cellulose or cellulose derivatives are treated with reagents (*e.g.*, caustic soda with Glauber's salt and aqueous acetone, respectively) to remove  $\beta$ - and  $\gamma$ -cellulose or their derivatives, and are stretched after treatment with a swelling agent (zinc chloride, acids, or aqueous acetone) which is subsequently removed. (B) To render them flexible and pliable, threads having a high tensile strength, *i.e.*, above 2 g. per denier, produced from cellulose esters other than nitrates are wholly or partly hydrolysed, whereby spaces are created between the cellulose crystallites in the formed threads. F. R. ENNOS.

**Washing and subsequent treatment of threads of artificial silk spun in the form of cakes.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 345,076, 6.3.30. Ger., 12.3.29).—The spun cakes are wrapped or tied up in wide-meshed or reticulated fabrics such as net, flattened, and, after squeezing under water to remove air or gas bubbles occluded between the threads, are suspended on rods which are continuously or intermittently rotated or moved, and subjected to the usual processes for washing and subsequent treatment. F. R. ENNOS.

**Manufacture of flexible gramophone records.** G. HILHOUSE (B.P. 345,875, 16.5.30).—Two discs of celluloid with a sheet of paper between are heated at 120–140° for 30–40 sec. under pressure (1700 lb./in.<sup>2</sup>) without the use of an adhesive, and the blank thus produced is allowed to cool. The paper should be adapted to receive print, and its diameter should not be less than that of the discs. F. R. ENNOS.

**Acetylation of cellulose.** E. ROTH (B.P. 345,069, 28.2.30. Fr., 28.2.29).—The time required for acetylation is much reduced if, as soon as the reaction mixture becomes capable of being pumped, it is forced repeatedly through one or more nozzles to effect a more complete mixing of the reactants than is possible by kneading. The nozzles are preferably of the injector type, a current of air or other gas being used to prevent clogging. It is advantageous to use more glacial acetic as diluent than is usual. Suitable apparatus is described.

D. J. NORMAN.

**Manufacture of cellulose acetate.** KODAK, LTD. FROM EASTMAN KODAK Co. (B.P. 345,800, 11.3.30).—Cellulosic material mixed with a catalyst (sulphuric or *p*-toluenesulphonic acid) is heated with saturated acetic anhydride vapour in a closed vessel from which the air has been substantially removed, the acetic acid formed during the reaction being retained in contact with the material until acetylation is complete. F. R. ENNOS.

**Pulp digester.** E. MORTERUD (U.S.P. 1,777,217, 30.9.30. Appl., 10.10.29).—The digester is provided internally at the middle with two annular sieve tubes, one of which is fixed to the digester wall, the other, of considerably smaller diameter, being fastened thereto by stays. These two sieve tubes are both connected to the inlet side of a circulating pump (preferably mounted inside the digester), the outlet from which is connected to other sieves at the top and bottom of the digester.

D. J. NORMAN.

**Treatment of fibre of non-coniferous origin.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,780,539, 4.11.30. Appl., 11.3.27).—To increase its softness and tear resistance, the fibre liberated, *e.g.*, by the sulphite process, after pretreatment with a lignin-reactive agent (chlorine) is digested at ordinary temperature with a solution of caustic soda, washed, and bleached in alkaline solution. F. R. ENNOS.

**Preparation of cellulose pulp.** H. L. HORN, Assr. to BAGASSE DEVELOPMENT, INC. (U.S.P. 1,780,750, 4.11.30. Appl., 2.12.29).—Cellulose-bearing materials such as bagasse, straw, grass, wood, cornstalks, etc. are simultaneously cooked by introduction of steam into the digester containing the material with water beaten by loose rods within the digester, and electrically and chemically treated by passing a current through the water to which sodium chloride has been added; lime and soda ash are added at a later period of the treatment and the pulp is drained from the liquor for use.

F. R. ENNOS.

**Production of sulphite pulp.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,780,842, 4.11.30. Appl., 5.1.27).—Raw cellulosic material is digested at raised temperature and pressure with sulphurous acid and ammonium sulphate solution. After separation of the pulp, the spent liquor is neutralised, preferably with basic calcium phosphate, and evaporated to *d* 1.16–1.26 for use as fertiliser.

F. R. ENNOS.

**Producing low-density pulp [for explosives].** L. W. BABCOCK, Assr. to HERCULES POWDER Co. (U.S.P. 1,777,710, 7.10.30. Appl., 4.9.26).—The pulp, particularly bagasse, is ground and boiled with water under

atmospheric pressure for  $\frac{1}{4}$ —1 hr. to induce swelling without materially changing the porosity of the fibre. The dried pulp may be used in the preparation of low-density explosives. D. J. NORMAN.

**Making articles of wood and other pulp and drying same.** G. E. COBLENS and A. W. MORRIS (B.P. 344,547, 16.12.29).—The wet pulp is moulded into the required shape between mesh dies and subjected to high pressure to remove as much water as possible and establish intimate contact between the pulp and the dies. The pressure is then released and a current of electricity (preferably 110 volts A.C.) is passed through the moist pulp (the two-mesh dies serving as electrodes) to complete the drying by electro-endosmosis.

D. J. NORMAN.

**Absorbent paper and like felted fabrics.** J. STRACHAN (B.P. 344,846, 5.12.29).—Finely-divided colloidal cellulose, *e.g.*, waste vegetable parchment or cellulose silk that has been ground to pass 60-mesh, is mixed with rag, wood, or esparto pulp and the resulting stock run on a paper machine. The ratio of colloidal cellulose to wood or rag cellulose may conveniently be 2 : 1 for blotting paper and 1 : 1 for stereo-moulding paper.

D. J. NORMAN.

**Manufacture of fibre board.** R. W. CUTLER and J. R. COOLIDGE, III, Assrs. to MONTAN, INC. (U.S.P. 1,779,532, 28.10.30. Appl., 27.5.27).—The strength and water-resistance of fibre boards made from unvulcanised fibre are considerably increased by impregnating the board with a wax which remains hard below 60°, *e.g.*, montan wax. Various methods of effecting the impregnation are described.

D. J. NORMAN.

**Coating for writing tablets, boards, or like articles.** J. B. E. LAMBRECHT, C. DECORTE, and SOC. ANON. CHEMIFAX (B.P. 344,489, 2.12.29).—The coating consists of a cellulose derivative, *e.g.*, nitrocellulose, and/or any substance which is substantially insoluble in water and gives a continuous compact film, *e.g.*, rubber, in admixture with resin, *e.g.*, dammar, and/or acid-resisting agents, *e.g.*, paraffins, barium sulphate, or quartz. The coating solution is prepared by mixing solutions of the cellulose derivative and resin in different organic solvents (preferably volatile) and suspending or emulsifying in this mixture the acid-resisting agents. For very resistant coatings the quantity of resin etc. should exceed that of the cellulose derivative.

D. J. NORMAN.

**Filtration process.**—See I. **Paper.**—See VI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Use of liquid chlorine in the bleaching of cotton fabrics.** BLANCHISSERIE ET TEINTURERIE DE THAONLES-VOSGES (Sealed Note No. 2301, 31.1.14. Bull. Soc. Ind. Mulhouse, 1931, 97, 60—62). Report by C. SUNDER and J. LICHTENBERGER (*Ibid.*, 62—64).—During the bleaching of cotton fabrics by treatment with a calcium or sodium hypochlorite solution the concentration of this liquor is maintained constant by continuous circulation downwards in the form of a shower through a vertical chamber where it meets an upward flow of chlorine gas issuing from a cylinder of liquid chlorine.

SUNDER and LICHTENBERGER cite numerous similar processes. A. J. HALL.

**Blood-albumin.**—See XIX.

## PATENTS.

**Mordanting hair or the like for production of felt.** V. BÖHM (B.P. 345,404, 24.1.30. Austr., 30.1.29).—The skins are treated with a solution containing nitric acid, an oxidising agent capable of acting independently of and simultaneously with nitric acid, *e.g.*, chlorates, perchlorates, permanganates, or persulphates, and a metal salt, *e.g.*, of aluminium, tin, or zirconium in quantity greater than would be necessary for catalytic acceleration of the action of the oxidising agents. A suitable solution contains (g./litre) zinc nitrate 100, nitric acid 100, and potassium chlorate 15—50.

D. J. NORMAN.

**Colouring of paper.** I. G. FARBENIND. A.-G. (B.P. 314,068, 12.6.29. Ger., 23.6.28).—Paper is coloured, either in the mass or by immersion, with the oxazines (?) obtainable by heating diarylaminobenzoquinones in a high-boiling solvent or according to B.P. 313,094 (B., 1931, 337), and sulphonating the product. Examples are: sulphonated oxazines from 3 : 6-dichloro-2 : 5-di-(*N*-ethyl-3-carbazolylamino)-*p*-benzoquinone (blue), 3 : 6-dichloro-2 : 5-di- $\beta$ -naphthylamino-*p*-benzoquinone (violet), and 3 : 6-dichloro-2 : 5-di-(*o*-phenoxyanilino)-*p*-benzoquinone (red).

C. HOLLINS.

**Coloration of cellulose ester materials.** BRIT. CELANESE, LTD. (B.P. 339,997, 10.6.29. U.S., 8.6.28. Cf. B.P. 313,404; B., 1930, 1107).—Cellulose acetate silk (etc.), after hydrolysis to the extent of 56% of the ester, is dyed by means of those dyes commonly used for cellulose ester materials.

A. J. HALL.

**Manufacture of artificial silk of dull lustre from viscose.** I. G. FARBENIND. A.-G. (B.P. 345,405, 24.1.30. Ger., 24.1.29. Addn. to B.P. 312,687; B., 1930, 1063).—An acid amide or a substituted acid amide, *e.g.*, di-*o*-tolyl-diethylcarbamide, which is insoluble in water and in viscose, but soluble in the acid spinning bath, is dispersed in the viscose solution prior to spinning.

D. J. NORMAN.

**Production of white or coloured matt surfaces on ethers or esters of cellulose or its transformation products.** I. G. FARBENIND. A.-G. (B.P. 345,673, 23.12.29. Addn. to B.P. 309,194; B., 1930, 859).—The lustre of cellulose ester or ether yarns and fabrics is reduced by impregnating (instead of printing as described previously) with a solution of urea, then drying, steaming for 20—30 min., washing, and drying. The delustring effect of the urea may be increased by adding water-soluble aliphatic hydroxycarboxylic acids (glycollic and lactic acids), or their salts, together with suitable dyes if desired.

A. J. HALL.

**Oiling and dressing of fibres for spinning.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,710, 16.1.30).—Condensation products, or salts thereof, *i.e.*, esters, amides, or ester-amides of alkylolamines (*e.g.*, mono-, di-, or tri-ethanolamine, hydroxyethylaniline, cyclohexyldiethanolamine, or acid amides thereof, hydroxyethylmorpholine, and the condensation products of these amines with aldehydes) with organic



carboxylic acids, sulphonic acids, or sulphonated carboxylic acids of high mol. wt. are used for oiling wool etc.; they do not form insoluble soaps with hard water, and can be easily removed from the fibre subsequently by washing with water. Suitable acids are those derived from fats or coal-tar oils. A. J. HALL.

**Sizing of textile fibres.** I. G. FARBERIND. A.-G. (B.P. 345,207, 10.12.29. Ger., 24.12.28).—Polyvinyl alcohols are used optionally in conjunction with other sizing agents and oils, fats, waxes, etc. An aqueous solution containing 2–3.33% of the alcohol and 0.2–0.33% of sulphonated oil is suitable. The size is equally suitable for cotton, artificial silk, and wool, and is readily removed by washing with warm water or 0.1–0.2% soap solution. D. J. NORMAN.

**Treatment [sizing] of textile materials.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 344,775, 27.8.29).—Yarns etc. intended for weaving are sized with aqueous solutions of carboxylic esters of cellulose containing ester radicals having as substituents carboxyl and/or hydroxyl groups; such esters may be degraded, if desired, to render them water-soluble. A. J. HALL.

**Manufacture of crêpe yarn.** D. PÄCHT (B.P. 345,396, 18.1.30).—The raw fibres are sized and, if desired, coloured on the way to the twisting machine, and then twisted and wound while still moist. The resulting yarn shows increased strength and elasticity and is free from internal stresses. Suitable apparatus is described. D. J. NORMAN.

**Treatment of silk.** C. H. KIDWELL, ASSR. to KIDWELL & Co., INC. (U.S.P. 1,777,203, 30.9.30. Appl., 28.2.30).—Hosiery knitted from real silk (degummed or not) are made smoother and less subject to "slippage" by treatment for about 1 min. in a solution containing 99 vol.-% of 85% formic acid, 1% of 95% sulphuric acid, and 2.5% of glycerin, followed by washing with dilute ammonia solution. Materials knitted with cotton, when so treated, acquire increased tensile strength and elasticity of fibre. A. J. HALL.

**Gasproof balloon fabric.** C. M. CARSON, ASSR. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,779,389, 21.10.30. Appl., 9.3.29).—Light, strong, closely woven fabric is given 8–10 successive coats of a mixture of vulcanised latex, gelatin, and a softener. A suitable mixture contains gelatin 25 g., glycerin 50 g., vulcanised latex (30% rubber) 100 c.c., water 500 c.c. The fabric is finally treated with a flexible varnish and dusted with aluminium powder. D. J. NORMAN.

**Manufacture of [rubbered] threads of textile material.** J. E. C. BONGRAND and L. S. M. LEJEUNE (B.P. 344,414–5, 25.11.29).—Spun, twisted, or stranded threads or fabrics are impregnated *in vacuo* or under pressure, or by a combination of these two methods, with (A) an aqueous dispersion of rubber, guttapercha, or the like, or (B) a cellulose ether either in aqueous dispersion or in solution in an organic solvent. A further surface-coating of rubber or cellulose ether may be applied if desired. The treated threads show high strength even when made from relatively short fibres,

and in the case of (A) may be used, after vulcanisation, in the manufacture of fabrics for pneumatic tyres.

D. J. NORMAN.

**Dyeing, washing, degreasing, and like machines.** C. CALLEBAUT and J. DE BLICQUY (B.P. 346,542, 8.3.30).

**Artificial silk for treatment in cake form.**—See V. Bleaching composition.—See VII. Compound from fish-liver oil.—See XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Production of nitric acid by the oxidation of ammonia.** J. FAUSER (Chim. et Ind., 1931, 25, 556–569).—The use of increased pressure to accelerate the further oxidation of nitric oxide is twice as economical in power as the use of artificial cooling, and no difficulty now exists in the construction of turbo-compressors of chromium steel for this purpose. In modern practice the oxidation of the ammonia is performed at atmospheric pressure and the gases are then compressed. The efficiency of oxidation of ammonia is, other things being equal, reduced by increase of pressure, and at 4 atm. not more than 91% conversion is attainable and the life of the catalyst is much shorter. The author uses a gas mixture enriched with oxygen, containing 11%  $\text{NH}_3$  and 22%  $\text{O}_2$ , which enables the optimum temperature of 850–950° to be reached without heat exchangers. The removal of heat, which is necessary in an absorption tower, can be effected only by the use of an excessive liquid feed, which adds greatly to pumping costs. It is therefore proposed to replace the towers by horizontal cylinders of resistant steel. The lower part of the cylinder is filled with acid, through which the gas passes in a number of jets; the empty upper part serves as oxidation space. The total volume required is 30% less than with towers packed with Raschig rings. An empirical equation is given for the coefficient of absorption of a cylinder at a given pressure and concentration of acid. At 3 atm. and 20°, 12 cylinders will give 95% absorption and 19 cylinders 99% without the use of alkali. The acid runs in countercurrent to the gas by U-pipes from cylinder to cylinder, and the cylinders are cooled externally with water; the exit gases pass through a turbine to recover their residual energy. Some difficulty has occurred owing to oscillations set up in the acid in the cylinders, which, if in resonance with the general oscillations of the system, not only interfere with absorption, but are mechanically dangerous. Costs are discussed and the conclusion is reached that a pressure of 3.0–3.5 atm. is the most economical. C. IRWIN.

**Concentration of phosphoric acid by submerged combustion.** C. F. HAMMOND (Inst. Chem. Eng., Mar., 1931. Advance proof. 6 pp.).—The author's apparatus has a central vertical burner in a refractory-lined combustion chamber. An intimate mixture of gas and air in theoretical proportions renders combustion complete and the products pass directly into the liquid to be heated. The burner is calculated to have a theoretical flame temperature of 2145° and the inner wall of the combustion chamber (suitably insulated) to be at 1470°. The burner casing is surrounded by

an open-ended cylindrical column within which the exit gases rise and cause a liquid current by air-lift effect, so cooling the casing. The containing vessels are made of chemical stoneware. This leaves only the burner casings as sources of contamination, which must be excluded especially for acid of B.P. quality. After various materials had been tried certain corrosion-resisting steels were found to be satisfactory, the best results being obtained with casings machined from the solid metal rolled, annealed, and pickled. The behaviour of a given steel is not necessarily the same with crude phosphoric acid as with pure acid, but steels have been obtained giving negligible corrosion with either. The increase in sulphate content due to sulphur in the (town's) gas used was only slight. Pure acid can be concentrated with a loss of  $P_2O_5$  in fume of less than 1%. With crude acid yielding an exhaust containing silicon tetrafluoride, flushing of the exhaust main was necessary, but the phosphorus loss was still very small. Crude acid concentration requires good circulation as deposits of calcium sulphate having an eroding effect may otherwise be formed. The yield is thus much better than with standard-type evaporators and the small size of the burner casing, the only part exposed to attack, is an important advantage. The capital cost is competitive as would be the fuel cost if producer gas were used.

C. IRWIN.

**Formation of hydrocyanic acid in the electric discharge.** K. PETERS and H. KÜSTER (Brennstoff-Chem., 1931, 12, 122—127; cf. B., 1931, 429).—By subjecting a 1:1 mixture of methane and ammonia to an electric discharge under reduced pressure (20—40 mm. Hg), and using high gas velocities, yields of hydrocyanic acid up to 70% of theory have been obtained ( $CH_4 + NH_3 \rightarrow HCN + 3H_2$ ). The yield increased with increasing input of electrical energy, the rate of increase corresponding with the increase with rising temperature as calculated from the thermal equilibrium constant. The yield, calculated on the input of methane, increased with diminishing concentration of methane and was practically quantitative for concentrations of less than 15%; with mixtures containing more than 85% of methane and, therefore, less than 15% of ammonia the latter was similarly converted quantitatively into hydrocyanic acid. With high electrical energy inputs the greater part of the excess methane was converted into acetylene, whilst excess ammonia was decomposed into its elements. By passing the discharge through a 3:7 methane-ammonia mixture a good yield of crystalline ammonium cyanide was obtained. In the production of acetylene by this process the addition of some ammonia to the initial gas diminished the amount of carbon deposition without affecting the yield of acetylene, the tar and deposited carbon reacting with the ammonia to give hydrocyanic acid. The mechanism of the reaction is discussed.

A. B. MANNING.

**Volumetric determination of hydrofluoric acid with especial reference to the determination of hydrofluoric and sulphuric acids in etching baths.** F. H. ZSCHÄCKE (Chem.-Ztg., 1931, 55, 246—247).—The acid solution (10 g. weighed in a platinum dish) is diluted to 250 c.c. and 25 c.c. are titrated with *N*-sodium hydro-

oxide, first cold, then hot, using phenolphthalein as indicator. The solution is cooled, treated with 20 g. of sodium chloride and 5 c.c. of 20% potassium thiocyanate, and titrated with *N*-ferric chloride until a pale yellow colour persists. A mixture of 10 c.c. of alcohol and 10 c.c. of ether is then added and titration continued until the ethereal layer, after thorough shaking, acquires a pale pink colour. The first titration gives the total acidity and the second the total fluorine content. Soluble salts are determined by evaporating the solution to dryness and igniting the residue, which is then extracted with hot water to remove alkali sulphates, again ignited and weighed, and analysed as usual for lead, iron, aluminium, and lime.

A. R. POWELL.

**Determination of calcium oxide.** J. ZAWADZKI and W. LUKASIEWICZ (Rocz. Chem., 1931, 11, 154—157).—Calcium oxide present in Portland cement is extracted with a 1:1 mixture of absolute alcohol and phenol, the alcohol is distilled off from the filtrate, and dissolved calcium oxide is titrated with standard acid.

R. TRUSZKOWSKI.

**Determination of calcium carbonate in lime marl.** A. VÜRTHEIM and G. H. JONGEN (Chem. Weekblad, 1931, 28, 206—209).—The method described depends on the reaction  $2NH_4Cl + CaCO_3 \rightleftharpoons CaCl_2 + (NH_4)_2CO_3$ , which, as a result of the decomposition of the ammonium carbonate, is displaced to the right when the mixture is heated. The sample (1 g.) is finely powdered and treated with 300 c.c. of a 5% ammonium chloride solution. The mixture is distilled with a small flame until about 50 c.c. remain, care being taken to avoid the deposition of solid on the walls of the flask. To the distillate are added 200 c.c. of 0.1*N*-hydrochloric acid, and the excess of acid is titrated, using lacmoid as indicator. Since part of the ammonia is derived from decomposition of the excess of ammonium chloride, the residue from the distillation should be filtered and titrated, and the result, after deduction of 0.1 c.c. for the alkali liberated by the glass, deducted from the ammonia titre of the distillate. The results obtained are more concordant than those of Scheibler's method, and the sample used is larger.

H. F. GILLBE.

**Preparation and properties of colloidal aluminium hydroxide for medicinal use.** J. J. L. ZWIKKER (Pharm. Weekblad, 1931, 68, 328—343).—Colloidal aluminium hydroxide, possessing the necessary ease of peptisation in dilute hydrochloric acid, slow power of neutralisation, and the property of gelatinising in more strongly acid solution, and thus resembling "Alucol," may be prepared by Kohlshütter's method (B., 1919, 174 A; 1923, 655 A). A preparation suitable for medicinal use may be obtained by using a 10-fold excess of a 5% ammonia solution containing 5% of ammonium sulphate, stirring the mixture for 30 min., and setting it aside for 4—5 days at the ordinary temperature or for 18 hrs. at 40°. Care must be taken to avoid adsorption of organic material (including alcohol) by the colloid during drying. After drying for 18 hrs. at 40° the material contains about 35% of water.

H. F. GILLBE.

**Content of iron soluble in hydrochloric acid in asbestos for Gooch crucibles.** E. CATTELAÏN (Ann.

Falsif., 1930, 23, 530—531).—The necessity for treating asbestos with hot concentrated hydrochloric acid before use in Gooch crucibles etc. is emphasised. Samples tested contained 1.19—8.55% Fe (soluble in hydrochloric acid) and traces of manganese.

E. B. HUGHES.

**Determination of sulphur in pyrites.** I. A. LEVIN and G. V. RABOVSKI (J. Chem. Ind., Moscow, 1931, 8, 159—161).—0.7—0.8 G. of pyrites is dissolved in 20 c.c. of nitric acid, and the solution is repeatedly evaporated to dryness, with the addition of hydrochloric acid, in order to expel oxides of nitrogen. The final residue is moistened with 3—5 c.c. of dilute hydrochloric acid, diluted to 100—150 c.c., and excess of ammonia is added. The precipitate of ferric hydroxide is filtered off and washed, and the filtrate is diluted to 200 c.c. To 25 c.c. of the solution 7 c.c. of formalin, previously neutralised to methyl-red, are added, and the acidity is determined, using 0.25*N*-sodium hydroxide in the presence of phenolphthalein. The results so obtained for sulphuric acid are practically identical with those obtained gravimetrically.

R. TRUSZKOWSKI.

**Volumetric determination of selenium.** B. ORMONT (Z. anal. Chem., 1931, 83, 338—339).—Polemic against Benesch (A., 1929, 42). Complete extraction of selenium from lead-chamber slimes by digestion with sodium sulphide is impossible as part is retained as lead selenide in the lead sulphide formed by the action of the sodium sulphide on the lead sulphate in the slime.

A. R. POWELL.

**Distillation of phosphorus from tricalcium phosphate in the presence of alkali-metal salts.** N. E. PESTOV and A. E. SPOBNOV (J. Chem. Ind., Moscow, 1931, 8, 129—132).—The amount of phosphorus distilling off at a given temperature when phosphorite is heated with charcoal is greatest if a mixture of the composition: 1 mol.  $\text{Ca}_3\text{P}_2\text{O}_8$ , 3 mols.  $\text{SiO}_2$ , 1 mol.  $\text{Na}_2\text{SO}_4$ , and 0.5 mol.  $\text{Al}_2\text{O}_3$  be used. Inferior yields are obtained if sodium carbonate or chloride is used in place of sulphate.

R. TRUSZKOWSKI.

**Manufacture of bromine in France.** M. KALTENBACH (Chim. et Ind., 1931, 25, 543—555).—The mother-liquors of Stassfurt, of Alsace, and of Zarzis in North Africa each contain 2.0—4.5 g. of bromine per litre. That of the last-named lake can be concentrated up to 8 g./litre by solar evaporation. The water of the Dead Sea contains 4.8 g./litre. During the war period a number of bromine-recovery plants were built in Tunis. These ceased work in 1918, and French bromine production in Alsace only commenced in 1926. The six plants built or building in Alsace are described. Mother-liquor preheated to 75° is passed down a column against a countercurrent of steam and chlorine. The heavy bromine vapour is forced upwards by a reversal of the general direction of the gases in each tray of the column, the inlets being at the top and the outlets at the bottom. The column is packed with perforated tiles, the steam is introduced at the bottom, the chlorine at the third tray up. The stripped mother-liquor is passed through iron turnings to recover any entrained bromine, neutralised with limestone, and treated with a little sodium thiosulphate. The condensed bromine drawn off from the

top is separated from water in a divider and the water returned to the column. The bromine passes into a lead receiver filled with Lunge plates, maintained at 55°, and fitted with a reflux condenser. Here it is freed from chlorine and bromine chloride. The colour of the bromine water gives an indication of the correct adjustment of the chlorine feed. The earlier plants erected gave a yield of about 70%. A more recent design provides a better contact between the gas and the liquid by an elaboration of the distribution channels and the staggering of the perforated tiles. This considerably increases the capacity. Heat exchangers have been abandoned in favour of steam preheating. A chlorine regulator is described in which the chlorine flow is maintained constant by the attendant by observation of a water-level. Absorption of excess chlorine is now performed in a small separate column fed with cold mother-liquor. Bromine is condensed with as little cooling as possible to restrict dissolution of chlorine. Alsatian mother-liquors contain organic matter which necessitates a final purification of the bromine with concentrated sulphuric acid.

C. IRWIN.

**Manufacture of potassium chloride and bromine at the mines of Kali Ste.-Thérèse.** BOURDIEU (Mem. Soc. Ing. Civ. France, 1930, 83, 1010—1028).

**Antimony yellow.**—See XIII.

#### PATENTS.

**Manufacture of chemically pure sulphuric acid.** W. S. ALLEN, Assr. to GEN. CHEM. CO. (U.S.P. 1,777,025, 30.9.30. Appl., 14.11.27).—The sulphuric anhydride concentration of gases from the contact process is increased by bringing them into contact with oleum, the temperature of which is regulated so as to cause liberation of sulphuric anhydride. The enriched gases are treated with steam, the temperature of the reaction preventing the formation of mist, and the gaseous sulphuric acid produced is condensed. The diluted oleum is utilised for absorbing sulphuric anhydride from further amounts of the gases. W. J. WRIGHT.

**Manufacture of concentrated nitric acid.** "MONTECATINI" SOC. GEN. PER L'IND. MINERARIA ED AGRICOLA, Asses. of G. FAUSER (B.P. 345,662, 23.12.29. It., 27.2.29).—Ammonia is oxidised at atmospheric pressure, the nitrogen oxides, after condensation of the aqueous vapour, being passed through a chromium-steel compressor into an absorption tower in countercurrent to water. The dilute nitric acid containing nitrogen oxides passes into another tower, where it is brought into contact with compressed oxygen and converted into concentrated nitric acid.

W. J. WRIGHT.

**Smelting of phosphoric material in an electric furnace.** W. R. SEYFRIED, Assr. to SWANN RESEARCH, Inc. (U.S.P. 1,777,582, 7.10.30. Appl., 14.5.25).—A mixture of phosphate rock and carbon is smelted by means of an arc at the lower part of the furnace, from which air is excluded, and the resulting gases are oxidised in the upper zones of the furnace by the admission of preheated air sufficient to convert the phosphorus into pentoxide and thus preheat the charge. The carbon monoxide-phosphorus pentoxide mixture from the furnace is burned in a combustion chamber through

which pass the pipes leading the air to the electric furnace, and the pentoxide is absorbed in water in a scrubbing tower.

A. R. POWELL.

**Manufacture of phosphoric acid and cyanamide.** T. F. BAILY (U.S.P. 1,775,802, 16.9.30. Appl., 5.2.29).—A 5:3 mixture of phosphate rock and coke breeze is smelted in an arc furnace to produce calcium carbide, carbon monoxide, and phosphorus. The gases are passed through a condenser to remove the phosphorus and the carbon monoxide is used for heating purposes. The phosphorus is burned with just sufficient air to form phosphorus pentoxide and the latter is absorbed in water to form phosphoric acid, leaving almost pure nitrogen; this gas is passed into the cyanamide furnace together with the carbide formed in the first stage, which has meanwhile been finely ground.

A. R. POWELL.

**[Fumigation by] evolution of (A) hydrocyanic acid, (B) hydrocyanic acid and an irritant gas.** H. LEHRECKE, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,196—7, 4.11.30. Appl., 20.10.25. Ger., [A] 27.10.24, [B] 31.10.24).—(A) Hydrogen cyanide is absorbed in a porous substance such as kieselguhr or coke impregnated with a metal salt, *e.g.*, ferric or aluminium chloride, which forms a compound with the cyanide, and with a highly deliquescent salt, *e.g.*, aluminium, zinc, or calcium chloride. On exposing the material to moist air, hydrogen cyanide is evolved owing to the heat generated by the absorption of water by the deliquescent salt. (B) A hypochlorite is added to the mixture so as to produce an irritant gas (cyanogen chloride) which serves as a danger signal when using the material for fumigation.

A. R. POWELL.

**Manufacture of concentrated caustic alkali lyes.** I. G. FARBENIND. A.-G. (B.P. 344,545, 14.12.29. Ger., 15.12.28).—An aqueous suspension of strontium or barium hydroxide and a solid alkali carbonate is heated at the boil for, *e.g.*, 2 hrs.; the alkaline-earth carbonate is collected, washed with water (the washings being used for a fresh charge), and calcined to oxide for re-use in the process.

L. A. COLES.

**Manufacture of ammonium carbamate.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,494, 24.3.30).—Ammonia and carbon dioxide at 5–50° are caused to react in an aqueous solution of urea, which may already contain ammonium carbamate.

W. J. WRIGHT.

**Manufacture of diammonium phosphate.** Soc. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTR., Asses. of E. VOITURON (B.P. 345,451, 25.2.30. Ger., 26.2.29).—Phosphoric acid is treated in a saturator at the b.p. with more than 1 mol. of ammonia per mol. of phosphoric acid, the proportion of ammonia not exceeding that which can be completely fixed. The generated steam and heated air pass to a heat exchanger, and the liquor passes to another saturator into which sufficient ammonia to promote crystallisation of diammonium phosphate is introduced. The crystals are dried in a hydro-extractor, and the mother-liquor is led to the heat exchanger in which it is heated to 100°. It is then mixed in a scrubber with fresh phosphoric acid, which also combines with any ammonia escaping from the

second saturator and condensed steam from the heat exchanger.

W. J. WRIGHT.

**Hypochlorite bleaching composition.** L. T. HOWELLS, Assr. to ELECTRIC SMELTING & ALUMINUM CO. (U.S.P. 1,779,274, 21.10.30. Appl., 24.7.28).—Mixtures of sodium hypochlorite and fluosilicate and of calcium hypochlorite, sodium fluosilicate, and sodium fluoride, either in the powder state or in solution, are claimed.

A. R. POWELL.

**Flotation concentration of phosphatic material.** J. W. LITTLEFORD, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,780,022, 28.10.30. Appl., 3.7.28).—Ground phosphate rock is washed to remove slimes, agitated with a dilute solution of sodium hydroxide, and subjected to flotation in a cascade series of cells using oleic acid and/or soap, with or without pine oil or fuel oil, as a frothing agent.

A. R. POWELL.

**Slaking of lime.** W. H. HENDERSON (U.S.P. 1,780,821, 4.11.30. Appl., 6.4.28).—Ground quicklime is automatically fed into the top of a conical vessel up which is passed a continual stream of steam-heated water from a pipe introduced into the apex of the vessel. A continuous stream of milk of lime overflows from the upper wide end of the vessel, and the impurities settle to the bottom and are withdrawn from time to time.

A. R. POWELL.

**Manufacture of barium sulphide solutions.** H. HOWARD, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,776,029, 16.9.30. Appl., 2.12.26).—The solution obtained by lixiviating with water the product obtained by heating barytes with carbon is treated with a slight excess of barium hydrogen sulphide solution over that required to convert any barium hydroxide in the leach liquor into barium sulphide. The lithopone made from the solution of barium sulphide thus obtained is free from oxides soluble in acetic acid.

A. R. POWELL.

**Separation of metallic compounds [zinc sulphate from copper and iron sulphates].** O. A. FISCHER (U.S.P. 1,779,841, 28.10.30. Appl., 13.7.26).—Roasted sulphide ores containing zinc and copper sulphates with more or less ferric sulphate are heated at 450–600° with carbonaceous material, whereby copper and iron sulphates are decomposed and the zinc sulphate remains unchanged and may subsequently be leached out.

A. R. POWELL.

**Manufacture of metal [iron diethyl-]amino-carbonyls.** P. S. DANNER and J. E. MUTH, Assrs. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,780,643, 4.11.30. Appl., 26.9.27).—Equimolecular quantities of iron pentacarbonyl and diethylamine are heated at 100° under 3 atm., until the pressure falls almost to atmospheric, whereby *iron diethylaminopentacarbonyl*, (?)  $\text{Fe}(\text{CO})_3\text{NHET}_2$ , useful as an anti-knock material for petrol, is formed.

A. R. POWELL.

**Working up beryllium minerals.** I. G. FARBENIND. A.-G. (B.P. 345,902, 28.6.30. Ger., 28.6.29).—The minerals are treated with hydrogen fluoride at above 100°, preferably at 600°, until silicon is expelled as its tetrafluoride; beryllium fluoride is extracted from the residue with hot water, and the hydroxide is precipitated by the addition of ammonia.

L. A. COLES.

**Working up beryllium sublimates containing sulphur and aluminium to obtain beryllium compounds.** H. LÖWENSTEIN (U.S.P. 1,777,122, 30.9.30. Appl., 8.10.29. Ger., 25.9.28).—The sublimates obtained by heating beryllium silicates with carbon and pyrites in an electric furnace are extracted with alkali, and the filtered solution is treated at room temperature with sufficient acid to precipitate beryllium compounds, aluminium compounds remaining in solution.

W. J. WRIGHT.

**Recovery of soluble beryllium compounds [from minerals].** H. LÖWENSTEIN (U.S.P. 1,777,267, 30.9.30. Appl., 18.9.29. Ger., 25.9.28).—Beryl or other beryllium mineral is heated in an arc furnace with iron pyrites and carbon sufficient to convert all the iron and silicon into ferrosilicon and to volatilise the beryllium and aluminium as sulphides partly separable by fractional condensation.

A. R. POWELL.

**Treatment of dolomite and other carbonates of magnesium.** J. BLUMENFELD, Assr. to Soc. de Prod. Chim. des Terres Rares (U.S.P. 1,778,659, 14.10.30. Appl., 13.3.30. Fr., 11.3.29).—Calcined dolomite is digested with an aqueous solution of ammonium chloride sufficient to dissolve only the lime, the ammonia evolved is mixed with part of the carbon dioxide formed in the calcination, and the mixed gases are passed into a solution of calcium chloride formed in the first stage of an earlier operation to regenerate ammonium chloride and calcium carbonate. The undissolved magnesia is separated from the calcium chloride solution and treated with a slight deficiency of hydrochloric acid to obtain pure magnesium chloride solution, which is evaporated to recover the solid salt. The latter is electrolysed to obtain magnesium and chlorine which is converted into hydrochloric acid for use again in the process.

A. R. POWELL.

**Separation of calcium and magnesium chlorides.** A. K. SMITH and C. F. PRUTTON, Assrs. to Dow Chem. Co. (U.S.P. 1,780,098, 28.10.30. Appl., 16.6.25).—The calcium and magnesium chloride mother-liquor obtained in the treatment of brine is evaporated and any tachydrite which separates is removed and treated for the separation of calcium as described in U.S.P. 1,627,068 (B., 1927, 628). The solution, containing the chlorides in the ratio  $\text{CaCl}_2 : \text{MgCl}_2 < 2 : 1$ , is further evaporated at  $120^\circ$  under reduced pressure, whereby the salt  $\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  crystallises. Evaporation is continued until the crystals begin to be contaminated with  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ; the temperature is then raised to  $140^\circ$ , causing the latter salt to dissolve, and the double salt crystals are collected and purified according to U.S.P. 1,768,797 (B., 1931, 246).

A. R. POWELL.

**Dehydration of magnesium chloride.** E. LACELL (B.P. 345,518, 5.4.30. Ger., 5.4.29).—The hydrated chloride is added in small portions to a relatively large mass of the fused chloride, or to a mixture of at least 1 mol. of potassium chloride with 2 mols. of magnesium chloride, maintained at or above  $500^\circ$  by the passage of an alternating current through the material.

L. A. COLES.

**Catalyst of high mechanical strength.** R. WIETZEL and E. WILLFROTH, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,875, 30.9.30. Appl., 29.2.28. Ger.,

3.3.27).—A component of a catalyst for the destructive hydrogenation of distillable carbonaceous materials is mixed into a paste with a soluble magnesium salt, preferably in aqueous solution, the product being heated to expel acid, and further components being then incorporated.

W. J. WRIGHT.

**Manufacture of [adsorptive] metal oxide [catalysts].** R. GRIESSBACH and J. EISELE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,775,640, 16.9.30. Appl., 29.11.26. Ger., 11.12.25).—Thorium, thorium and aluminium, or aluminium and ferric hydroxides freshly precipitated by means of ammonia from a nitrate solution are washed free from soluble salts and beaten up to a thin paste with boiling water; nitric acid is added cautiously until a clear colloidal solution is obtained, which is evaporated to a jelly and on drying yields a glassy mass. Ignition of this mass at  $500$ – $600^\circ$  affords a highly adsorption oxide mixture of great catalytic activity.

A. R. POWELL.

**Improving the properties of activated bauxite and product thereof.** R. C. BENNER and A. P. THOMPSON, Assrs. to Gen. Chem. Co. (U.S.P. 1,778,517, 14.10.30. Appl., 21.1.27).—Bauxite is heated at  $400$ – $450^\circ$  for 4 hrs. to expel water of hydration and, while still warm, soaked in a 20–25% solution of ferric nitrate *in vacuo*, so that the solution enters the pores. After draining off excess solution the mass is again heated at  $400$ – $450^\circ$  for 4 hrs. The product is less friable than untreated bauxite and is highly active in promoting the reaction between sulphur dioxide and hydrogen sulphide.

A. R. POWELL.

**Production of titania [from bauxite residues].** R. M. PALMER (U.S.P. 1,760,992, 3.6.30. Appl., 25.5.26).—The residues from the Bayer process or from the sulphuric acid digestion of bauxite are concentrated on shaking tables and the concentrates, with or without a preliminary heat-treatment, are separated magnetically to obtain an ilmenite concentrate and a rutile tailing, which may be utilised in known manner for the recovery of ferrotitanium and pure titania, respectively.

A. R. POWELL.

**Purification of [fused] aluminium oxide.** J. S. MASIN, Assr. to SWANN RESEARCH, INC. (U.S.P. 1,777,570, 7.10.30. Appl., 1.6.25).—Artificial corundum obtained by fusing bauxite with carbon in the electric furnace is purified by crushing it to the size desired for abrasive purposes and treating the powder with a solution containing 6% of hydrofluoric acid and 16% of sulphuric acid. After washing and drying, the finest particles which are very impure are removed by screening.

A. R. POWELL.

**Preparation of activated adsorptive clay.** W. S. BAYLIS, Assr. to FILTROL Co. of CALIFORNIA (U.S.P. 1,776,990, 30.9.30. Appl., 1.9.27).—Bentonite is treated with sufficient acid to remove one half of the alumina in the principal hydrosilicate, and the product is separated from the water-soluble salts formed.

W. J. WRIGHT.

**Recovery of arsenic, antimony, and tin from mixtures of their salts [obtained in the Harris lead-refining process].** C. A. HEBERLEIN, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,779,272,

21.10.30. Appl., 21.5.26).—The alkaline slag from lead refineries is crushed and digested with cold water to remove the excess alkali and the greater part of the sodium stannate relatively free from arsenate and antimonate. The residue is then leached with hot water, which dissolves all the arsenate and the remaining stannate, leaving the antimonate insoluble. The solution is evaporated and cooled to recover sodium arsenate and the mother-liquors containing stannate are returned to the cold-leaching vats. A. R. POWELL.

**Manufacture of tin chlorides.** W. S. SMITH (U.S.P. 1,777,132, 30.9.30. Appl., 18.6.27).—Chlorine is passed into molten tin and the molten stannous chloride is removed. The vapours of volatile chlorides are fractionated to condense impurities, and the stannic chloride vapour is condensed to a liquid. W. J. WRIGHT.

**Production and employment of oxygen [for use, e.g., in gas-protection appliances].** W. W. TRIGGS. From INHABAD-GES.M.B.H. (B.P. 304,292, 22.10.29).—A mixture comprising approx. 58% of potassium chlorate, 23% of potassium perchlorate, 8% of ferrosilicon, 7.2% of asbestos fibre, 0.3% of cobalt carbonate, and 3.5% of calcium hydroxide, which has been compressed moist and then dried, is ignited by a primer in a container, and the oxygen liberated is passed through a filter before use. L. A. COLES.

**Conversion of carbon dioxide into monoxide particularly in the reduction of iron ores.** B. M. S. KALLING and C. VON DELWIG (B.P. 345,234, 7.12.29. Swed., 10.12.28).—The carbon dioxide or gas containing it passes through a rotating, inclined shaft furnace partly filled with carbon, preferably charcoal mixed with a little coke to increase the electrical conductivity, and the mass is heated locally by two sets of radially disposed electrodes which, by their shape, raise the carbon into the gas space above. L. A. COLES.

**Production of carbon dioxide at low temperatures.** C. A. THOMAS and C. A. HOCHWALT (U.S.P. 1,777,338, 7.10.30. Appl., 16.11.25).—Chlorosulphonic acid is caused to react with sodium hydrogen carbonate in the presence of a solution of calcium chloride. The mixture will generate carbon dioxide at temperatures down to  $-40^{\circ}$ . A. R. POWELL.

**Manufacture of cyanogen chloride.** J. P. SCHMITT-NÄGEL, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,779,984, 28.10.30. Appl., 25.1.27).—A suspension of finely-ground sodium cyanide in six times its weight of carbon tetrachloride containing 0.2% of 95% ethyl alcohol is treated with dry chlorine at  $0^{\circ}$  to  $-10^{\circ}$  until the cyanide is almost, but not quite, completely decomposed. Fractional distillation of the product affords pure cyanogen chloride, or the filtered liquid may be used directly in the manufacture of organic derivatives in which the presence of water is deleterious. A. R. POWELL.

**Obtaining hydrogen sulphide from aqueous solutions of alkali carbonate, bicarbonate, and hydrosulphide.** Soc. ANON. HOLLANDO-BELGE POUR LA FABR. DU COKE, Assecs. of C. OTTO & Co. G.M.B.H. (B.P. 345,873, 13.5.30. Ger., 13.5.29).—After the aqueous solution has been treated with carbon dioxide to give a gas mixture rich in hydrogen sulphide, it is treated with this gas mixture to convert the carbonate

into bicarbonate and hydrosulphide. The solution is then treated with gas rich in carbon dioxide, the hydrosulphide being thus converted into bicarbonate and hydrogen sulphide. W. J. WRIGHT.

**Electrical fine-cleaning of sulphur dioxide-containing gases.** LODGE-COTTRELL, LTD. From METALLGES.A.-G. (B.P. 344,748, 26.6.30).—The reactions, e.g., deposition of sulphuric acid mist and arsenic compounds, initiated in an electrical precipitator are completed in a reaction space following the precipitator, the end of the space containing an electrical or mechanical separator, preferably a filter packed with quartz, coke, etc., to remove precipitated impurities. L. A. COLES.

**[Catalyst for] manufacture of sulphur trioxide.** CALCO CHEM. CO., INC., Assecs. of A. P. BEARDSLEY and N. A. LAURY (B.P. 345,556—7, 13.5.30. U.S., 13.5.29).—(A) A catalyst for the contact process is prepared by repeatedly immersing calcined cellite in ammonium metavanadate solution, drying the product at  $110^{\circ}$ , treating it with rubidium chloride solution, and again drying. (B) Cæsium chloride is substituted for rubidium chloride in the above process. W. J. WRIGHT.

**Production of phosphorus [in ferrophosphorus manufacture].** J. W. WALTON, Assr. to TENNESSEE PRODUCTS CORP. (U.S.P. 1,772,849, 12.8.30. Appl., 22.9.27).—In the production of ferrophosphorus by smelting iron ore and phosphate rock in the blast furnace the former is reduced to metal much higher in the furnace than the zone in which the rock is reduced to phosphorus; hence the gases from the lower zones just above the tuyères have a high content of phosphorus vapour. These gases are withdrawn from the furnace and passed through a cooling system to condense the phosphorus, or they may be mixed with air to convert the phosphorus into pentoxide, which is absorbed in water in a suitable absorption system. A. R. POWELL.

**Liberation of iodine in iodine-containing mineral waters.** F. ELLINGER, Assr. to MIJNBOW EN HANDELSMAATS. "WONOH GEDEH" (U.S.P. 1,775,897, 16.9.30. Appl., 7.3.29. Holl., 10.3.28).—The water is treated with lime to remove bicarbonates as insoluble calcium carbonate and is then neutralised with sulphuric acid. The iodine is liberated from this solution by treatment with the theoretical quantity of permanganate and a little more than the theoretical quantity of sulphuric acid. Absorption of the liberated iodine is effected with activated charcoal. A. R. POWELL.

**Refrigerating medium.** Separation of acidic gases. Solidification of gases. Container for corrosive materials.—See I. Acid from petroleum refining. Filtering material.—See II. Catalytic processes.—See III. Cements and fertiliser materials.—See IX. Metal-hardening baths. Vanadium from its compounds.—See X. Granulation of fertilisers.—See XVI. Potassium salt from distiller's waste.—See XVIII.

## VIII.—GLASS; CERAMICS.

### PATENTS.

**Production of mirror glass.** PILKINGTON BROS., LTD., F. B. WALDRON, and A. HARRISON (B.P. 345,676, 27.12.29).—Glass sheets are conveyed automatically

through apparatus in which they are successively cleaned, coated with silver, coppered electrolytically, washed, and sprayed with paint. L. A. COLES.

**Solution for and method of polishing glass.** I. METH (U.S.P. 1,777,321, 7.10.30. Appl., 24.9.28).—The glass is immersed in a mixture of 7–9 vols. of sulphuric acid, 2–4 vols. of water, and 2–4 vols. of hydrofluoric acid, to which is added 3–5% of oleum and 0.1% of phosphorus pentoxide. A. R. POWELL.

**Manufacture of reinforced glass sheets.** N. B. MATTINGLEY, and LANCEGAYE SAFETY GLASS, LTD. (B.P. 344,454, 6.12.29).—The glass sheets are coated with a composition comprising a cellulose derivative (the nitrate), camphor, a gum (gum benzoin), and a high-boiling solvent of cellulose acetate and nitrate (triacetin); the sheets are then united with an interposed cellulose acetate sheet by compression and heat.

L. A. COLES.

**Reinforcement of fragile [glass and ceramic] articles and materials.** BRIT. CELANESE, LTD. (B.P. 345,758, 11.2.30. U.S., 12.2.29).—The articles are provided with an external coating 0.005–0.03 in. thick, which may be built up in layers of different composition, comprising synthetic resins (phenol- or diphenylolpropane-aldehyde; urea- or toluenesulphonamide-formaldehyde) and cellulose derivatives.

L. A. COLES.

**Strengthened glass and the like.** J. CHAMBERLAIN and H. PERIAM (B.P. 345,688, 2.1.30. Addn. to B.P. 316,755).—Two or more layers of solutions containing cellulose derivatives and/or synthetic resins are applied to glass sheets, the solvents are removed by evaporation after each application, and the whole is united by heat and compression.

L. A. COLES.

**Manufacture of objects from plastic particularly ceramic substances by rotation and pressure.** PORZELLANFABR. KAHLA ZWEIGNIEDERLASSUNG FREIBERG (B.P. 346,148, 31.3.30. Ger., 22.11.29).

**Fused alumina.**—See VII. **Enamelling iron etc.**—See X. **Refractory supports for electric tubes.**—See XI.

## IX.—BUILDING MATERIALS.

**Furnace dust in the manufacture of Portland cement.** O. REBUFFAT and V. MAZZETTI (Giorn. Chim. Ind. Appl., 1931, 13, 74–76).—Furnace dust is composed mainly of two constituents: (1) A more readily fusible silico-aluminate less rich in silica and lime, but richer in alumina, iron, and magnesium than the clinker; this predominates in the coarser portion of the dust. (2) A very fine, distinctly crystalline silico-aluminate richer in silica than the clinker and practically free from magnesia. The concentration of the magnesia in the more fusible part and the marked increase of silica content of the more refractory part are characteristic of furnace dust and are related to the mechanism of its formation. The finer portions of the dust are of uniform type in the different samples of furnace dust examined, and agree in composition with a mixture of 4 mols. of dicalcium silicate and 1 mol. of tricalcium aluminate.

T. H. PORE.

**Cement chemistry in theory and practice.**  
IV. Problem of hardening and its signification in

cement research. V. Chemistry of high-strength cements. VI. Technical aspect of high-strength cements. H. KÜHL (Cement, 1931, 4, 3–15, 161–168, 272–287; cf. B., 1931, 159).—IV. Conclusions in favour of the crystalloid theory of hydraulic hardening of cement, reached by reason of the observation of crystals of monocalcium hydrosilicate and calcium aluminium sulphate, together with an amorphous gel when cement reacts with a considerable quantity of water, are fallacious as no trace of these needles and plates are evident in thin sections of old cement test pieces. Michaelis' view that hardening is due to shrinkage of a gelatinous mass is accepted. It is suggested that a gel is formed on the surface of cement particles which cements the particles together, and the very slow hydration of the grain abstracts water and hardens the gel. Such internal adsorption is not completed for a number of years, as evidenced by the complete change of specimens 20 years old towards polarised light, the almost optically isotropic gel gradually becoming microcrystalline in structure.

V. The transition between sound and unsound cement is gradual with no clearly-defined boundary. Increase in lime up to a certain limit increases both the tensile and crushing values and then tends to cause unsoundness. The mixture should be adjusted so as to form alite and celite in the greatest possible quantities, and with this in view the composition of the clay should be carefully controlled. The highest strengths are obtained from cements either very rich in silica or very low in silica, e.g., %  $\text{SiO}_2$ /% ( $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) = 3–4, or 1.2–1.4 for the low-silica product. The burning and grinding processes should be efficient, and in addition to gypsum other catalysts, e.g., chlorides of the alkali and alkaline-earth metals and of cadmium, aluminium, and iron resulted in increased strength, whilst manganese, tin, and especially zinc, copper, and lead were injurious to strength.

VI. The "ideal" raw mix, theoretically, is given by the expression  $x = [2.8\text{SiO}_2 + 1.1\text{Al}_2\text{O}_3 + 0.7\text{Fe}_2\text{O}_3 - \text{CaO (for clay)}] / [2.8\text{SiO}_2 + 1.1\text{Al}_2\text{O}_3 + 0.7\text{Fe}_2\text{O}_3 - \text{CaO (for limestone)}]$ , in which  $x$  represents the limestone to be used with 1 pt. of clay. A decided danger exists in a too finely-ground clinker, not only on account of the reduced density, but also of the reduced strength. More accurate methods of determining the grade than the usual screening are discussed.

C. A. KING.

**Effect of storage conditions on the properties of cement.** D. A. G. REID (Cement, 1931, 4, 261–263).—The loss on ignition of cement stored in a closed timber shed, in a warm oven representing extreme conditions of heat, and in a cold-storage chamber was in all cases found to increase and may be taken as indicative of the degree of deterioration. No serious damage resulted provided the loss on ignition was less than 2%, or possibly 4% for rapid-hardening cement. The quantity of water required increased and the time of setting fluctuated without any definite trend. The ultimate strength was not so much affected as the rate of attaining that strength. A metal drum was definitely the best container and a wooden cask the next best, but there was little to choose between a four-ply paper and a jute sack.

C. A. KING.



**Wood staining.** A. C. HEINEMANN (Farben-Ztg., 1931, 36, 1259—1260).—The technology of the process is discussed and various types of water-stain in use are described. The drawback of raising the grain of the wood, to which all of these are prone, is avoided by the use of "wax-stains," i.e., partly saponified emulsions of beeswax, carnauba or other hard wax, coloured with potassium chromate, copper sulphate, or acid dyes, together with pigments and fillers if desired.

S. S. WOOLF.

**Calcium oxide.**—See VII. **Blood-albumin.**—See XIX.

## PATENTS.

**Production of hydraulic cement.** G. O. CASE, E. M. ELLIS, and L. H. MONTGUE (B.P. 345,103, 26.9.29).—A mixture of calcium carbonate (limestone) with clinker prepared by burning calcareous and argillaceous material is ground for use as cement, the quantities being such that the proportion of limestone to free calcium oxide in the clinker gives a mixture containing, approx.,  $\text{CaCO}_3 : \text{CaO} = 8 : 1$ , and  $\text{CaCO}_3 : \text{aluminium silicate and free silica} = \text{about } 3 : 1$ . L. A. COLES.

**Production of calcium aluminate cements and fertiliser materials.** H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,778,224, 14.10.30. Appl., 18.6.27).—A mixture of calcium phosphate and alunite (basic potassium aluminium sulphate), preferably with the addition of carbon, is heated at 1200—1600° in an electric furnace to yield a slag which, on grinding, serves as a calcium aluminate cement; potassium compounds and phosphorus volatilise and are worked up to a potash-phosphate fertiliser, or the phosphorus may be recovered separately as phosphoric acid or as the element.

L. A. COLES.

**Filtering of suspended sediments [e.g., cement slurry].** C. PONTOPPIDAN (B.P. 344,412, 21.11.29).—Filtration is facilitated by the addition of an electrolyte comprising a soluble compound of calcium, magnesium, barium, aluminium, iron, potassium, sodium, or ammonium, e.g., calcium sulphate. [Stat. ref.]

B. M. VENABLES.

**Waterproofing of concrete constructions.** G. C. MARTIN (U.S.P. 1,779,481, 28.10.30. Appl., 14.5.28).—Freshly laid wet concrete is coated with a layer of hot asphaltum, whereby the steam produced causes rapid setting of the cement and effects an intimate bond between the concrete and the asphaltum.

A. R. POWELL.

**Coating material [for roof shingles].** J. V. FREEMAN (U.S.P. 1,775,193, 9.9.30. Appl., 8.6.27).—Calcined fireclay from coal-mine dumps is ground coarsely, saturated with a solution of ferrous sulphate or spent pickling liquor, dried, and again calcined to obtain a homogeneous, granular, red material suitable for the manufacture of red tiles. A. R. POWELL.

**Dry kiln [for timber etc.].** F. M. CREIGHTON, Assr. to MOORE DRY KILN Co. (U.S.P. 1,777,255, 30.9.30. Appl., 16.9.25).—A kiln for the continuous drying of timber etc. is operated with positive circulation by means of a fan and suitable ducts, the air currents being upwards at the dry end and downwards at the green end; humidifying sprays, directed in the same direction as the air,

are provided at the dry and green ends. Heating means such as steam pipes are provided in the lower part of the kiln, graduated from nothing at the green end to a maximum at the dry end. Air is vented out at positions that may be varied according to the class of timber being treated. B. M. VENABLES.

**Floor-covering materials.**—See XIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Constitution and formation of the phosphide eutectic in cast iron.** M. KÜNKELE (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., 1930, 12, 23—31; Chem. Zentr., 1931, i, 516).—In grey cast iron a ternary (mixed crystals- $\text{Fe}_3\text{C}-\text{Fe}_3\text{P}$ ) and a binary (mixed crystals- $\text{Fe}_3\text{P}$ ) eutectic occur; the latter is, however, actually a degenerated eutectic of which the third constituent, graphite, is crystallised on graphite flakes already present. A. A. ELDRIDGE.

**Influence of silicon content and speed of cooling on the constitution of the phosphide eutectic in cast iron.** P. BARDENHEUER and M. KÜNKELE (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., 1930, 12, 33—38; Chem. Zentr., 1931, i, 516).—In alloys free from manganese, up to 2% Si the ternary phosphide eutectic is formed under the conditions employed; when more than 2% Si is present the "pseudobinary" eutectic is formed. A manganese content of 0.7% is without influence. Slow cooling or increase in phosphorus content favours the formation of the pseudobinary eutectic.

A. A. ELDRIDGE.

**Effect of speed of cooling on the transformation temperature, structure, and fine structure of iron-carbon alloys.** F. WEYER and N. ENGEL (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., 1930, 12, 93—114; Chem. Zentr., 1931, i, 515).—An apparatus for the quenching of wire in a current of hydrogen is described; cooling speeds up to 5000° per sec. can be attained. A three-dimensional model expresses the relation between carbon concentration, temperature, and speed of cooling. The appearance of a martensitic structure is connected with that of a tetragonal space-centred phase, the axial ratio increasing from unity for pure iron to 1.064 for 1.8% C.

A. A. ELDRIDGE.

**Rôle of hydrogen cyanide in the corrosion of iron.** J. F. G. HICKS (J. Physical Chem., 1931, 35, 893—904; cf. B., 1929, 521).—The relative susceptibility to the action of hydrogen cyanide of various iron compounds which are possible intermediates formed during internal corrosion in gas mains has been determined. The accelerating effect of oxygen and carbon dioxide on corrosion of iron by hydrogen cyanide has also been studied. Hydrogen cyanide is the chief factor in the internal corrosion of gas mains, with carbon dioxide and oxygen next in order of attack; it is not, however, the cause of corrosion, which it cannot initiate. The cause is the dissolution of metallic iron in water. Corrosion by hydrogen cyanide does not differ from other similar processes with respect to mechanism. The final product is Prussian-blue. L. S. THEOBALD.

**Influence of metallic coatings on the mechanical properties of steel undergoing nitridation.** T. YOSIKI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 15, 143—154).—Nitridation of steel was effected by heating the steel at 650° for various time intervals in a stream of ammonia. The tensile strength of the specimens increased with the duration of the treatment, whilst the ductility decreased at first, afterwards becoming nearly constant. The values of these properties were reduced by previously plating the steel with tin. Bending tests showed that coatings of tin, tin-lead alloys, cadmium, and cadmium-lead alloys applied by hot-dipping offer considerable resistance to the penetration of nitrogen into the steel. Some results of impact tests on a chromium-nickel steel are recorded.

E. S. HEDGES.

**Apparatus for the works' determination of the total zinc coating on steel.** GOEKE (Chem. Fabr., 1931, 147—148).—The apparatus comprises a 100-c.c. glass bottle provided with a large hood-shaped glass stopper in which the specimen is placed prior to a test. The stopper carries a short tube with stopcock and a long tube terminating at the bottom of the bottle in a short upward bend and extending horizontally outside the bottle to a measuring burette, above which it terminates in a stopcock. The sample of galvanised steel is placed in the hood, the bottle and long tube are filled with *N*-hydrochloric acid containing 3% of antimony trichloride, and after fixing in the stopper, the specimen is dropped into the acid and the stopcock above the burette opened. The volume of acid displaced when multiplied by 0.00272 gives the weight of zinc present in g.; this weight divided by 7100 times the area of the specimen gives the thickness of the coating in mm.

A. R. POWELL.

**Influence of carbon on the critical points and hardness of 12% chromium steels.** T. MURAKAMI and Y. FUJII (Chikashige Anniv. Vol., 1930, 25—47).—By measurement of the thermal dilatation and magnetic properties of steels containing about 12% Cr the influence of carbon on the critical points has been studied. Increase of the carbon content lowers slightly the temperature of the A1 transformation and raises that of the A2 transformation; the A<sub>1</sub> change is not indicated in the magnetic analysis. The lowering of the critical point or the extent of the Ar<sub>2</sub> transformation depends on the maximum heating temperature and on the rate of cooling; at low rates the lowering is more pronounced in the low-carbon steels, but with rapid cooling it becomes marked with high-carbon steels. The hardness (Brinell) of fully annealed specimens increases as the carbon content is increased from 0.09% to 2.04%. The hardness-quenching temperature curves exhibit maxima at 1000°, independently of the carbon content; the curves for different specimens intersect only at higher temperatures, and there is a maximum hardness at 0.3% C, and a minimum at 1% C when the quenching temperature is 1200°. The cooling rate, provided that it is greater than that of air-cooling, has but little influence. The hardness-tempering temperature curves of the low-carbon steels, quenched from 900°, exhibit maxima at 500°; all the specimens exhibit these maxima when quenched from 1100°, and

a 2.04% carbon steel attains a considerably greater hardness than when in the quenched condition. Photomicrographs of the steels are reproduced and discussed.

H. F. GILLBE.

**Microstructure and hardness of quenched manganese steels.** T. MURAKAMI and K. HATSUTA (Chikashige Anniv. Vol., 1930, 229—236).—Manganese steels containing 2.07—12.44% Mn and 0.38—1.19% C, quenched from 900° and 1200°, have been investigated. The quenched low-manganese steels consist of martensite, and the austenite increases, at the expense of the martensite, as the manganese content increases; free carbide exists in the high-carbon steels quenched from 900°. The transformation austenite→martensite takes place readily during quenching in low-carbon, high-manganese steels, and the austenite content increases with increase of the carbon content owing to suppression of the transformation. Martensite is readily formed by quenching at 1200° specimens which consist of austenite if quenched from 900°. Quenching in oil or water produces practically the same structure. The effects described are attributable, not to the rate of cooling, but to quenching stress, which favours the production of martensite. The Shore hardness number of the quenched steels decreases with increase of the manganese content, especially with high-carbon steels, and is independent of the quenching liquid; the quenching temperature has but little influence on the hardness of the high-carbon steels, but its effect is appreciable with steels containing 0.4—0.7% C.

H. F. GILLBE.

**Determination of silver in steel.** W. BERG (Chem.-Ztg., 1931, 55, 259).—The steel (10—15 g.) is dissolved in 250 c.c. of 1:1 hydrochloric acid, the solution evaporated to dryness, the residue dissolved in dilute hydrochloric acid, and the insoluble silver chloride and silica are collected. The filtrate is treated with hydrogen sulphide, the precipitate dissolved in aqua regia, and the solution made ammoniacal and filtered. The filtrate is joined with the solution obtained by treating the siliceous residue with ammonia and the silver precipitated by making the solution feebly acid with hydrochloric acid. After keeping overnight in a dark place, the silver chloride is collected in a glass filter crucible, washed, dried at 130°, and weighed.

A. R. POWELL.

**Treatment of Hollinger precipitate to produce fine gold.** M. SCOTT (Bull. Inst. Min. Met., 1931, No. 318, 10 pp.).—The mill solution of gold etc. in calcium cyanide is treated with 0.045 lb. of zinc per ton, whereby a precipitate is obtained containing 35% Au, 7% Ag, 1.5% Cu, 9.3% Pb, 14.6% Zn, 11.7% CaO, 4.4% S, 0.8% (Fe, Al)<sub>2</sub>O<sub>3</sub>, and 0.7% SiO<sub>2</sub>. The precipitate is digested with hydrochloric acid and steam, such treatment removing 53% of its weight and leaving a residue containing 75.8% Au, 15.4% Ag, 2.3% Cu, 1.54% Zn, 0.76% S, and small amounts of lead, lime, and silica. This residue is digested with hot sulphuric acid as in the usual parting method, and the resulting sponge gold is washed first with hot water, then with sodium acetate solution to remove lead sulphate, and is finally melted with silica, borax, and manganese dioxide to obtain gold bars of 99.4—99.7% purity. The silver sulphate liquor is diluted with water and the

silver fractionally precipitated with aluminium powder, dried, briquetted, and melted with soda and borax. The filtrate from the silver is treated with more aluminium to precipitate the copper and last traces of silver. All the waste solutions from the process are passed to a settling tank in which the lead collects as sulphate together with silver chloride and a little fine gold. This precipitate is periodically smelted in a small water-jacketed cupola to obtain lead bullion, which is cupelled to obtain doré bars for further refining.

A. R. POWELL.

**Extraction of copper from oxidised ores by cyanide solution.** E. T. DUNSTAN (J. Chem. Met. Soc. S. Afr., 1931, 31, 190—193).—Leaching tests with a crude commercial cyanide solution on Rhodesian oxidised copper ores showed that a good extraction of copper could be obtained, but that cyanide consumption was exceedingly high and no cyanide could be regenerated by electrolysis of the liquors to recover the copper.

A. R. POWELL.

**Cyanidation of a copper-gold ore.** E. H. SMITH (Canad. Chem. Met., 1931, 15, 45—46).—The importance of an adequate addition of lime to an ore pulp containing copper sulphide minerals is illustrated with reference to the cyanidation of an Ontario gold ore; using 6 lb. of lime per ton, the cyanide consumption was 1.5 lb./ton and the zinc consumption for the complete precipitation of gold about 0.1 lb./ton, whilst the melted bullion was 700-fine. With only 1.5 lb. of lime per ton the corresponding figures were 6 lb./ton; 0.22 lb./ton, and 400-fine, respectively, and the cyanide liquors contained such a large proportion of copper that part had to be run to waste in every cycle.

A. R. POWELL.

**Effect of combinations of strain and heat treatment on properties of age-hardened copper alloys.** W. C. ELLIS and E. E. SCHUMACHER (Amer. Inst. Min. Met. Eng., Tech. Pub., 1931, No. 395, 13 pp.).—Hard drawing after heat treatment increased the tensile strength of a copper (96%)–nickel–silicon alloy. The properties were further improved by low-temperature ageing. Copper–cobalt–silicon alloys showed less pronounced ageing effects.

CHEMICAL ABSTRACTS.

**Changing over from acid to alkaline circuit in flotation practice at the mill of the Zinc Corporation, Ltd., Broken Hill, N.S.W., Australia.** R. D. NEVETT (Proc. Austral. Inst. Min. Met., 1930, 343—353).—The ore consists of galena, marmatite, calcite, and quartz with small quantities of pyrite, fluor spar, and rhodonite. Excellent separation of the galena and marmatite from one another and from the pyrites have been obtained by selective flotation in an alkaline circuit of  $p_H$  8.1—8.8. The ground ore pulp after thickening is agitated with 0.8 lb. of "Aero-cyanide" per ton for 6 min., then passed to a series of 14 flotation cells in which the lead is floated with 0.08 lb. of "Vallo C" oil and 0.01 lb. of potassium xanthate per ton. The tailings from these cells are treated with 2 lb. of copper sulphate, 0.8 lb. of sodium aerofloat, and 0.25 lb. of eucalyptus per ton, whereby the marmatite is floated. The average grade of lead concentrate contains 75.7% Pb and 3.6% Zn, and that of the zinc concentrate 50.4% Zn, 8.9% Fe, and 2.2% Pb. Flow-sheets

for the slime and sand sections of the plant are included.

A. R. POWELL.

**All-flotation [of ores] at North Broken Hill, Ltd.** A. LOWRY (Proc. Austral. Inst. Min. Met., 1930, 355—377).—The ore is crushed so that 16% remains on a 40-mesh screen, and the slimes are removed by a series of classifiers. The coarse sand is made into a 2 : 1 pulp with a 0.005% solution of sodium carbonate in a vortex mixer in which is also incorporated 0.04 lb. of eucalyptus-coal tar (1 : 1), 0.02 lb. of potassium xanthate, and 0.06 lb. of sodium silicate per ton of ore. The pulp passes to the flotation cells of the impeller type with additional aeration by means of compressed air passed down the impeller shaft; flotation of galena as a clean product containing 75% Pb is almost instantaneous and the cost of reagents is only 1.6d. per ton of ore (14% Pb) treated. The tailings from the lead cells are treated in an acid circuit for the recovery of their zinc content. The slime overflow from the classifiers is thickened and floated in a separate unit in an alkaline circuit for the recovery of the galena as a concentrate containing 69.3% Pb, 8% Zn, and 45 oz. of silver per ton. The total recovery of lead from both sections exceeds 95%.

A. R. POWELL.

**Determination of calcium and magnesium in roast blende.** L. SCHREIBER (Ann. Chim. analyt., 1931, [ii], 13, 98—102).—The mineral is extracted with hydrochloric acid for  $\frac{1}{2}$  hr. to remove hydrogen sulphide, nitric acid is added, and the whole taken to dryness to render silica and barium insoluble. Successive treatment is given with hydrochloric acid, a large volume of water, hydrogen peroxide, and ammonia. After filtration, if all the lead has been precipitated, calcium and magnesium are precipitated directly as oxalate and phosphate, respectively; otherwise ammonium acetate is added.

T. McLACHLAN.

**Oxygen in aluminium and its determination.** T. HARADA (Chikashige Anniv. Vol., 1930, 237—243).—Grard's method for the determination of oxygen in aluminium has been modified by passing the chlorine, before use, over heated carbon, in order to convert any free oxygen into carbon monoxide, and by purification of the residue, prior to its analysis, with hydrochloric acid. The alumina present in aluminium containing less than 0.38% O is in the form of a solid solution, whilst above this limit it exists merely as an admixture. Melting the metal under ordinary conditions always raises the oxygen content to above this critical value, and microscopical examination thus serves to indicate whether or not a sample has been remelted. Various commercial samples of aluminium contained 0.29—0.48% O.

H. F. GILLBE.

**Selective dissolution of magnesium corrosion products.** L. WHITBY (J.S.C.I., 1931, 50, 83—85 t).—A boiling 20% aqueous solution of pure chromic anhydride may be used to remove rapidly corrosion products from magnesium and many magnesium-base alloys. Solubility of the metal in this reagent may be neglected for most purposes. The activating effect of chloride present in some corrosion products may be inhibited by adding a small amount of silver chromate to the chromic acid used; small quantities of sulphate exert no activating action.

**Anisotropy of zinc sheets.** E. SCHMID and G. WASSERMANN (Z. Metallk., 1931, 23, 87—90).—Rolled zinc has a non-homogeneous structure, so that the physical properties vary according to the orientation of the specimen to the direction of rolling. Thus with sheet 2.27 mm. thick a specimen taken parallel to the direction of rolling had a yield point of 148 kg./mm.<sup>2</sup>, a tensile strength of 28.5 kg./mm.<sup>2</sup>, an elongation of 12%, and a coefficient of thermal expansion, between 30° and 50°, of  $30.5 \times 10^{-6}$ , whereas the corresponding values for a specimen taken transverse to this direction were 18.8, 35.9, 3, and  $18.7 \times 10^{-6}$ , respectively. The surface structure of rolled sheets of zinc comprised a simple fibrous texture with the hexagonal axis in the normal of the sheet with a scattering of 80°. The existence of this surface texture tends to diminish the anisotropic behaviour of commercial rolled sheets of zinc.

A. R. POWELL.

**"Flash-point" test for lead.** R. S. RUSSELL (Proc. Austral. Inst. Min. Met., 1930, 307—319).—The test consists in noting the temperature at which rapid dissolution takes place when the lead is heated with concentrated sulphuric acid at a uniform rate. Tests on numerous specimens of lead of varying degrees of purity and of different ages have shown that the test is of no value in determining the resistance of the metal to corrosion. In general, antimony tends to lower the "flash point" and copper tends to raise it. Samples cut from a piece of lead which had been 22 years in a sulphuric acid chamber gave "flash points" varying from 183° to 313°.

A. R. POWELL.

**Changes in the mixed crystals in heavy-metal alloys.** D. UNO (Chikashige Anniv. Vol., 1930, 215—221).—A discussion, showing the characteristic differences in the hardening processes of steel and light- and heavy-metal alloys.

H. F. GILLBE.

**Testing of [metallic] materials for apparatus to be used at high temperatures and pressures.**

E. FRANKE (Chem. Fabr., 1931, 133—135).—Materials suitable for high temperature and pressure, such as cast steels containing molybdenum or vanadium or 3.5% nickel steel, require much more elaborate mechanical tests than were formerly considered necessary. In particular the effect of ageing at high temperatures must be determined. For this purpose the notched-bar test is employed. Another piece of the metal is then stretched 10% in the cold, immediately raised to 250°, maintained at that temperature for 30 min., and then similarly tested. Determinations of the creeping and stretching limits under the temperature to be used in practice may be necessary to ensure that no deformation can occur. The requirements of suitable testing machines are discussed.

C. IRWIN.

**Effect of rate of bending in notched-bar bending tests [of metals].** J. G. DOCHERTY (Engineering, 1931, 131, 347—350, 414—415).—For all the ductile metals tested, the energy absorbed in bending or fracture increased with the speed of the test and the "impact" effect is merely the increase due to the normal velocity effect. With the less ductile metals the decrease in load began much earlier in the test and the rate was much more rapid, indicating that the cracking effect pre-

dominates. Certain relations between the notched-bar and ordinary tensile tests were noted. C. A. KING.

**Hardness testing of electrodeposits and other thin metallic coatings.** H. O'NEILL (Trans. Faraday Soc., 1931, 27, 41—51).—A machine similar to that described previously (B., 1928, 787) has been found suitable for the indentation and scratch testing of coatings down to less than 0.0005 in. in thickness provided they are not much harder than 400 Brinell. Harder deposits might be tested by the use of a 0.5-mm. hemispherical diamond indenter. Scratch tests are described on nickel deposits from baths of different composition. F. G. TRYHORN.

**Arrangement of the micro-crystals of white tin deposited by electrolysis.** H. HIRATA, H. KOMATSUBARA, and Y. TANAKA (Chikashige Anniv. Vol., 1930, 261—273).—X-Ray and photomicrographic examination of electrolytically deposited white tin shows that at low-current densities and concentration of the electrolyte the crystals grow to a considerable size, but that when both these factors are high the deposit assumes a fibrous structure. The normals to the octahedral faces (111) of the crystals are oriented parallel to a common axis.

H. F. GILLBE.

**Stainless metals.** (SIR) H. C. H. CARPENTER (J. Roy. Soc. Arts, 1931, 79, 557—592).—A lecture.

**Nickel-plating baths.**—See XI.

## PATENTS.

**Hardening baths for iron and steel.** A. STÄHLER (B.P. 345,677, 27.12.29. Ger., 29.12.28).—A cementation bath consists of equal weights of calcium and potassium chlorides, together with 8% of potassium carbonate or hydroxide, 1.5% of potassium cyanide or ferrocyanide, and 4% of finely-divided carbon. The hardening mixture is preferably made into lumps, which are immersed in the bath inside a perforated cartridge. To prevent oxidation and as a heat-insulating medium a layer of calcium cyanamide is kept on the surface of the bath.

A. R. POWELL.

**Enamel coatings for iron and steel articles.** O. HOMMEL (B.P. 344,979, 31.12.29).—The metal surface is wetted, by spraying or dipping, with a solution of a cobalt, nickel, or manganese salt, with or without a binder (e.g., tannin or aluminium silicate), and the article is then heated at a temperature at which the solution reacts with the metal surface to form thereon a thin film of iron oxide and the oxide of the coating metal. The enamel coating is then applied in the usual way to this treated surface.

A. R. POWELL.

**Manufacture of stainless cast-iron articles.** B. VERVOORT (B.P. 344,399, 27.8.29).—The casting is made from an iron alloy containing 10—20% Cr, with or without nickel, and the parts which are to remain stainless are given a high polish.

A. R. POWELL.

**Protection of iron pipes, tubes, bars, etc.** J. & O. KREBBER, GES.M.B.H. (B.P. 345,379, 9.1.30. Holl., 10.1.29).—The pipes are coated with a mixture of asphalt with 10—15% of mineral oil and 40% of benzene, then, after drying, with a layer of millboard strip wound spirally, and finally with a layer of bitumen and talc, stone dust, sawdust, or lime.

A. R. POWELL.

**Removing a lead coating from [iron] articles.** G. DE DUDZEELE (B.P. 345,107, 29.3.30).—The coated article is treated with a saturated solution of sodium chloride containing 5% of sodium hydrogen sulphate, 2–5% of sodium nitrate, 2–5% of sodium acetate, and 1–10% of sulphuric acid. A. R. POWELL.

**Acid inhibitors [for steel-pickling baths].** E. I. DU PONT DE NEMOURS & Co. (B.P. 344,419, 28.11.29. U.S., 21.6.29).—A solution in concentrated sulphuric or hydrochloric acid of the condensation product of an amine, *e.g.*, aniline or toluidine, an aldehyde, *e.g.*, butaldehyde, and carbon disulphide is claimed. A. R. POWELL.

**Protective compound [inhibitor for steel-pickling baths].** L. J. CHRISTMANN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,779,961, 28.10.30. Appl., 18.9.29).—The condensation product of aldehyde-ammonia and phenylthiocarbamide is claimed. A. R. POWELL.

**Cleaning and preservation of metallic [iron or steel] surfaces. [Inhibitor for pickling baths.]** J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,779,787, 28.10.30. Appl., 2.8.28).—The inhibitor claimed is made by heating creosote oil with sulphuric acid at 120–125°. A. R. POWELL.

**Preventing the dissolution of metal [iron] in acids [pickling baths].** J. H. GRAVELL (B.P. 344,404, 26.10.29).—A solution of a condensation product of an aldehyde with ammonia, an amine, or a derivative of thiocarbonic acid is added to the pickling bath in such a way that it forms therein a finely-divided suspension which is stabilised by the addition of a protective colloid, *e.g.*, sulphite-cellulose waste liquors, starch flour, gums, etc. [Stat. ref.] A. R. POWELL.

**Alloys [low-carbon special steels].** (Sir) R. A. HADFIELD (B.P. 344,500, 5.12.29).—Steel resistant to oxidation at high temperatures and having a high creep limit contains less than 1–1.5% each of carbon, chromium, and copper and less than 3% Mo, the copper content being less than the chromium content; *e.g.*, the steel may contain 0.09–0.12% C, 0.43–0.58% Cr, 0.34–0.38% Cu, 0.9% Mo, 0.05% Si, and 0.15–0.35% Mn. A. R. POWELL.

**Steel [alloy for rails etc.].** P. P. MARTHOUREY (B.P. 344,822 and Addn. B.P. 344,893, [A] 11.12.29, [B] 13.12.29. Fr., [A] 11.12.28).—(A) The steel contains 0.2–0.6 (0.325)% C, 0.2–4 (3.1)% Ni, 0.4–2.5 (1.4)% Cr, 0.3–2.0 (0.4)% Mn, 0.1–0.5 (0.225)% Si, 0.1–1.5 (0.2)% Mo or W, and the usual small amounts of phosphorus and sulphur. Up to 0.5% V, 0.1% Ti, and/or 0.3% Cu may also be added. (B) The nickel content is raised to 6% and the manganese content reduced to 0.3%; the surface layers are hardened by heating rapidly with a non-oxidising oxy-acetylene flame followed by air-cooling. A. R. POWELL.

**Alloy steel.** J. A. NELSON, Assr. to BARBER-COLMAN Co. (U.S.P. 1,778,226, 14.10.30. Appl., 14.9.25).—Steel for cutting tools, files, etc. contains 1.05–1.15% C, 7.5–8.5% Cr, 8–9% W, 1–1.5% V, 7–9% Co, 0.2–0.3% Si, 0.2–0.3% Mn, and less than 0.03% S and 0.03% P. The tools are soaked at 1040°, quenched, and tempered at 535°. A. R. POWELL.

**Compositions of matter for making tools and the like.** FIRTH-STERLING STEEL Co., Assees. of E. B. WELCH (B.P. 345,167, 11.6.30. U.S., 27.9.29).—Tungsten carbide, fused zirconia, or carborundum powder is mixed with cobalt powder which has been coated with a film of copper or chromium or with films of nickel or copper and of chromium, the mixture is ground together until homogeneous, pressed into the desired shape, and heated in a non-oxidising atmosphere at 1500–1600° until thoroughly sintered. A. R. POWELL.

**Manufacture of [cutting] tools.** F. KRUPP A.-G. (B.P. 345,171, 14.6.30. Ger., 6.11.29).—Between the steel shank and the cutting edge of hard alloy is inserted a sheet of molybdenum, nickel-iron, or chromium-iron alloy having a coefficient of expansion equal to that of the hard alloy and the whole is welded together. A. R. POWELL.

**Manufacture of iron-nickel alloys [for loading coils].** GEN. ELECTRIC Co., LTD., G. C. MARRIS, G. R. POLGREEN, and S. V. WILLIAMS (B.P. 345,736, 22.1.30).—A mixture of nickel and iron oxides in the desired proportions (*e.g.*, 80:20) is ground finely and reduced at 850° in hydrogen. The resulting slightly sintered sponge is pulverised, washed with 5% hydrochloric acid at 50°, then with cold water, dried, and reheated at 1000° for 1 hr. in a reducing atmosphere. The product is ground in a ball-mill to obtain smooth even particles which are mixed with 2.5% of fine talc powder, annealed at 900°, and formed into magnetic cores in known manner. A. R. POWELL.

**[Iron-aluminium] alloy and articles [furnace parts] manufactured therefrom.** H. E. COLEY, LTD., and C. J. NEWTON (B.P. 345,693, 4.1.30).—An alloy of 25% Al and 75% Fe with less than 0.1% C is claimed for use in the construction of metal retorts and furnace parts which come into contact with gases containing sulphur compounds. A. R. POWELL.

**Nitrogenising of alloys of iron or steel.** F. KRUPP A.-G. (B.P. 345,238, 16.12.29. Ger., 20.12.28).—The ammonia atmosphere in the usual nitrogenising containers is kept in violent motion by means of a revolving screw or the like. A. R. POWELL.

**[Nitrogenisation] treatment of iron and steel.** M. HIRSCH (U.S.P. 1,772,866, 12.8.30. Appl., 2.7.27).—Steel, especially molybdenum steel, may be case-hardened by heating it with carbamide, cyanamide, hexamethylenetetramine, or other nitrogenous compound at a temperature sufficient to cause the liberation of nascent nitrogen. A. R. POWELL.

**[Nitrogen case-]hardening of [chromium or manganese] alloy steels.** H. SUTTON, A. J. SIDERY, and B. EVANS (B.P. 345,659, 19.12.29, 23.6. and 25.8.30).—The surface of the metal is coated with copper, silver, platinum, cobalt, molybdenum, or arsenic by electroplating, spraying, or sherardising methods, and the metal is then heated at 450–650° for 4–90 hrs. in a current of ammonia. A. R. POWELL.

**[Reagent for the] concentration of [copper] ores by flotation.** E. K. BOLTON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,000, 28.10.30. Appl.,

30.11.25).—A mercaptothiazole, especially 1-mercapto-3-methylbenzthiazole or  $\alpha$ -naphthathiazole, is claimed.

A. R. POWELL.

**Selective agent for concentrating ores.** R. LUCKENBACH (U.S.P. 1,780,024, 28.10.30. Appl., 21.12.28).—A mixture of 7 pts. of a heavy petroleum oil of an asphalt base and 2 pts. of horse-grease oil is claimed as a flotation agent.

A. R. POWELL.

**Obtaining metals from sulphide ores.** C. GOETZ (B.P. 345,351, 23.10.29).—The coarsely-ground ore is mixed with finely-divided bituminous coal and a catalyst, *e.g.*, lime, hematite, or ferrous sulphate, and the mixture is briquetted with the aid of oil or tar. After drying, the briquettes are broken into coarse pieces which are heated at 600–1000° for several hrs., whereby the sulphides are reduced to metals with the evolution of organic sulphides, hydrogen sulphide, and sulphur. The metals are then recovered from the mass by gravity concentration or magnetic separation. (Cf. B.P. 342,293; B., 1931, 353.)

A. R. POWELL.

**Recovery of metals from metalliferous materials, *e.g.*, ores.** MEYER MINERAL SEPARATION CO., ASSEES. OF R. F. MEYER (B.P. 344,492, 3.12.29. U.S., 28.12.28).—Roasted sulphide ores of zinc, copper, cobalt, or nickel or roasted manganese ores are treated in a damp, pulverulent condition with sulphur dioxide and/or chlorine to convert the above metals into compounds soluble in water, which are then leached out in the usual way. The residue, if desired, is in a suitable condition for treatment with cyanide for the extraction of gold and silver.

A. R. POWELL.

**Manufacture of metal [coarsely crystalline copper].** J. G. DONALDSON and H. L. COLES, ASSRS. TO GUARDIAN METALS CO. (U.S.P. 1,775,159, 9.9.30. Appl., 21.12.26).—Molten copper is deoxidised with 20% of cuprosilicon, and 0.125–0.25% of finely-ground "alberene stone" (an aluminium magnesium silicate) is thoroughly incorporated with the molten metal, which is then allowed to solidify at such a rate that a coarsely crystalline structure is obtained with the silicate deposited in the amorphous material between the grains.

A. R. POWELL.

**Production of feathery copper powder.** W. KOEHLER, ASSR. TO KOEHLER CHEM. CO. (U.S.P. 1,777,371, 7.10.30. Appl., 22.1.30).—A solution containing 0.5–6% Cu, 0.5–6% of a metal more electropositive than copper, and 0.5–10% of free acid is electrolysed at 25–50° with a cathode current density of 40–240 amp./ft.<sup>2</sup>, and the deposited, voluminous, feathery deposit of copper is removed by brushing at frequent intervals. The preferred bath contains 2% Cu and 2% Na or Zn as sulphates with 6% of free sulphuric acid, and is operated at 30° with a cathode current density of 70 amp./ft.<sup>2</sup>

A. R. POWELL.

**[Copper-nickel] alloy.** R. WELLESLEY (B.P. 345,445, 24.2.30).—An alloy made by melting 25–46 oz. of copper, 16–30 oz. of nickel, 6–14 oz. of zinc, 3.5–7.5 oz. of 20% manganese-chromium, 1.5–4 oz. of 20% manganese-copper, 1–1.5 oz. of 15% molybdenum-nickel, and 1–2.5 oz. of an alloy containing 45.5% Sn, 32% Al, 4.5% Cd, 13.5% Mg, and 4.5% Bi is claimed.

A small quantity of vanadium, silicon, and/or boron may also be added.

A. R. POWELL.

**Production of coked metalliferous agglomerates [zinc oxide briquettes].** NEW JERSEY ZINC CO., ASSEES. OF E. H. BUNCE (B.P. 344,569 and 344,570, 23.12.29. U.S., [A] 30.7.29, [B] 11.9.29).—(A) Zinc oxide material and bituminous coal are formed into briquettes containing 6–12% of moisture, and these are brought into contact with a stream of hot gases, so that the surface layers are rapidly heated to about 800°, at which temperature they are promptly converted into a shell of coke; heating is continued until coking is completed throughout the briquette. (B) Heating is effected by large volumes of hot non-oxidising gases passed through the briquettes in a relatively narrow shaft furnace or in a relatively thin bed in a coking chamber, gas and briquettes moving in either case counter-current to one another.

A. R. POWELL.

**Refining of zinc.** F. R. KEMMER, ASSR. TO AMER. CYANAMID CO. (U.S.P. 1,779,973, 28.10.30. Appl., 2.5.28).—Electrothermal zinc is heated at 700° in an electric resistance furnace, whereby it separates into a lower layer of lead, a middle layer of iron-zinc alloy, and an upper layer of relatively pure zinc. This zinc is tapped off and cooled to 450° to allow more iron-zinc alloy to separate, and the purified zinc is then redistilled in an electric furnace. The iron alloy crusts are returned to the electrothermal reduction furnace and the residues from the redistillation of the zinc are worked up for the recovery of gold, silver, and copper.

A. R. POWELL.

**Annealing and zinc-coating ferrous wires, strip, etc.** W. H. POTTER (B.P. 345,598, 19.7.30).—The wire or strip is passed through a molten mixture of 10% of calcium cyanamide, 35% of sodium carbonate, and 55% of sodium chloride floating above a bath of lead at 800°, thence through an acid pickling bath, a water rinsing bath, an alkaline flux, and finally through zinc at 450°.

A. R. POWELL.

**Aluminium alloy.** METAL CASTINGS, LTD., and A. H. NICHOLSON (B.P. 345,823, 1.4.30).—The alloy contains 3.5–4.5% Cu, 0.8–1.3% Ni, 1.2–1.7% Mg, 0.25–1% W, and 0.25–1% V, the sum of the nickel, tungsten, and vanadium contents being 1.8–2.3%.

A. R. POWELL.

**Casting of magnesium and its alloys.** H. KELLEY, ASSR. TO DOW CHEM. CO. (U.S.P. 1,772,490, 12.8.30. Appl., 13.1.26).—The mould cores are saturated with sulphur dioxide by allowing sulphur to burn in the core-baking oven. The presence of this gas in the mould prevents burning of magnesium or its alloys in the sprue or on the surface of the casting.

A. R. POWELL.

**Provision of insulating coatings on aluminium and its alloys.** A. R. DUNTON, A. A. POLLITT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 345,437, 17.2.30).—The metal is boiled in a 0.5% solution of barium hydroxide containing in suspension 0.5% of barium, calcium, or strontium sulphate, whereby it becomes coated with an adherent film which can be varnished or impregnated with oil to improve its resistance to corrosion and electrical insulating properties.

A. R. POWELL.

**Recovery of tin from scrap tinned metal and like materials carrying recoverable tin.** H. WADE, J. W. HINCHLEY, and J. D. PARSONS (B.P. 344,899, 13.12.29. Addn. to B.P. 327,997; B., 1930, 618).—A solution of lead hydroxide in hot sodium hydroxide solution is used as the detinning solution. The deposited lead is redissolved in acetic acid and the solution treated at 70–95°, with sodium hydroxide to obtain lead hydroxide for use again. The sodium stannate solution is treated with lime to regenerate sodium hydroxide for use as a precipitant for the lead acetate solution.

A. R. POWELL.

**Refining of bismuth.** T. H. DONAHUE, Assr. to CERRO DE PASCO COPPER CORP. (U.S.P. 1,778,292, 14.10.30. Appl., 8.7.29).—A stream of chlorine is passed through molten bismuth, whereby lead and other impurities are converted into a chloride slag. When clouds of bismuth chloride vapours begin to be evolved the chlorine is stopped, the slag removed, and the metal stirred with molten caustic soda to remove adsorbed chlorine.

A. R. POWELL.

**Lead alloy, particularly for cable sheathings.** M. THIELERS (U.S.P. 1,779,784, 28.10.30. Appl., 15.3.28. Swed., 18.3.26).—An alloy of lead with 1% Sb and up to 0.5% (preferably 0.2%) Bi is claimed to be resistant to chemical action and intercrystalline fatigue.

A. R. POWELL.

**Recovery of vanadium from ores or compounds.** B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,779,856, 28.10.30. Appl., 29.10.27).—The ore (etc.) is ground finely and mixed with water and the pulp treated with chlorine or sulphur chloride to dissolve the vanadium.

A. R. POWELL.

**Manufacture of [precious metal] catalysts.** G. SIEBERT GES.M.B.H. (B.P. 345,890, 6.6.30. Ger., 7.6.29).—Nets, gauzes, or plaited work made of platinum, silver, gold, nickel, copper, or alloy wires or strips plated or otherwise coated with rhodium are claimed.

A. R. POWELL.

**Production of tarnish-resisting silver and silver-plate.** D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,779,809, 28.10.30. Appl., 5.11.23).—The article is electroplated with alternate layers of silver and a metal which when alloyed with silver increases its resistance to tarnishing, then annealed in a non-oxidising atmosphere at 220° to produce diffusion of the metals into one another, and finally worked hot or cold. Alternatively, the alloying metal may be caused to diffuse into the silver by a process similar to the sherardising process.

A. R. POWELL.

**[Palladium] alloys for electrical contacts.** E. F. KINGSBURY, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,779,602—3, 28.10.30. Appl., [A] 16.5.24, [B] 2.3.26).—The alloy comprises (A) 60% Pd and 40% Cu, or (B) 70% Pd and 30% W. A small part of the palladium may be replaced by (A) zinc or (B) nickel.

A. R. POWELL.

**Preparation of chromium-plated ferrous articles.** V. M. VERTUCCI (U.S.P. 1,780,213, 4.11.30. Appl., 7.10.26).—The article is heated to dull redness, allowed to cool slowly, pickled in 20% hydrochloric acid at 85°,

washed, rinsed in sodium hydroxide solution, cleaned cathodically in 10% sulphuric acid with 150 amp./ft.<sup>2</sup> for 5 min., and plated directly in the usual chromium bath.

A. R. POWELL.

**Treatment of cast iron. Ferrous metal or alloy.** A. F. BURGESS. From LINK-BELT CO. (B.P. 338,924—5; 31.5.29).—See U.S.P. 1,760,239 and 1,760,240; B., 1931, 119.

**Production of non-corrodible [manganese-copper-nickel] alloys.** C. CONTAL (B.P. 346,226, 20.6.30. Fr., 21.6.29).—See U.S.P. 1,771,773; B., 1931, 207.

**Mercury boilers. [Crushing surfaces of] mills. Discs for centrifuges.**—See I. Hydrogenation catalysts.—See II. Salt mixtures from lead refining. Reduction of iron ores. Phosphorus.—See VII. Mirror glass.—See VIII.

## XI.—ELECTROTECHNICS.

**Measurement of  $p_H$  of nickel-plating baths.** J. BARBAUDY, A. GUÉRILLOT, H. MIACHON, and R. SIMON (Compt. rend., 1931, 192, 739–741).—Liquid from the nickel-plating bath is passed continuously through a cooling arrangement and then, after saturation with quinhydrone, past a (gold) quinhydrone electrode and the porous pot containing solid potassium chloride and a calomel electrode. Actual recording is by means of a Leeds and Northrup potentiometer sensitive to 1 millivolt.

C. A. SILBERRAD.

**Boiler feed-water.**—See I. Hydrocyanic acid.—See VII. White tin. Hardness of electrodeposits.—See X. Phenol-formaldehyde resins.—See XIII. Determination of  $p_H$  of soils.—See XVI. Purity of syrups.—See XVII. Photographic plates. Images on photoelectric cells.—See XXI.

## PATENTS.

**Electric furnaces comprising a hearth and resistances arranged above the charge.** E. F. RUSS (B.P. 344,662, 4.3.30. Ger., 4.3.29).—Hot air is forced from the region of the electric heaters to that of the charge, in a direction parallel to the surface of the charge, by an agitator arranged in an enlarged channel placed outside the furnace chamber.

J. S. G. THOMAS.

**[Charging of] electric furnaces.** P. L. J. MIGUET and M. P. PERRON (B.P. 344,493, 4.12.29. Fr., 6.12.28).—Various raw materials are separately fed through feed columns arranged radially with respect to a central upper electrode co-operating with a hearth electrode.

J. S. G. THOMAS.

**Polarised electrical [electrochemical] couple.** P. E. EDELMAN, Assr. to E. BANNING (U.S.P. 1,773,665, 19.8.30. Appl., 12.4.28).—The couple comprises an aluminium electrode coated with coloured dielectric material prepared by treatment of the electrode with a solution of a compound of a molybdate of ammonium, sodium, or potassium, and a solution of a bitartrate compound; a co-operating electrode, e.g., of nickel; and electrolyte, e.g., a dilute solution of ammonium bitartrate containing gum arabic.

J. S. G. THOMAS.



**Apparatus for electrical precipitation of suspended particles from gases.** LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 344,758, 10.7.30).—Apparatus comprising plate- or pipe-shaped collecting electrodes arranged at a constant distance apart, and twisted band or strip discharge electrodes, the width of which varies progressively in the direction of gas flow, is claimed. J. S. G. THOMAS.

**Collecting electrode for electrical precipitators [for gases].** C. B. CLYNE, ASSR. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,773,951, 26.8.30. Appl., 11.12.26).—A collecting electrode comprising plate sections of electrically semi-conducting cementitious material having their edges abutting closely against one another but unbonded, and common reinforcing means embedded or cast in all the sections, is claimed.

J. S. G. THOMAS.

**Electrical purification of gases, in particular furnace gases.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 344,550, 16.12.29. Ger., 17.12.28).—Gas before entering an electric filter is heated and then moistened, *e.g.*, with water, so that particles suspended in the gas become sufficiently conducting for purposes of separation without the gas being cooled below the dew point.

J. S. G. THOMAS.

**Photoelectric cell.** R. C. BURT, Assr. to CALIFORNIA INST. OF TECHNOLOGY (U.S.P. 1,776,993, 30.9.30. Appl., 26.5.26).—The transparent glass container of the cell is immersed in molten alkali salt, *e.g.*, sodium nitrate, and an electric field is applied between the salt and an electrode arranged within the container, whereby alkali metal is caused to migrate through the glass container and form a cathode surface on the inner surface of the container. J. S. G. THOMAS.

**Electrodes [support] for electric-discharge apparatus.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of SOC. GÉN. DE CONSTRUCTIONS ELECTRIQUES ET MÉCANIQUES (ALSTHOM) (B.P. 344,680, 24.3.30. Fr., 23.3.29).—Basalt is fused to the metal of the electrode and/or the metal receptacle to form a gastight, insulating support.

J. S. G. THOMAS.

**Manufacture of refractory articles [for use, *e.g.*, as support members in vacuum electric tubes].** WESTINGHOUSE LAMP CO., Assees. of C. V. IREDELL (B.P. 344,572, 24.12.29. U.S., 29.12.28).—Powdered refractory insulating material, *e.g.*, alumina or thoria heated at about 1500° and mixed with about 2% of talc, and an organic wax, *e.g.*, paraffin wax, is moulded under pressure and fired. J. S. G. THOMAS.

J. S. G. THOMAS.

[Contact protector for] luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 344,397, 4.12.29. Ger., 9.7.29).

**[Fluid-cooled] resistance welding electrodes.**  
E. G. BUDD MANUFG. Co., Assees. of J. J. PAUGH (B.P.  
346,585, 25.4.30. U.S., 29.4.29).

["Eureka" fuse for gas-filled] electric incandescence lamps. GEN. ELECTRIC Co., LTD., and A. G. PEARCE (B.P. 344,577, 31.12.29).

**Light-sensitive electric cells.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. W. HULL (B.P. 344,499, 5.12.29. U.S., 27.12.28).

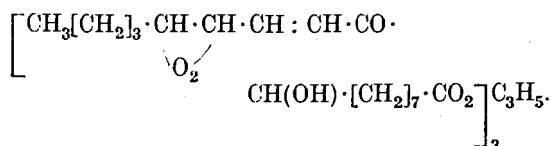
**[Electrode for] electric-discharge tubes.** GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 344,497, 4.12.29. Ger., 29.6.29).

**X-Ray tubes [with rotating anticathode].** N.V.  
PHILIPS' GLOEILAMPENFABR. (B.P. 344,692, 4.4.30.  
Holl., 19.4.29. Addn. to B.P. 286,436).

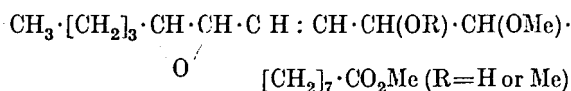
Vaporisation of liquids. Thermo-regulator.—See I. Dry carbon. Products from hydrocarbons. See II. Catalytic processes.—See III. Cellulose pulp. Drying wood articles.—See V. Smelting of phosphoric material. Treatment of dolomite. Magnesium chloride. Reduction of iron ores. Cleaning of gases.—See VII. Mirror glass.—See VIII. Alloy for loading coils. Copper powder. Zinc refining. Coatings on aluminium. Cable sheathings. Silver-plate. Alloys for contacts. Chromium-plate.—See X.

## XII.—FATS ; OILS ; WAXES.

**Oxidation products of drying oils. I.  $\beta$ -Elæostearin from tung oil.** R. S. MORRELL and S. MARKS (J.S.C.I., 1931, 50, 27–36 r).— $\beta$ -Elæostearin ( $C_{18}H_{29}O_2$ ) $_2C_3H_5$  absorbs 4 atoms of oxygen in benzene or carbon tetrachloride solution to yield a diperoxide ( $C_{18}H_{29}O_6$ ) $_2C_3H_5$ , which readily undergoes change to a gel which has the structure



This compound is of keto- or enol type according to the conditions. Treatment with methyl sulphate and extraction with light petroleum yields ethers which contain either one or two methoxyl groups. These methylated compounds undergo decomposition when distilled *in vacuo*; oxidation with alkaline potassium permanganate gives valeric, azelaic, glyoxylic and glycollic (traces), and oxalic acids; reduction with hydrogen in the presence of palladium chloride has given two oils having the compositions:



The original oxidation product is also found to be amphoteric in that the peroxide group near the glyceryl end of the molecule is acidic, while the remote peroxide group is basic. Attention is drawn to the importance of this observation in connexion with the yellowing of oil films in the dark, the degradation of oil films on exposure to air, the polymerisation of oil films on exposure to air and of heat-thickened oils, the action of "driers" on oils, and the formation of "bloom" on oil films in damp atmospheres.

**Determination of rancidity in oils and fats.**

A. TAFTEL and C. REVIS (J.S.C.I., 1931, 50, 87—91 r).—A modified form of the Kreis test is described. A simple and rapid quantitative method for determining the degree of rancidity in oils and fats has been worked out. It depends on the fact that peroxides liberate free iodine quantitatively from barium iodide in glacial acetic acid solution, and that the iodine set free by peroxides present in rancid oils is prevented, by the presence of barium iodide, from attacking the double linkings in the oil molecule. By the use of this method it has been shown that oils such as groundnut oil, which have become rancid at temperatures up to at least 60°, contain a form of peroxide which is rapidly reduced by hydriodic acid. Oils which have become oxidised at fairly elevated temperatures, *e.g.*, 120°, contain a mixture of these easily reducible peroxides together with some other form of active oxide which is reduced only with difficulty, whilst oils which have been air-blown at 170° contain practically only the latter type and very little of the former type. The intensity of the Kreis reaction is correlated with the amount of easily reducible peroxides in an oil. The difficultly reducible peroxides have no connexion with the Kreis reaction. The Kreis reaction is not an infallible test for rancidity, whilst the new methods appear to be of universal application.

**Solubility of fats in various solvents. Solubility of (III) camellia oil, (IV) rape oil, in isopropyl alcohol of various concentrations.** K. HASHI (J. Soc. Chem. Ind., Japan, 1931, 34, 104—105 B, 105—106 B).—The solubilities in aqueous isopropyl alcohol solutions were determined as before (*cf.* B., 1931, 500). The results are similar to those recorded previously, but here the curves of clouding point concentration of the alcohol (*b*) for various oil concentrations are convex to the abscissa (*b*). In solutions containing over 80% of camellia oil turbidity sets in slowly and concordant results are not always obtained on repetition. For solutions in aqueous isopropyl alcohol of concentration (wt.-%) 100, 95, 90, 87.5, and 84.99, the critical temperatures are: (III) 25°, 48°, 83.5°, >111.5°, >139°; and (IV) 41.3°, 65.5°, 100.3°, —, >148°, respectively; and the critical concentrations (oil-%): (III) 39.0, 42.0, 64.5, >80, >80; and (IV) 38%, 40.3, 58.3, —, >73.4.

E. LEWKOWITSCH.

**Antioxidants and the autoxidation of fats.** H. A. MATTILL (J. Biol. Chem., 1931, 90, 141—151).—By a modification of the method of Greenbank and Holm (B., 1925, 556) measurement has been made of the rate of oxygen uptake by a standard mixture of lard and cod-liver oil in presence of various compounds. Monohydric phenols were only very slightly active in increasing the induction period, but polyhydric phenols exhibited very considerable activity when they contained hydroxyl groups in the *ortho* or *para* position.  $\alpha$ -Naphthol was also active, although  $\beta$ -naphthol exhibited only slight activity. Benzoquinone and  $\beta$ -naphthoquinone show a marked inhibiting action, but  $\alpha$ -naphthoquinone is ineffective. Inositol and cholesterol and other naturally occurring sterols were without action, as were also all compounds which did not contain a free phenolic hydroxyl group. W. O. KERMACK.

**Unsaponifiable matter and so-called isooleic acids contained in toilet soaps.** S. UENO, G. INAGAKI, and H. TSUCHIKAWA (J. Soc. Chem. Ind., Japan, 1931, 34, 90—92 B).—The amounts of unsaponifiable matter found in 12 samples of commercial toilet soaps did not correspond with the amounts of hardened oils present, as deduced from determination of the isooleic acid contents (Richardson's method, B., 1924, 564). It is concluded that the former content of unsaponifiable matter is an unreliable guide for the detection of hardened oils (*cf.* Wittka, B., 1927, 944).

E. LEWKOWITSCH.

**Manganese soaps. II.** H. WAGNER and G. HOFFMANN (Farben-Ztg., 1931, 36, 1214—1216; *cf.* B., 1931, 211).—Manganese hydroxide and raw and burnt umbers were ground in linseed oil and films were allowed to dry on glass and metal plates for 14 days, when their water absorptions after 1, 3, 5, and 15 days were noted and graphed. Manganese hydroxide had the most pronounced tendency to swelling, whilst umbers were less affected, although in all cases after swelling films deficient in cohesion and elasticity resulted. The addition of 5% and 10% of manganese hydroxide to Mars yellow (containing 53% Fe<sub>2</sub>O<sub>3</sub>, 20% CaCO<sub>3</sub>, and 17% CaSO<sub>4</sub>) causes decreased drying time but increased swelling of paints made therefrom. At concentrations of manganese hydroxide above 5%, the film properties are, in general, adversely affected; the use of pigments rich in manganese hydroxide for rust-prevention is not recommended. Photomicrographs illustrating the appearance of the different films after swelling are included.

S. S. WOLFE.

**Migration of sodium chloride in soap cakes.**

J. MIKUMO (J. Soc. Chem. Ind., Japan, 1931, 34, 116 B).—Kristen's observations (Seifensieder-Ztg., 1927, 54, 665) of the migration of salt in boiled and settled, or even in milled, soaps have been confirmed. The direction of the migration is determined chiefly by the difference of vapour tension between the inside and outside of the cake; if kept in a very moist atmosphere, salt may concentrate in the outer layers. Probably during cooling and storage curd soaps become more fibrous and release part of the water of hydration as free water (syneresis).

E. LEWKOWITSCH.

**Hydrogenation of fatty oils by the so-called "wet process." I. Hydrogenation in presence of nickel acetate under atmospheric pressure.**

S. UENO and T. YUKIMORI (J. Soc. Chem. Ind., Japan, 1931, 34, 109—111 B).—Using a nickel acetate-kieselguhr catalyst, the rate of decrease of iodine value is not always proportional to the amount of catalyst used. In all cases this catalyst is inferior in activity to a nickel-kieselguhr catalyst which has been reduced prior to admixture with the oil.

E. LEWKOWITSCH.

**Hydrogenation of [fatty] oils at high temperatures and under high pressures. I. Hydrogenation with nickel under constant high pressures.** S. UENO, T. YUKIMORI, H. TSUCHIKAWA, and S. UEDA (J. Soc. Chem. Ind., Japan, 1931, 34, 111—115 B).—Hydrogenation of a refined sardine oil (for 1 hr.) with a reduced nickel-kieselguhr catalyst has been conducted

under pressures of from 50 to 10 atm. over the temperature range 150—230°. (Analytical details of the samples are given.) The amount of isooleic acid produced is very small (especially at high pressures) compared with that obtained at ordinary pressure. The temperature exerts a marked influence on the velocity of the hydrogenation, the reaction being most rapid at 180—200°. E. LEWKOWITSCH.

**Absorption of oxygen by unsaturated [fatty] oils. I. Influence of the solvent on the rate of absorption.** S. UENO, Z. OKAMURA, and T. SAIDA (J. Soc. Chem. Ind., Japan, 1931, 34, 106—108 B).—The oil containing cobalt linoleate as drier was tested in various solvents and the rates at which oxygen was absorbed were compared. The greatest acceleration of the oxidation was obtained in solution in glacial acetic acid; some other solvents fall into the (decreasing) order (for linseed oil) decalin, ethyl acetate, turpentine, chloroform, acetone. For sardine oil the order of the last four is slightly different. E. LEWKOWITSCH.

**The silver-foil or "coin" test for sulphured [fatty] oils.** R. MARCILLE (Ann. Falsif., 1930, 23, 527—530).—The "coin" test is modified so that it will detect as little as 5% of sulphured oil in a pressed oil. It is shown also that pressed olive oils will give a positive reaction, if they are allowed to remain in contact with "foots" containing soluble sulphates, the latter being reduced to sulphides by micro-organisms. The positive "coin" test must therefore be confirmed by other reactions characteristic of sulphured oils.

E. B. HUGHES.

**Twitchell reagent. III.** K. NISHIZAWA and Y. MATUKI (Chem. Umschau, 1931, 38, 73—78).—A fuller account is given of work already noted (B., 1930, 870).

E. LEWKOWITSCH.

**Composition of linseed oils obtained in various ways and stored under various conditions.** K. H. BAUER and A. FREIBURG (Chem. Umschau, 1931, 38, 78—80).—Oils obtained by (a) cold pressing, (b) hot pressing, and (c) extraction with light petroleum from the same sample of Calcutta linseed were analysed. The ordinary constants for these samples were much alike: the drying curves revealed slightly greater differences, the hot-pressed oil giving the worst curve; the fatty acid compositions, determined by the Eibner method, however, showed considerable differences. Samples of the oils were stored for 1 year in filled, stoppered, glass bottles in daylight: the iodine value of the oils rose 2—9 units, and the saponif. values decreased a few points; the illuminated oils all dried in about one third the time of the original samples. Most "astounding" changes, however, had apparently occurred in the fatty acid composition, as is shown by the following figures (%) for the oils (a), (b), (c), respectively (figures for the illuminated specimens in parentheses):  $\alpha$ -linolenic acid 15.88 (29.1), 20.3 (19.99), 18.81 (26.71);  $\beta$ -linolenic acid 3.96 (2.02), 0.21 (0.5), 3.89 (1.53);  $\alpha$ -linoleic acid 19.31 (22.01), 15.09 (8.7), 18.13 (8.86);  $\beta$ -linoleic acid 36.82 (1.8), 40.76 (8.38), 35.52 (15.76); oleic acid 6.65 (31.3), 9.07 (50.14), 8.91 (33.0). Analyses were attempted by applying the thiocyanogen method to the liquid acids separated by the thallium and

lead salt methods; extraordinary differences in the apparent composition of the fresh and stored oils again was found, but the discrepancies between the figures so found and those given by the Eibner method were very large and irregular both in sense and magnitude. The authors can offer no explanation of these "remarkable" results, beyond the uncertain nature of available analytical methods. E. LEWKOWITSCH.

**Formation of drying oil films.** C. P. A. KAPPELMEIER (Chem. Weekblad, 1931, 28, 174—183).—An account of earlier and recent knowledge and theories. Drying is to be regarded as caused by chemical and colloidal factors acting together. S. I. LEVY.

**Factors of quality in copra.** F. C. COOKE (Malay. Agric. J., 1931, 19, 128—136).—The deterioration of copra under mould action is discussed in relation to the moisture content and storage conditions; 4—6% of water represents average stable conditions. A badly-rotted copra will contain a high percentage (e.g., 70%) of ether-extractable fatty matter, but this contains a high proportion of free fatty acid. A mouldy copra of high free fatty acid content, if stored in good conditions of free ventilation, becomes clean and dry (moulds falling off as dust) and the acidity diminishes. Compression into bales under a pressure of 1.5 cwt./in.<sup>2</sup> for transport is satisfactory. E. LEWKOWITSCH.

**Reactions of cod-liver oil and castor oil.** L. EKKERT (Pharm. Zentr., 1931, 72, 209—212).—One drop of cod-liver oil dissolved in 1 c.c. of absolute alcohol is mixed with 3 drops of a 1% alcoholic solution of an aldehyde, and poured cautiously on to 1 c.c. of concentrated sulphuric acid: a coloured ring is produced at the junction of the liquids; on shaking, the liquids become coloured throughout, and are further examined in the light of the quartz lamp. The various shades (browns to violet-reds) obtained by the use of various aldehydes are detailed. Similar colours are obtained with castor oil. E. LEWKOWITSCH.

**Oil from the seeds of *Asteriastigma macrocarpa*.** D. H. PEACOCK and C. THOUNG (J.S.C.I., 1931, 50, 7—8 T).—The results of André (B., 1926, 98) were confirmed. Oleic acid isolated by the lead salt method amounted to about 7% on the fatty acids obtained. By fractional distillation of the ethylesters followed by crystallisation of the acids from light petroleum chaulmoogric acid was isolated. Hydnocarpic acid could not be isolated. The optically active fatty acids amounted to about 75% at least of the fatty acids. A saturated fatty acid of equivalent weight equal to that of palmitic acid but of m.p. 50° was also isolated.

**Determination of inorganic impurities in sulphonated oils.** R. HART (Chem. Umschau, 1931, 38, 81—83; cf. Nishizawa and Winokuti, B., 1931, 307).—The derivation of the formulæ deduced in previous papers (B., 1921, 226 A, 438 A) is re-stated. Titration of the oils (for combined sulphate) may be facilitated by adding a small measured amount of acid to the salt and ether mixture; after settling, more indicator is added to the lower layer and titration is completed. For greater accuracy the brine washings of the ether layer should be added. The average acid content of

a number of commercial sulphonated castor oils, washed twice with Glauber's salt solution, was found to be equivalent to about 3 mg. KOH per g., i.e., at least 90% (and probably more) of the combined  $\text{SO}_3$  had been neutralised in the washing. E. LEWKOWITSCH.

**Fatty acid components of the oil from (A) Malayan gaviol *Fomistoma Schlegli*, and some properties of the oil from (B) *Pecten (Patinopecten) Yossensis*, Jay. S. UENO and N. KUZAI (J. Soc. Chem. Ind., Japan, 1931, 34, 92—93 B).**—The oil from (A) was a pale yellow liquid (at 18°), and gave no blue colour with antimony trichloride or acid earth. It had  $d_{4}^{15}$  0.9166,  $n_D^{20}$  1.4623, iodine value (Wijs) 77.4, saponif. value 197.6, acid value 19.4, Reichert-Meissl value 0.54, acetyl value (Cook) 22.4, unsaponifiable matter 0.44%, ether-insoluble bromides trace. The mixed fatty acids (m.p. 33.9—35.2°, iodine value 76.5, neutralisation value 202.3) yielded 64.3% of liquid acids (lead salt-ether method) having iodine value 92.8, neutralisation value 201.0, ether-insoluble bromides 0.052% (consisting of oleic acid with traces of linoleic, linolenic, and more unsaturated acids), and solid acids having iodine value 6, neutralisation value 214.0, m.p. 51.2—52.1°. The saturated acids consisted chiefly of palmitic acid with a small amount of stearic acid: cholesterol, but no hydrocarbon, was present in the unsaponifiable matter.

The deep brown odorous oil from (B) showed a weak green colour reaction with acid earth. The refined oil had  $d_{4}^{100}$  0.8784,  $n_D^{20}$  1.4781, saponif. value about 187.7, Reichert-Meissl value 1.2, acid value 1.2, iodine value (Wijs) 204.0. The mixed acids [m.p. 30.9°, iodine value 199.8 (!)] yielded 50.46% of ether-insoluble bromides containing 70.34% Br. E. LEWKOWITSCH.

**Burmese species of plants yielding chaulmoogra oils.** D. H. PEACOCK and G. K. AIYAR (Burma Forest Bull., 1930, No. 21, 11—13).—The seeds of four species were examined. *Taraktogenos Kurzii*: The kernels, about 58—70% of the whole fruit, contained about 50% of oil, saponif. value 183—203 (av. 197), iodine (Wijs) value 94—113 (av. 110);  $[\alpha]_D^{25-30}$  in chloroform +38° to +55° (av. +47°). *Asteriastigma macrocarpa*: The seeds contained 36.4—56.9% of oil having  $d_{4}^{35}$  about 0.9501, iodine value 103—127 (av. 112), saponif. value 192—204 (av. 195), unsaponifiable matter about 3%;  $[\alpha]_D$  +38.8° to +55.6° (av. +53°),  $n_D^{20}$  about 1.4790. Inactive oleic acid content about 7%. Corresponding values for *Hydnocarpus verrucosa* and *H. dawnensis* are: 10.9%, 35.0%; 0.8519, 0.8531; 81.1, 91.4; 202.0, 182.3; 1.54%, 6.2%; +43.6°, +38.8°; 1.4752, 1.4760. T. McLACHLAN.

**Phthaleinoscope.**—See I. Viscosity of oils.—See II. Wood staining.—See IX. Fat of sow's milk.—See XIX.

#### PATENTS.

**Condensation products or intermediate compounds from fish-liver oils.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, and W. A. SEXTON (B.P. 345,734, 22.1.30).—Alkali-soluble compounds suitable for use in the textile industry are prepared by condensing the halogen derivatives of squalene (e.g., the dodecylbromide) with a phenol in the presence of aluminium chloride. The compounds are precipitable by acetic

acid, fuse between 120° and 130°, and yield highly coloured azo compounds with diazonium salts.

E. LEWKOWITSCH.

**Filter device [for fish oils etc.]. Turbine lubrication.**—See I. Sizing of textiles. Oiling of fibres.—See VI.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**"Chalking" [of paint films].** IV. Practical trials of chalking of oil paints. I. R. KEMPF (Farben-Ztg., 1931, 36, 1173—1176, 1216—1217, 1256—1259; cf. B., 1931, 169).—An intermediate report is given on exposure tests on paints based on (a) individual pigments, i.e., basic carbonate white lead, titanium white, titanium dioxide, and lithopone, and (b) mixtures of zinc oxide, basic carbonate and basic sulphate white leads, and lead chromate, each with three types of lithopone, in different vehicles, e.g., boiled linseed oil, tung oil, and stand oils (linseed and tung oils), other variables being locality and aspect of exposure, and nature of priming paint. Full details are given of compositions and degree of chalking after 3, 5, and 10 months, as measured by the author's "stamping method" (B., 1930, 156), the results being illustrated graphically and photographically. The development of cracks and wrinkling is also recorded. The results are discussed at considerable length and the pigments are found to behave similarly (as to order of chalking) in the various vehicles tested. White lead carbonate and lithopone-lead chromate were found to be the more free from chalking tendency of the two groups. The suggestion that cracking and chalking are inversely proportional is not upheld. S. S. WOOLF.

**Paint testing.** P. NETTMANN (Farben-Chem., 1931, 2, 22—23).—Accelerated weathering tests are considered to fail to produce significant results owing to our incomplete knowledge of the processes of film destruction under natural weathering. The disruptive forces acting in a finishing coat-undercoat system on iron are discussed hypothetically and illustrated by diagrams, and the thermodynamics of the system are outlined.

S. S. WOOLF.

**Antimony yellow [pigment].** J. MILBAUER (Chem.-Ztg., 1931, 55, 222).—When antimonite is treated with concentrated caustic soda in a current of air in a porcelain ball mill, it changes gradually into a bright yellow pigment, a solid solution of antimony trisulphide in antimony oxides. Its exact composition varies according to the amount of soda used; if no iron is present the best yellow (6% S) is obtained by working for 3 hrs. with ingredients in the proportions of 1 pt. of the trisulphide to 6 pts. of 25% aqueous caustic soda. This pigment resembles Indian yellow and pale ochre, has  $d$  2.43, covering power 320  $\text{cm}^2$ , is stable up to 200° and towards atmospheric conditions, but is not compatible with metallic pigments which react with sulphides.

E. LEWKOWITSCH.

**Sources of error in the colorimetric determination of iron in red lead.** J. F. SACHER (Farben-Chem., 1931, 2, 120—122).—The work of various investigators on the determination of quantities of the order of 0.0005—0.01% Fe in red lead is summarised. A source

of error in the usual colorimetric method is the hydrolytic decomposition of ferric thiocyanate to an insoluble, brown, basic salt, this effect depending jointly on the concentration of the solution, the acidity, and the amount of thiocyanate added. A further error arises owing to the light-sensitivity of the ferric thiocyanate.

S. S. WOOLF.

**Determination of sp. gr. of pigments.** W. ESCH (Farben-Chem., 1931, 2, 154—156).—The following method, originally intended for use on pigments for the rubber industry, is capable, with limitations, of application to paint pigments. A standard rubber mixing is prepared from "1a latex pale crêpe rubber" (100 pts.), lead-free zinc oxide (7 pts.), mineral rubber (6 pts.), double-pressed stearic acid (3 pts.), accelerator "Captax" (1 pt.), and "velvet" sulphur (3 pts.). This mixing has *d* 1 before or after vulcanising. A suitable proportion of the pigment to be tested is incorporated (e.g., 1 of carbon black : 3 of "mixing") and the whole is vulcanised. The sp. gr. of the product may be determined simply, and that of the pigment arrived at by calculation.

S. S. WOOLF.

**"Water-spotting" of oil and nitrocellulose lacquers.** F. KOLKE (Farben-Chem., 1931, 2, 106—108).—The occurrence and probable causes of water-spotting are discussed, such factors as moisture, heat, rain, water-soluble constituents in the paint, deposition of limestone dust, etc. accounting for the phenomenon in the case of oil enamels. The function of linseed oil as a "water accumulator" is mentioned, and, where spotting occurs with clear oil varnishes, it is probably due to swelling owing to incorrectly bodied oil, the superiority of tung oil and tung oil enamels in this respect being significant. Nitrocellulose lacquers are treated separately, the hardness of the lacquer film and the nature of the resin constituent here being the most important considerations, but the use of certain red and black aniline dyes may be a contributory cause of the defect. Zinc oxide is known to favour the formation of water spots in oil and cellulose enamels, but it is shown that the pigment portion of a lacquer cannot entirely account for the trouble.

S. S. WOOLF.

**Butyl and amyl lactates as solvents for [nitro]-cotton lacquers.** O. GERHARDT (Chem.-Ztg., 1931, 55, 222).—Butyl lactate (b.p. 177°/760 mm.) is equal and amyl lactate (b.p. 115°/56 mm.) superior in solvent power to ethyl lactate, and their tolerance for diluents (cf. Wolff, B., 1928, 519) is much greater. A 10% nitro-cotton (WAS 10) can be diluted without turbidity by 11 pts. (by wt.) of benzene; it dries to a clear film with 9 pts. (or 10 pts., with a low-viscosity cotton). The corresponding dilution figures for amyl and ethyl lactates are 1:16 or, for clear drying, 1:14—15; and 1:7 or 1:5—5.5, respectively. Lacquers with these solvents, especially the amyl ester, flow well and give good films.

E. LEWKOWITSCH.

**Resins. VIII. Their identification.** E. STOCK (Farben-Ztg., 1931, 36, 1176—1177).—A comprehensive range of resins was tested by Donath's method (cf. B., 1930, 957). The finely-powdered resin (1 g.) is boiled for 1 min. with 5 c.c. of nitric acid (*d* 1.32—1.33), the

cooled liquor diluted (1:1) with distilled water, and ammonia then added in excess. The colours developed immediately and after 5 min., 1 hr., and 1 day are tabulated for the resins tested; a progressive intensification of the coloration is reported, in all cases excepting that of "Albertol" resins. Rosin gives an immediate orange coloration that deepens in 1 day to the "deep brown-red" described by Donath. In particular groups of resins, distinctions are pointed out in the results at one or other of the time periods observed, but it is apparent that this reaction will not distinguish rosin from other resins with surety, nor detect it in mixtures. Nevertheless it shows the advantage over the Liebermann-Storck reaction that the colorations produced are not fugitive.

S. S. WOOLF.

**Phenol- and cresol-formaldehyde resins.** H. STÄGER (Helv. Chim. Acta, 1931, 14, 285—304).—A series of synthetic resins of phenol-, cresol-, and mixed phenol-cresol-formaldehyde type (prepared by condensation under alkaline conditions) is examined in the crude state by a variety of physical and some chemical methods. The latter, in general, including the loss in weight on thermal hardening, the content of volatile products, and the bromine absorption and mol. wt. of the benzene-soluble fraction of these volatile products, are found insufficient to characterise the resin. The progressive decrease in alcohol-solubility that accompanies thermal hardening is much less in the case of the cresol resins; this does not hold true for alkali-solubility, however, thus confirming Koebner's hypothesis of the formation of insoluble sodium salts. The initial decrease in the "cementing coefficient" (cf. B., 1929, 520) with increasing thermal hardening is slower the higher the phenol content of the resin: the effect on this property of heat-treatment in the case of shellac contrasts markedly with the behaviour of these synthetic resins, a continuous increase in strength being observed. The electrical properties of the synthetic resins vary consistently in the various classes, and show a decided break at a phenol content of about 30%. The breakdown voltage increases with increasing phenol content, and in the pure cresol resins increases with decreasing *m*-cresol content. The dielectric constant also varies directly with the phenol content and, in a simple cresol resin, inversely with the *m*-cresol content; it increases in every case with progressive thermal hardening, but the interrelationships between the various types of resin remain unchanged. Dielectric loss is related to composition in the same way as the dielectric constant, but in the case of resins containing more than 30% of phenol increases very rapidly with the potential gradient, and cannot be measured when the latter exceeds 5 kilovolts/cm.; the values are very low for the pure cresol resins and less dependent on potential gradient. It is concluded that there is an essential difference between the phenol- and cresol-formaldehyde resins in that the degree of polymerisation attained under similar conditions is less, and the polar characteristics of the products are consequently more pronounced in the former. *m*-Cresol is the most reactive of the cresols.

H. A. PIGGOTT.

**Urban glue.**—See XV. **Blood-albumin.**—See XIX.

## PATENTS.

**Separation of anethole from pine oil.** L. T. SMITH, ASSR. to HERCULES POWDER CO. (U.S.P. 1,777,704, 7.10.30. Appl., 11.8.27).—Pine oil is fractionated and the fraction, b.p. 220—250°, is refractionated to give a distillate, b.p. 232—237°, from which anethole is obtained by crystallisation. E. H. SHARPLES.

**Manufacture of surface-decorated [floor-covering] materials [e.g., linoleum].** ARMSTRONG CORK CO., ASSEES. of H. R. WOERNER (B.P. 344,660, 3.3.30. U.S., 7.3.29).—A masking material for use in the process of B.P. 319,655 comprises a filler, e.g., whiting, suspended in a medium of sufficient viscosity to maintain the filler in suspension and of sufficiently low surface tension to spread readily. Glycerin or glycol in admixture with soap, or aqueous gum (tragacanth) solution containing soap and potassium carbonate, is suitable. D. J. NORMAN.

**Manufacture of inlaid linoleum or the like.** ARMSTRONG CORK CO., ASSEES. of J. E. WILEY (B.P. 346,211, 21.5.30. U.S., 24.5.29).

**Coating for writing tablets etc.**—See V. **Balloon fabric.**—See VI. **Barium sulphide solutions.**—See VII. **Reinforced glass etc.**—See VIII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Röntgenographic study of chicle.** C. W. STILLWELL and G. L. CLARK (Kautschuk, 1931, 7, 62—66).—Röntgenographic investigation shows raw chicle to consist of at least three crystalline substances and one or more amorphous compounds; the crystalline constituents are chicle-gutta, which is identical with the hydrocarbon of gutta-percha or of balata, a resin, and calcium oxalate monohydrate, the last-named forming the crystalline portion of the benzene-insoluble fraction. Refined chicle has a similar structure to that of the raw material, except that its gutta is more highly dispersed, the high temperature involved in the purification process converting it into an amorphous condition which is rendered stable by the presence of the other constituents. D. F. TWISS.

**Chemistry of [rubber] latex.** II. P. SCHOLZ and K. KLORZ (Kautschuk, 1931, 7, 66—68).—The dry solid content of latex is determined by carefully evaporating approx. 2 g. in a flat nickel dish (diam. 10 cm.) over a flame or hot plate with avoidance of bubbles, until a clear film remains free from opaque spots. The dry film while still warm is rolled up so as to reduce its free surface and is then removed and weighed. The process takes 6—10 min. and gives results accurate to  $\pm 0.1\%$ . The rubber crêpe yield of latex is measured by weighing about 25 g. of the latex into a Petri dish (diam. 10 cm.). Coagulation is then effected by adding 10—20 c.c. of 10% sulphuric acid by a pipette, beginning at the edge and proceeding spirally to the centre. After a rest of 10 min. the resulting cake of coagulum is removed without pressure and washed with running water at 50° for 2 hrs. until on contact it no longer reddens litmus paper. The coagulum is then pressed between cloth and dried at 70—85° until a clear amber colour throughout. The direct estimation of the rubber crêpe yield may be obviated by approximate calculation

from the total solids figure (T.S.); the crêpe yield in g. per 100 c.c. is given by the formula  $0.9T.S.$ , and the percentage by wt. by  $0.9T.S./d$  (where  $d$  = sp. gr.) or  $0.92T.S.$  assuming an average sp. gr. of 0.98. D. F. TWISS.

**Application of accelerators and anti-agers [in rubber manufacture].** A. H. SMITH (Kautschuk, 1931, 7, 70—75).—Conditions are reviewed for the satisfactory technical use of various well-known accelerators of vulcanisation and anti-agers. For the best results generally it is advisable to use the minimum of sulphur, and in order to ensure good flexing and ageing care must be taken to avoid overheating. Hardness must be increased by suitable compounding and not by increased use of sulphur. Excessive use of softeners frequently detracts from good ageing. Vulcanisation should be effected at the lowest possible temperature. D. F. TWISS.

**Sp. gr. of pigments.**—See XIII.

## PATENTS.

**Treatment of rubber. [Dispersing rubber in water.]** W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,780,657, 4.11.30. Appl., 23.8.22).—An aqueous dispersion of rubber, or of an analogous hydrocarbon such as gutta, is obtained by first dissolving it in benzene or similar solvent and then mixing the solution with water and a colloidal dispersing agent, the rubber solvent being finally removed. The first portion of the water is preferably added as an aqueous solution of ammonia, and the colloid dispersing agent, such as soap, is introduced with or after the remainder of the water, but before removal of the solvent by distillation under reduced pressure or in any other way. D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,780,604, 4.11.30. Appl., 28.11.27).—*s*-Diaryl-substituted guanidines in which one of the aryl substituents contains a piperidyl group *para* to the imino-group, e.g., phenyl-*p*-piperidyl-phenylguanidine, are effective accelerators of vulcanisation. D. F. TWISS.

**Balloon fabric. Rubber threads.**—See VI.

## XV.—LEATHER; GLUE.

**Microscopical study of the effects of cold temperatures on [raw] hides and skins.** F. O'FLAHERTY and W. T. RODDY (J. Amer. Leather Chem. Assoc., 1931, 26, 172—180).—Sections of freshly slaughtered hides and skins were examined microscopically before and after the skins had been subjected to low temperatures and other treatment. No structural damage was observed, though slight dehydration had taken place, in skins which had been subjected to 4 weeks' treatment in a refrigerator at 0°, but some tissues were ruptured by folding such skins. No damage was found in the product obtained when the frozen skins were kept for 2—4 hrs. at room temperature before soaking, and least change was noticed in such skins if they were then soaked in dilute salt solution. Better results were obtained in skins which had been salt-cured and frozen than in those subjected to either treatment alone. No difference in structure was observed after liming between frozen and unfrozen skins. D. WOODROFFE.

**Moisture determination and its application to leather.** A. COLIN-RUSS (*J. Soc. Leather Trades' Chem.*, 1931, **15**, 113—126, 166—182).—A weighed quantity of leather is placed in a charging tube, which rests on the end of a release mechanism fitting into the upper side tube of a long, thick-walled reaction tube closed at its lower end and containing some calcium carbide. About the middle of the reaction tube is a lower side tube, with a 0.5-cm. dip containing calcium carbide, and leading to a manometer for measuring volume at constant pressure. The reaction tube is provided with a vapour jacket for maintaining it at any definite temperature. The moisture in the sample is calculated from the volume of acetylene generated when the sample falls on to the calcium carbide and has been left until a constant reading has been obtained. This method has been applied to determine the moisture in the crust skiver, heavily-tanned leather, and certain heavily greased leathers. The apparatus was standardised by means of ammonium oxalate monohydrate in presence of currying grease. It was shown that the agreement between the results obtained by the carbide and desiccation methods was good for all the above leathers except the crust skiver. The result by the carbide method agreed with that by the oven method for the sumac-tanned leather, and was higher than that given by the desiccation method. D. WOODROFFE.

**Evaluation of hide- and bone-glue.** E. GOEBEL (*Farben-Chem.*, 1931, **2**, 174—176).—The determination of water in glues is discussed in detail. Direct drying of powdered samples, in one or more stages, tends to give high results, since in the grinding process moisture is absorbed from the air, whilst correct sampling also offers difficulty. More exact methods comprise the conversion of relatively large quantities of the glue into a homogeneous gel, from which reliable small samples can be withdrawn and dehydrated, the amount of water in the original glue being obtained by calculation. Distillation with xylene, tetrachloroethane, etc. offers a rapid method of obtaining approximate results.

S. S. WOOLF.

**Collagen, gelatin, and glue. Röntgenospectrographic studies.** J. R. KATZ and O. GERNGROSS (*Collegium*, 1931, 67—79).—The well-defined crystal-interference lines and the wide indefinite interference of liquids and amorphous substances are shown on the röntgenospectrograph of pure, practically ash-free, isoelectric gelatin. From further studies it is inferred that dry gelatin consists largely, if not entirely, of long crystals with the long axis parallel to that of the fibres. The crystal interference was destroyed by heating a gelatin solution until it would no longer set on cooling. The adhesive power, gel-forming properties, and elasticity of a gelatin can be measured by the amount of its crystal interference of X-rays. The elastic nature of gelatin is due partly to the parallel orientation of the fibre-forming elements and the subsequent arrangement of the fibres parallel to each other. The interferences observed in gelatin and collagen fibres were practically the same. Evidence of a slightly spiral arrangement of the long collagen elements was obtained.

D. WOODROFFE.

**Colloid-chemical nature of Urban glue.** A. EIBNER and E. ROSSMANN (*Z. angew. Chem.*, 1930, **43**, 943—947).—An account of Urban's process and its application to painting is given and the properties of the product are discussed from the colloid-chemical point of view.

E. S. HEDGES.

**Blood-albumin.**—See XIX.

PATENTS.

**Filter device [for glue].**—See I. **Foils from gelatin.**—See V. **Mordanting hair.**—See VI.

## XVI.—AGRICULTURE.

**Temperature correction in the hydrometer method of mechanical analysis of soils.** C. RICHTER (*Soil Sci.*, 1931, **31**, 85—92).—Uniform corrections for all temperatures and concentrations of the soil suspension are not applicable. A sliding scale of correction factors is suggested. A. G. POLLARD.

**Laboratory study of the field percolation rates of soils.** C. S. SLATER and H. G. BYERS (*U.S. Dept. Agric., Tech. Bull.*, 1931, No. 232, 23 pp.).—Following the development of a soil-core auger which satisfactorily cuts cores of undisturbed soil, a procedure has been developed for evaluating soil-percolation rates. The field-percolation rate of soil is governed more by the water passageways it contains, such as root channels and structural cleavages, than it is by the character or volume of the pore space of the soil mass. The percolation rates of cores serve as a means of studying field permeability, but they vary too much for cores of the same soil to fix rates by this means alone, unless largely replicated. The ratios for erosion and permeability of soils are inversely proportional in terms of the factors of the percolation ratio. E. HOLMES.

**Determination of sulphur in soil.** M. JARACH (*Giorn. Chim. Ind. Appl.*, 1931, **13**, 73).—The frothing and danger of projection encountered when soil is fused with sodium peroxide are avoided as follows. A mixture of 10 g. of the sieved (1 mm.) soil with 26 g. of anhydrous sodium carbonate and 13 g. of potassium nitrate is fused at 900° in a 100-c.c. porcelain crucible in an electric furnace. The cold crucible is transferred to a beaker and covered with water containing a few drops of alcohol, and then left for a day. When the whole contents of the crucible have been extracted, the liquid is filtered through asbestos by suction, the filtrate being evaporated to dryness on a steam-bath and the residue heated at 150° for 5 hrs. The mass is then treated with hydrochloric acid, heated again at 150° to render the silica insoluble, dissolved in dilute hydrochloric acid, and filtered. The sulphate is precipitated as barium sulphate in the usual way. T. H. POPE.

**Interaction between ammonia and soils as a new method of characterising soil colloids.** A. N. PURI (*Soil Sci.*, 1931, **31**, 93—97).—The reaction between soils and bases is slow unless excess of base is present. The base-exchange capacity is determined by treating soil with dilute acid to remove all bases and subsequently saturating with ammonia. The ammonia adsorbed is equivalent to the base capacity of the soil. Soils



containing free calcium carbonate have approximately the same  $p_H$  after the above treatment as they had in the natural condition. (Cf. B., 1931, 36, 79.)

A. G. POLLARD.

**Laws of soil colloidal behaviour. IV. Isoelectric precipitates.** S. MATTSON (Soil Sci., 1931, 31, 57—77; cf. B., 1931, 173).—From sodium humate purified by electrodialysis, humates of iron and aluminium are prepared by precipitation from the respective chlorides. In the presence of phosphates or silicates, phospho- or silico-humates are formed. Humates are differentiated from phosphates and silicates by being precipitated quantitatively by ferric or aluminium chloride, by exhibiting an electropositive maximum on the acid side of the isoelectric point, and where much humus is present, in having a second isoelectric point. Ferric "humates" are isoelectric at a lower  $p_H$  than that of the corresponding aluminium compounds. The "humate" ion displaces the phosphate ion and, to a greater extent, the silicate ion from combination with iron or aluminium. The nature of the precipitation of aluminium salts in mixed phosphate-silicate solutions is examined. The phosphate ion displaces the silicate ion, but the reverse action is not apparent, the phosphate content of the complex precipitate being somewhat increased in the presence of silicate. The mineral soil colloids react with iron and aluminium salts in the same way as does the humus complex, forming isoelectric precipitates.

A. G. POLLARD.

**Biochemistry of arable soil.** A. A. J. VANDE VELDE, A. VERBELEN, and L. DEKOKER (Compt. rend., 1931, 192, 766—767; cf. B., 1930, 1082).—The retention of acids or alkalis by soil after shaking with the requisite solutions is measured. The proportions of hydrochloric and sulphuric acids adsorbed by individual soils are approximately the same, but those of acetic acid are much smaller. The differences between these values is a measure of the adsorptive power of soils ("volumetric adsorptive value"). Variations in the latter value in numerous soils were closely parallel with those obtained by the use of dyes and of centrifuged milk.

A. G. POLLARD.

**Fungus flora of the soil.** H. L. JENSEN (Soil Sci., 1931, 31, 123—158).—The fungi present in numerous Danish soils of varying type and  $p_H$  are examined and characterised. Fungal numbers are not related to soil type or reaction except that heavy soils usually contain few fungi. The liming of acid soils increases the numbers of bacteria and *Actinomyces*, but has little effect on the fungi. Manurial treatment (notably with farmyard manure) increases the number of fungi, bacteria, and *Actinomyces* in soils. In soils of  $p_H > 6.0$  there is a definite correlation between the numbers of fungi and of bacteria plus *Actinomyces*. The majority of soil fungi decompose cellulose, and their development in both acid and alkaline soils is stimulated by additions of cellulosic material. The proteolytic power of many soil fungi is considerable.

A. G. POLLARD.

**Lime studies. Significance of buffering in practical liming questions; the rule of the base-fixing adsorption zone of soils; buffer grouping, and a new classification of soils in relation to**

**buffering.** S. GOY [with O. ROOS] (Z. Pflanz. Düng., 1931, 20A, 1—43; cf. B., 1931, 454).—New methods of grouping soils according to their buffer values are described. Within the base-fixing area of the buffer curves of soils there is a zone corresponding to  $p_H > 5.5$  in which definite plant injury occurs, and a zone of possible injury the characteristic  $p_H$  values of which vary with the buffer values, being high in poorly buffered soils and *vice versa*. The relative importance of  $p_H$  values of total and exchange acidity in respect of lime-requirement determinations is discussed.

A. G. POLLARD.

**Method of oxidising and dissolving soil for the determination of total and filterable manganese and phosphorus.** E. M. ENMERT (Soil Sci., 1931, 31, 175—182).—The soil sample is prepared for analysis of total manganese and phosphorus by treatment with sulphuric acid and sodium chlorate. The available manganese and phosphorus is determined by shaking soil with water (2 c.c. per g. of soil) for 10 min. and filtering through a No. 2 Whatman filter paper. Colloidal matter present in the filtrate is assumed to be available to the plant.

A. G. POLLARD.

**Nature of soil acidity as affected by the silica : sesquioxide ratio.** L. D. BAVER and G. D. SCARSETH (Soil Sci., 1931, 31, 159—173).—The total base-exchange capacity,  $p_H$ , and physico-chemical constants of the acid of the adsorptive complex ("soil acid") for a number of soils are recorded. Soil acids of different soils are dissimilar and in weathered soils are characteristic of the type of weathering rather than of the parent rock. The colloids from weathered soils have high silica : sesquioxide ratios, buffer capacities, and base-exchange capacities and exhibit the greater acidity. The buffer capacity of the colloids depends mainly on the nature of the soil acid. The exchange complex of soils may be formed by the removal of certain constituents from natural aluminosilicates, by mutual flocculation of the colloidal oxides of iron, aluminium, and silica, or by direct precipitation. There is no uniform relationship between the nature of soil acids and the silica : sesquioxide ratio, although in certain groupings of soils direct relationships are indicated. The chemical composition of soil colloids may be represented with advantage in a form in which the free oxides of iron, aluminium, and silica are excluded.

A. G. POLLARD.

**Direct determination of  $p_H$  of soils in their natural moist condition.** A. V. TROFOMOV (Z. Pflanz. Düng., 1931, 20A, 74—111).—The  $p_H$  values of soils, determined by the quinhydrone electrode, vary with the moisture content and are affected by the oxidation-reduction potential of the soil. Methods for determining  $p_H$  values of soils at their natural moisture contents are described, in which a benzoquinone-quinhydrone electrode is used in soils which oxidise quinhydrone and a quinol-quinhydrone electrode for soils reducing quinhydrone. The drying out of soils causes a steady increase in acidity and *vice versa*. The closest relationship between moisture content and  $p_H$  occurs in moisture ranges below maximum capacity. Within a given range of moisture variation the greatest increases in acidity during drying occur in soils of high hygroscopicity.

A. G. POLLARD.

**Determination of the percentage base saturation of soils and its value in different soils at definite  $p_H$  values.** W. H. PIERRE and G. D. SCARSETH (Soil Sci., 1931, 31, 99—114).—A comparison of methods for determining the exchangeable base status of soils is recorded. The percentage base saturation is most satisfactorily calculated from determinations of the total exchange capacity by the barium acetate-ammonium chloride method and of the exchangeable hydrogen by the barium acetate method. It is suggested that the liming of soils results in the formation of an additional adsorptive complex. In soils of the same  $p_H$ , the percentage base saturation varies considerably, the more highly weathered soils showing the lower values. The percentage base saturation of soils of similar  $p_H$  is not related to the nature of the bases present, nor to the content of organic matter, but shows a general correlation with the silica : sesquioxide ratio and the total exchange capacity of the extracted colloids. The avidity or strength of acids present in soils of the same  $p_H$  differs widely, highly weathered soils, in general, containing the weaker acids. Good correlation was observed between the avidities of soil acids and the percentage base saturation at  $p_H$  4.80.

A. G. POLLARD.

**Determination of the phosphate requirement of soils by the Dirks and Scheffer method as compared with the Mitscherlich pot-culture method.** LAMBERG (Superphosphat, 1931, 7, 61—62).—Comparative examination of numerous soils by the two methods showed very close agreement. The Dirks and Scheffer method (B., 1930, 876) is simple, rapid, and eminently suitable for routine work.

A. G. POLLARD.

**Colorimetric determination of phosphate in relation to soil examination.** E. PFUHL (Superphosphat, 1931, 7, 67—69).—Comparative analyses of soil phosphates are recorded and the advantages of the colorimetric method discussed in regard to speed and simplicity of working without undue loss of accuracy.

A. G. POLLARD.

**Mobility of phosphoric acid in soils.** L. SCHMITT (Superphosphat, 1931, 7, 54—58).—Existing data are quoted to show that no appreciable leaching of phosphate occurs in soils after application of superphosphate under normal cultural conditions.

A. G. POLLARD.

**Changes in the availability of phosphorus in irrigated rice soils.** R. P. BARTHOLOMEW (Soil Sci., 1931, 31, 209—218).—Irrigation of rice lands with water containing appreciable amounts of iron, aluminium, and calcium reduced the water-solubility of the soil phosphate. The increased water-soluble organic phosphorus content of the soil, apparent three months after irrigation, is attributed to anaerobic bacterial activity.

A. G. POLLARD.

**Effect of phosphoric acid in accelerating maturity [of sugar beet].** C. BONNE (Superphosphat, 1931, 7, 27—31).—Neither the yield nor the sugar content of sugar beet is reduced by heavy dressings of superphosphate. The ripening of different strains of sugar beet is accelerated to different extents by phosphate fertilisers, which may produce this effect on soils already having a sufficiency of phosphate for the growth of the crop.

A. G. POLLARD.

**Effect of increasing applications of superphosphate on early cabbage.** II. SCHWARZE (Superphosphat, 1931, 7, 63).—Field trials are recorded in which the use of superphosphate in spring and as a top dressing in early summer increased the total yield and materially hastened the maturing of cabbage. A. G. POLLARD.

**Influence of combined nitrogen on growth and nitrogen fixation by *Azotobacter*.** J. E. FULLER and L. F. RETTGER (Soil Sci., 1931, 31, 219—234).—Fixation of considerable amounts of nitrogen by *Azotobacter* occurred in media and in an atmosphere freed from combined nitrogen. Nucleic acid, tryptophan, tyrosine, and guanine slightly reduced nitrogen fixation without affecting the growth of the organism. Peptone stimulated the growth of the organism, but definitely reduced nitrogen fixation. *Azotobacter* utilised glutamic and aspartic acids, cystine hydrochloride, and glycine. Increased growth resulted, but nitrogen fixation was depressed and in some cases actual losses of original nitrogen occurred. Creatine, creatinine, and urea accelerated growth, but practically inhibited fixation. Indole and skatole in relatively small concentrations were toxic to *Azotobacter*. The sulphate, carbonate, nitrate, and chloride of ammonium and the nitrates of sodium and potassium depressed nitrogen fixation to varying extents, but increased the growth of the organisms.

A. G. POLLARD.

**Influence of various non-nitrogenous compounds on the growth of certain bacteria in soils of low productivity.** H. J. CONN and M. A. DARROW (N.Y. State Agric. Exp. Sta., Tech. Bull., 1930, No. 172, 40 pp.).—The absence of certain commonly occurring non-sporing bacteria (notably *B. globiforme*, Conn) from two soils, and their failure to develop in these soils after inoculation, are examined. Addition to the soils of ammonium salts, nitrates, certain organic nitrogenous substances, or the sulphates, phosphates, and hydroxides of calcium, magnesium, sodium, and potassium enabled the organisms to develop. The effect of the alkali salts was more pronounced in soil of high colloidal content. Non-development in the original soils is ascribed to the deficiency of nitrogen in a form suitable for the utilisation of the bacteria, although the total nitrogen content of the soils was relatively high. The nature of the stimulative effect of the alkali salts is discussed and the possibility of utilising the organisms for determining the nitrogen availability in soils is noted.

A. G. POLLARD.

**Plant nutrient content of soils calculated from the Neubauer, the static, and the Mitscherlich methods.** CLAUSEN (Z. Pflanz. Düng., 1931, 10B, 145—155).—In a comparison of the three processes, the value of the static method is emphasised. In general, the Mitscherlich method yields results in closer conformity with those of field trials than does the Neubauer seedling method.

A. G. POLLARD.

**Composition and quality of Pennsylvania cigar-leaf tobacco as related to fertiliser treatment.** D. E. HALEY, J. B. LONGENECKER, and O. OLSON (Plant Physiol., 1931, 6, 177—182).—Fertiliser treatment of tobacco plants did not affect the K : Ca ratio in the cured leaves. The quantity of sulphur in the leaves was not

increased by considerable dressings of potassium sulphate. During the fermentation process of leaf curing there was an apparent increase in the soluble ash constituents of the leaves, a narrowing of the N : C ratio, and little or no loss of nitrogen. A. G. POLLARD.

**Manuring of the principal cereals and their mean yields in Nederling.** J. WEIGERT (Z. Pflanz. Düng., 1931, 10B, 136—142).—Records of ten-year manurial trials are presented and discussed. A. G. POLLARD.

**Nitrogen losses and action of cold- and hot-fermented manures.** D. MEYER [with P. OBST, F. WILCZEWSKI, and W. DIETRICH] (Z. Pflanz. Düng., 1931, 10B, 121—136).—During the cold-fermentation of manure losses of dry matter were higher and of nitrogen were lower than during hot-fermentation (Krantz). In pot cultures the average percentage utilisation of nitrogen from cold-fermented manure was higher than from the hot-fermented product except when drainage from the latter had been prevented during preparation. In field trials hot-fermented manure showed no superiority in crop production or percentage utilisation over the cold-fermented product. A. G. POLLARD.

**Decomposition in, and effect of straw manures on, soils.** H. ENGEL (Z. Pflanz. Düng., 1931, 20A, 43—73).—Relationships between the C : N ratio of straw manures and their ratio of decomposition in soils are examined and discussed in relation to the significance of carbon dioxide and nitrate production during decomposition. High carbon dioxide production and good humification cannot take place simultaneously. The value of straw manures lies mainly in the physico-chemical properties of the humus produced. With narrowing C : N ratio in manures, the carbon dioxide produced during decomposition decreases and nitrification increases. With a C : N ratio in the manure of 20 : 1, fixation of nitrogen appears to cease and a slow nitrification begins. With a wider ratio no nitrification takes place, but with C : N = 12 : 1 16% (approx.) of the total nitrogen appears as nitrate. The ease of decomposition of the organic matter of straw manures decreases with advancing humification. The effect of changes of C : N ratio on the nitrification process in manures varies with the type or organic matter present. No relationship exists between the ammonia-nitrogen content of manure and its action in soil (cf. Barthel, B., 1930, 73). Of the ammonia-free nitrogen content of manure 16—30% was nitrified in soil. A. G. POLLARD.

**Hydrogen-ion and aluminium concentrations in the soil solution, and percentage base saturation as factors affecting plant growth on acid soils.** W. H. PIERRE (Soil Sci., 1931, 31, 183—207).—Pot-culture experiments are described in which the  $p_H$  of a number of soils was artificially varied. The  $p_H$  at which plant injury first appeared in acidified soils varied with the soil type. The concentration of aluminium in the displaced soil solution is not directly related to the  $p_H$  of the soil, nor is it a primary cause of acid injury to plants. The concentrations of calcium and manganese in the soil solution are not related to plant injury. The ratio K : Ca in the soil solution decreases with increasing soil acidity and is connected with plant injury. In soils of similar  $p_H$  the percentage base saturation varies con-

siderably. The latter factor is closely related to plant injury in acid soils. When the aluminium concentration of the soil solution is high, plant growth is affected in soils of higher percentage base saturation than when aluminium is absent or present only in small proportion. A. G. POLLARD.

**Chibnall's method of extraction for investigation of the winter hardiness of plants.** W. E. TOTTINGHAM, R. G. SHANDS, and E. D. DELWICHE (Plant Physiol., 1931, 6, 167—176).—Analyses of the nitrogenous constituents and sugars in the protoplasmic extracts of wheat and lucerne indicates relationships between variations in composition and winter hardiness.

A. G. POLLARD.

**Influence of frequency of cutting on the productivity, botanical and chemical composition, and the nutritive value of "natural" pastures in Southern Australia.** J. G. DAVIES and A. H. SIM (Counc. Sci. Ind. Res., Australia, 1931, Pamphlet 18, 28 pp.).—Maximum removal of calcium from soil by herbage occurred when the latter was cut at intervals of 6—8 weeks. The phosphate content of herbage cut at 2- and 4-week intervals was practically identical, but decreased with less frequent cutting, reaching a minimum when the grass was allowed to reach maturity. With fortnightly cutting the phosphate content was maintained throughout the season. The crude protein content of herbage was greatest when cut at 2- and 4-weekly periods, the value being more than double that of the mature grass. The crude fibre content increased as the frequency of cutting diminished, the minimum being attained with fortnightly cutting.

A. G. POLLARD.

**Food reserves in relation to other factors limiting the growth of grasses.** L. F. GABER (Plant Physiol., 1931, 6, 43—71).—The effects of manuring and of the frequency of cutting on the yield and quality of grasses are examined. Productive capacity depends not only on adequate manuring and moisture supply, but also on the food reserves of the plants themselves. Too frequent or too close cutting may result in slow recovery and poorer yields. Frequent cutting results in heavy withdrawals of available nitrogen from the soil, and nitrogen deficiency becomes the chief factor in growth limitation. Where adequate supplies of nitrogen are present in soils, the consumption of carbohydrate reserves within the plant is rapid and slow replenishment may restrict further growth. A. G. POLLARD.

**Preservation of [green] feeding-stuffs.** A. KROCEK (Z. Zuckerind. Czechoslov., 1930, 55, 79—85).—In the silage of green fodder, a much nearer approach to pure lactic fermentation can be attained in concrete silos than in earth trenches. In the former the anaerobic conditions in the closely packed material repress acetic organisms, and respiration is too restricted to produce the rise in temperature necessary for active butyric fermentation, except with very woody material which, even when finely chopped, retains a considerable amount of air. Very moist or sappy material, especially if rich in proteins, suffers badly from soil bacteria which produce ammonia. The only sure safeguard against these is partial withering before silage. Where this is not practicable the material may be rendered more

absorbent, and therefore less wet, by very fine chopping or by admixture of absorbent materials; or a proportion of beet leaves or molasses may be incorporated, the sugar in which favours lactic fermentation. Tabulated data for stored green fodder, in most cases beet leaves, show a much smaller production of acetic and butyric acids in concrete silos than in earth trenches, lactic acid representing 70–80% of the total free acids in the former, as compared with 40% on the average in silage from earth trenches.

J. H. LANE.

**Rôle of nitrogen in the intensive working of meadows.** C. MATIGNON (Chim. et Ind., 1931, 25, 271–275).—A description is given of the intensive grazing systems advocated in England and of the recent adaptation of the principle to the growth of cereals for cattle feeding in the United States. A. G. POLLARD.

**Measurement of the nitrate-assimilating power of soils.** R. A. PENDLETON and F. B. SMITH (Proc. Iowa Acad. Sci., 1929, 36, 75–78).—Soils have a definite nitrate-assimilating power which may be favoured by addition of organic matter. CHEMICAL ABSTRACTS.

**Effect of nitrogen fertilisers on nitrification.** R. A. PENDLETON, P. E. BROWN, and F. B. SMITH (Proc. Iowa Acad. Sci., 1929, 36, 99–103).—The fertilisers, particularly sodium nitrate, increased the nitrate content and stimulated the nitrifying power of the soil.

CHEMICAL ABSTRACTS.

**Nitrogen fixation by *Rhizobium meliloti* and *R. japonicum*.** G. G. POHLMAN (J. Amer. Soc. Agron., 1931, 23, 22–27).—Under suitable cultural conditions certain species of *Rhizobium* may be able to fix nitrogen in absence of the host plant.

CHEMICAL ABSTRACTS.

**Spontaneous culture method for studying the non-symbiotic nitrogen-fixing bacteria of the soil.** R. H. WALKER and J. L. SULLIVAN (Proc. Iowa Acad. Sci., 1929, 36, 53–61).—A discussion. The test may serve as a biological comparison of the fertility of soils.

CHEMICAL ABSTRACTS.

**Variations in soil reaction affect nitrification.** P. E. BROWN and G. V. C. HOUGHLAND (Proc. Iowa Acad. Sci., 1929, 36, 93–97).—When differences in  $p_H$  are not great, the influence of greater acidity may be important; where wide variations occur, the effects of reaction may overshadow those of treatment. High acidity retards the nitrification process.

CHEMICAL ABSTRACTS.

**Nitrogenous fertiliser prepared by the Muhlert process.** F. GIESECKE and F. KLANDER (J. Landw., 1931, 79, 69–86; cf. Muhlert, B., 1930, 129).—Losses of nitrogen from the material (essentially ammonium sulphate and calcium carbonate) are relatively small. In pot trials the fertiliser proved as efficient as ammonium sulphate and should be valued on its ammonia content.

A. G. POLLARD.

**Soil-reaction effects on *Phymatotrichum* root rot.** W. N. EZEKIEL, J. J. TAUBENHAUS, and E. C. CARLYLE (Phytopath., 1930, 20, 803–815).—Cotton plants were grown in soil artificially acidified to varying extents and infected with *Phymatotrichum*. With increasing acidity there was a marked diminution in root rot at about  $p_H$  6.0, but none appeared at  $p_H$  5.0. Soil treatment with sulphur decreased root rot, but the

acidity produced did not extend to a sufficient depth to ensure complete control even when the acidity of the surface soil was sufficient to cause plant injury.

A. G. POLLARD.

**Factors influencing the severity of root-rot troubles of sugar cane.** H. H. FLOR (Louisiana Agric. Exp. Sta. Bull., 1930, No. 212, 40 pp.).—No evidence was obtained indicating that inorganic salts or soluble toxins were concerned in the incidence of root rot. Washing the soil did not affect the growth of sugar cane, but steaming the soil induced a more rapid growth. *Pythium* did not grow in culture solutions of  $p_H < 5.2$  or  $> 10.0$ , and was more tolerant to alkalinity than to acidity. Environmental and biological factors are considered.

A. G. POLLARD.

**Preliminary survey of some of the soils in Kenya.** D. S. GRACIE (Dept. Agric., Kenya, Bull., 1930, No. 1, 131 pp.).

**Phthaleinoscope.**—See I. Blood-albumin.—See XIX. Nicotine in insecticides.—See XX.

PATENTS.

**Granulation of fertilisers by spraying molten or liquid materials.** W. K. HALL, F. HEYWOOD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 345,658, 19.12.29).—A whirling motion is imparted by a rotating paddle wheel to a supply of molten ammonium nitrate etc. in a steam-jacketed vertical cylinder so that the material issues through a relatively large orifice (e.g., 1 in. in diam.) at the bottom in the form of a conical curtain which breaks up into drops before solidification. The shaft bearing the paddle wheel may also be provided with inclined vanes to impart a downward thrust to the material.

L. A. COLES.

**Spray oils [for plants etc.].** W. J. YATES, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 1,778,239–40, 14.10.30. Appl., 15.5.29).—A petroleum oil-water emulsion spray contains (A) up to 1% (calc. on the weight of oil) of a naphthylamine, (B) quinol or a chloro-substitution product thereof.

L. A. COLES.

**[Seed and plant] disinfectant composition.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,008, 28.10.30. Appl., 19.5.28).—The composition comprises a mercury alkyl halide (mercury ethyl chloride), a soluble thiosulphate, sulphite, sulphide, thiocyanate, or xanthate (preferably sodium thiosulphate), and an inert powder, e.g., infusorial earth.

L. A. COLES.

**Drying of grass etc.**—See I. Fertiliser materials.—See IX. Alkaloids from cocoa products.—See XX. Sewage sludge.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Factors influencing the ash content and sugar recovery.** J. G. THIEME (Archief, 1930, 38, 713–727).—During 1929 some factories in Java produced molasses of a very high purity, notwithstanding their modern equipment. This was traced to an abnormal composition of the non-sugars, especially in inorganic matter. It appears that ever since 1915 the ash factor of the juice of the cane has been gradually increasing, with a sudden rise during 1926–1929, which period coincides with the introduction of the POJ 2878 cane, viz., from

20.8 to 24.2. It would seem that a high ratio of ash to non-sugars is a characteristic of the modern canes, resulting in lower yields in the factory.

J. P. OGILVIE.

**Imbibition [in sugar factories].** N. M. RYDLEWSKI (Internat. Sugar J., 1931, 33, 40—41).—Experiments carried out in a Cuban factory using two mill tandems operating under conditions generally as similar as possible, except that in the first the imbibition water was applied to the bagasse entering the mills, and in the second on emerging, gave figures clearly in favour of the latter procedure.

J. P. OGILVIE.

**Carbonatation [of beet juices]. II. Influence of the reaction medium on the hydration of carbon dioxide in alkaline solutions.** J. DEDEK and J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1930, 55, 43—51).—The interaction between sodium hydroxide and dissolved carbon dioxide, which takes a measurable time in dilute aqueous solutions, is retarded in presence of sucrose or dextrose. Sucrose exerts its maximum effect, 5-fold retardation, at about 15% concentration, which is its concentration in raw beet juice; above this concentration the retardation is less, and above 50% it changes to acceleration. The retarding effect depends on the absolute concentration of the sugar, and not on the ratio of sugar to reacting substances. It is therefore due to an effect on the medium, but the important factor is not viscosity, for glycerol causes acceleration, and starch paste and gum arabic produce a retardation very slight in proportion to their effect on viscosity. The retarding or accelerating effects of different substances present together are not strictly additive.

J. H. LANE.

**Course of the first carbonatation [of beet juices]. II. Factory tests.** J. DEDEK and T. NIELSEN (Z. Zuckerind. Czechoslov., 1931, 55, 312—323; cf. B., 1930, 1125).—Alkalinity determinations made during the first carbonatation, using phenolphthalein and methyl-red, showed a distinct difference between the two readings, both running parallel at the beginning, but the latter subsequently remaining at a constant value much longer than the first. Observations were also made of the manner in which the dry substance, polarisation, purity, and electric conductivity fall during the course of carbonatation.

J. P. OGILVIE.

**Adsorption of the calcium salts of fatty acids in carbonatation [of beet juice].** T. NEMES (Z. Zuckerind. Czechoslov., 1930, 55, 159—162).—Salts of the fatty acids are adsorbed by the calcium carbonate formed during the carbonatation of raw beet juice, and the more readily the more complex is the fatty acid concerned.

J. P. OGILVIE.

**Influence of products of dextran fermentation (*Leuconostoc mesenteroides*) on the liming and carbonatation of [beet] juice.** W. KONN (Z. Zuckerind. Czechoslov., 1930, 55, 131—139).—Gelatinous matter formed by *Leuconostoc mesenteroides* was obtained from the pulp-catchers of a beet-sugar factory, and washed free from soluble matters. Suspensions containing 0.5% of dry matter, in water and in 15% sucrose solutions, were heated at 85° with 2% of pure lime for various periods and carbonated. The substance dis-

solved more or less during liming, according to the duration of heating, but it was almost entirely eliminated by subsequent carbonatation, whether the liquids were filtered after liming or not. Beet juice containing such matters is best heated to 85° before addition of lime, maintained at this temperature for 5—10 min. after liming, and then carbonated as usual, preferably with a further addition of lime before the second carbonatation.

J. H. LANE.

**Conductometric study of over-carbonatation [of sugar solutions].** K. ŠANDERA and V. PREININGER (Z. Zuckerind. Czechoslov., 1930, 55, 115—119; cf. B., 1930, 582).—In the carbonatation of sucrose solutions containing pure lime, the conductivity curve, plotted against diminishing alkalinity, showed a sharply defined minimum at the neutral point to phenolphthalein. In presence of 0.2% of potassium hydroxide the minimum occurred at an alkalinity of 0.04% CaO. In presence of 0.04% of ammonium hydroxide, which is only slightly more than beet juice contains after liming, the minimum occurred at an alkalinity of 0.02% or 0.032% CaO, according as pure lime or technical lime containing 3% of magnesia was used. On saturating beyond the minimum, the increase in conductivity for a given fall in alkalinity was much greater with the technical than with the pure lime, owing mainly to interaction of the ammonia with the magnesia in the lime. Among alkalis, ammonia has thus an exceptional influence on the optimum end-point of carbonatation, as judged by the elimination of electrolytes, especially where impure lime is used, and in this case it also increases the amount of electrolytes redissolved when carbonatation is carried beyond the optimum point.

J. H. LANE.

**Evaporation of [beet] juices in the presence of carbonatation scums, sodium sulphite, and active carbons.** V. STANEK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1931, 55, 339—348; cf. B., 1929, 572).—Evaporation of clarified beet juice in the presence of some of the last carbonatation scums gave a lighter juice than when the clear juice was so treated. Similarly when 0.0025—0.2% of sodium sulphite was used there was a striking improvement in the colour, though additions above 0.2% failed to increase the effect. Very small additions of active carbon ("Norit") gave a similar result.

J. P. OGILVIE.

**Filtration of the whole juice in sulphitation.** W. C. NIEBOER (Archief, 1930, 38, 499—505; Internat. Sugar J., 1931, 33, 72—73).—In order in the sulphitation method of clarification to filter the whole of the juice, an increase of 50% of the filter-press area compared with that previously used for the mud alone should suffice. A greater addition of lime is not advised to assist the filtration, but the presses should be pre-coated with kieselguhr. Whole-juice filtration is not only more rapid and economical, but improves the quality of the white sugar obtained.

J. P. OGILVIE.

**Adsorption [by carbons] from sugar solutions. II. Adsorption of water.** J. VAŠÁTKO and J. VÁCHA (Z. Zuckerind. Czechoslov., 1931, 55, 183—192).—Different carbons used for decolorising in the sugar industry can be divided into two groups according to their power of adsorbing water from aqueous solutions

of sucrose: (a) Standard-Norit, Polycarbon, and Supra-Norit; (b) Carboraffin, Supra-Norit 5X, and Radit, this latter group adsorbing much more water from sucrose solutions than the first, being characterised by great porosity. Between the two groups lies Supra-Norit 3X.  
J. P. OGILVIE.

**Decomposition of the reducing sugars in alkaline medium during [cane] sugar manufacture.** K. D. DEKKER (Internat. Sugar J., 1930, 32, 641—642).—Decomposition of the reducing sugars occurs as soon as cane juice becomes slightly alkaline, a mixture of dextrose, laevulose, and mannose being formed in equilibrium with the decrease of the cupric reducing power and an increase of colour, total acidity, and calcium salts. Among the acids produced are saccharinic and lactic. Glucose is also formed, finally to accumulate in the molasses to the extent of 5—12%.  
J. P. OGILVIE.

**Determination of calcium salts in cane juices and syrups.** M. VAN DER KREKE and K. D. DEKKER (Meded. Proefstat., Java, 1930, 1—11; Internat. Sugar J., 1931, 33, 73).—The modification of the soap test elaborated by Spengler and Brendel (Internat. Sugar J., 1929, 91) is recommended, being found to give results agreeing closely with those found by oxalate precipitation.  
J. P. OGILVIE.

**Determination of natural alkalinity [of beet juice].** I. B. MINTZ and others (Nauk. Zapiska, 1930, 9, 246).—A modification of the Duwell and Solon method (cf. Internat. Sugar J., 1930, 366) is recommended, in which the sample is carbonatated until the pink obtained by the addition of phenolphthalein has disappeared, after which the liquid is boiled, restored to its original volume, and finally titrated with 0.2N-hydrochloric acid.  
J. P. OGILVIE.

**Rapid determination of the true purity of syrups, using the conductivity depression method.** J. H. ZISCH (Facts about Sugar, 1930, 25, 741—745).—A quantity representing 2 g. of the syrup with 10 c.c. of dilute hydrochloric acid are made up to 100 c.c., mixed, and the resistance of the solution is read in ohms (*A*), the conductivity of the acid used being also ascertained (*B*); then *B*—*A* gives the conductivity depression, values for which corresponding to different true purity values for syrups have been compiled. J. P. OGILVIE.

**Dilution [of sugar juice] in relation to comparative purities.** N. DEERR (Internat. Sugar J., 1930, 32, 616—617).—Mathematical evidence is given in support of the following statement given in the "Methods" of the Hawaiian Sugar Technologists' Association: "To be strictly comparable, gravity solids determination should be made in solutions of the same non-sugar concentration."  
J. P. OGILVIE.

**Determination of unknown losses in juice purification.** J. BOERS (Archief, 1930, 38, 745—754).—After experiments on the determination of sucrose by different double-polarisation methods, the author concludes that in normal cases the unknown losses are not due to chemical losses during the process of juice clarification, or are due only to a small extent. Rather should they be attributed to mechanical losses at the filter-press station and to entrainment in the last body of the

evaporator or the pans, though during these boiling processes it seems certain that some chemical losses do take place.  
J. P. OGILVIE.

**Determination of small proportions of invert sugar in raw sugars.** L. EYNON and J. H. LANE (J.S.C.I., 1931, 50, 85—86 T).—A method is described for the volumetric determination of small proportions of invert sugar (less than 0.3%) in raw sugars with Fehling's solution in presence of methylene-blue as internal indicator. The procedure is the same as that previously described (*ibid.*, 1923, 42, 32 T) save that the determination is carried out in presence of a known quantity of added invert sugar.

**Action of non-sugars of refined sugars on the caramelisation test.** J. PUCHERNA (Z. Zuckerind. Czechoslov., 1930, 55, 143—151).—Various non-sugar substances were mixed with fine granulated and *pilé* sugars in amounts from 0.002 to 0.2%, and after drying at 100° 6.5 g. of the mixture were heated at 170° for 15 min. Substances without effect on this heating test included: sodium, potassium, and barium chlorides, potassium sulphate, sodium oxalate, sodium carbonate, and sodium and ammonium acetates, *i.e.*, salts reacting neutral or alkaline in aqueous solution. Substances causing the formation of colour comprised the amino-acid salts; whilst those inducing inversion, and colour formation after heating with 2N-alkali, were ammonium chloride, sulphate, and oxalate, potassium bisulphate, calcium chloride, ferrous sulphate, and ferrous ammonium sulphate.  
J. P. OGILVIE.

**Hygroscopicity of [beet] sugars.** M. GARINO (Ind. Sacc. Ital., 1930, 23, 483—486).—Of the impurities which normally accompany beet sugar, only caramelan is distributed in any notable quantity in the body of the crystal, caramelan and the ammonium, calcium, and potassium salts of glucic and apoglucic acids being found in traces only in the crystal network. Caramelan is the impurity which chiefly confers hygroscopicity on the sugar, although this defect in the case of white beet sugar may be due to a very thin coating of calcium chloride.  
J. P. OGILVIE.

**Deterioration of Philippine sugar at varying degrees of humidity.** Q. D. RENDON (Philippine Agriculturist, 1930, 19, 383—396).—Small bags of raw and refined sugars were stored in large desiccators, in which humidities ranging from 50 to 100% were maintained, analyses being made at the end of 60, 90, and 100 days. Although the data showed some variations, in general at humidities above 66% the sugars attracted moisture and deteriorated, the loss of sucrose thus caused increasing with the humidity.  
J. P. OGILVIE.

**Determination of starch in barley and in malt.** IV. A. R. LING (J. Inst. Brew., 1931, 37, 216—224; cf. B., 1924, 991).—The determination is first made of the percentages of "apparent maltose" produced when malts, the diastatic powers of which range from 32 to 100° expressed on the Lintner scale, are made to act at 57° under standard conditions on pure wheat starch paste maintained by an acetate buffer at *p*<sub>H</sub> 4.6. For the starch determination in the barley or malt, the latter are ground and, after extraction with alcohol are digested

under the same standard conditions with a malt extract of known diastatic power. The amount of apparent maltose is determined in the mash and corrected for that of the malt extract. The percentage of starch is calculated from the formula  $S = 100M/M_1$ , in which  $S$  is the percentage of starch in the dry barley or malt,  $M$  the percentage of "apparent maltose" produced in the dry pure starch by a malt of definite diastatic power, and  $M_1$  the percentage of "apparent maltose" produced from the dry barley or malt with a malt of the same diastatic power. In the case of barley the results are confirmed by Ewer's method, which depends on acid hydrolysis and polarimetric measurement, but with malt the polarimetric readings must be multiplied by a factor to bring the results into line with those obtained by the diastatic method.

C. RANKEN.

Root rot in sugar cane.—See XVI.

## PATENTS.

**Manufacture of pure l  vulose from inulin.** SCHERING-KAHLBAUM A.-G. (B.P. 345,926, 9.10.30. Ger., 22.10.29).—L  vulose solutions obtained by the acid inversion of inulin, after freeing from acid, are evaporated to dryness *in vacuo* at 50–60  , and the product is stirred with alcohol to dissolve impurities, separated from the liquor, and dried.

L. A. COLES.

**Separating solids from liquids. Polarimeter. Extraction of organic substances.**—See XX.

## XVIII.—FERMENTATION INDUSTRIES.

**Drying of hops. Institute of Brewing Research Scheme. Report on the eighth and ninth seasons' work at the experimental oast, 1928 and 1929.** A. H. BURGESS (J. Inst. Brew., 1931, 37, 186–196; cf. B., 1929, 533).—The time required to dry the hops is related to the air speed, temperature, and depth of loading according to the formula:  $T = [1/(V.P. - v.p.)] [(716 \cdot 5L/a^{1.047}) + (6260/a^{0.39})]$ , where  $T$  is the time of drying in min.,  $L$  the loss of weight in oz. per sq. ft. of kiln floor,  $a$  the air speed above hops in ft. per min.,  $V.P.$  the saturated vapour pressure (in. Hg) of water at the temperature of the air, and  $v.p.$  the vapour pressure (in. Hg) due to the moisture originally present in the air. A reduction of air speed towards the end of drying effects an economy of heat, but entails a larger drying period. The amount of  $\alpha$ -acid decreases with increase of drying temperature, whilst the  $\beta$ -fraction is unaffected. The preservative value of the hops, as shown by a biological test, decreases when they are dried above 70  . Hops preserved by sterilisation have a higher preservative value than the same hops dried in the usual way, and the optimum final moisture content of the hops when removed from the kiln is 5–7%.

C. RANKEN.

**The antiseptic content of worts and beers in relation to that of the hops.** T. K. WALKER and J. J. H. HASTINGS (J. Inst. Brew., 1931, 37, 206–207).—The grading of the relative antiseptic values of hopped worts as determined by the biological method are in close agreement with the preservative powers of the hops as shown by the gravimetric method. On the other hand, the value of the antiseptic powers of beers

made from those worts give the same order of grading as those obtained with the hopped worts, but show different relative values as between individual samples. It is suggested that the varying relative values for the worts and beers is due to the removal during fermentation in each case of an equal quantity of antiseptic power, and not of an amount which bears a definite ratio to that originally present.

C. RANKEN.

**Biological significance of nitrates in brewing liquor.** W. WINDISCH and F. WINDISCH (Woch. Brau., 1931, 48, 106–112).—As little as 2 g. of potassium nitrate per hectolitre added to wort markedly depresses the degree of fermentation and yeast reproduction, as shown by laboratory fermentations using two bottom, one top, and one distillery yeast. The effect is increased, but to a less proportional extent, by 5 and 20 g./hectolitre. It is caused by reduction of part of the nitrate to nitrite, which is not only poisonous to the yeast, but may impart a chlorine-like flavour to the beer (cf. A., 1930, 1477).

F. E. DAY.

**Influence of adsorbents on fermentation.** B. LAMPE (Z. Spiritusind., 1931, 54, 75–76).—The use of aeration of the mash to stimulate the rate of fermentation can be replaced by the addition to the fermentation liquid of 0.1 vol.-% of adsorbents, such as animal and wood charcoal, silicic acid, etc. Acceleration of the fermentation is 3–10 times as great as that produced by aeration, and the method has also the advantage that the production of acetaldehyde, which is increased by aeration, is diminished to such an extent that the use of an aldehyde column in the distillation apparatus is rendered unnecessary. The acceleration of the fermentation may be due to the reduction in the concentration in the liquid of the carbon dioxide by the charcoal, or to the adsorption of the yeast cells due to a mechanical attraction.

C. RANKEN.

**Removal of head during bottom fermentation.** L. SAILER (Woch. Brau., 1931, 48, 129–133).—The apparatus of Hallermann (B., 1931, 362) is criticised as permitting part of the slimy matter to remain in the beer and as allowing the separated material to be continuously extracted by liquid of relatively high alcohol content. An apparatus is described consisting of a grid of vertically flattened tubes, having keeled edges below, which is supported on the surface of the fermenting wort with the lower part of the tubes immersed. The head rises over the grid at first, and as it subsides adheres to the tubes and dries on. The apparatus is provided with lifting tackle for raising the grid during emptying and removing yeast from the fermentation vessel, and with supports for the grid while cleaning. The tubes are arranged to carry the cooling water. It is claimed that by the use of this arrangement the flavour and brilliancy of beer are greatly improved.

F. E. DAY.

**Importance of neutralisation and of the reaction of the medium in the lactic-acetic fermentation.** V. BOLCATO (Giorn. Chim. Ind. Appl., 1931, 13, 69–73).—The reaction of the medium exerts on the activity of lactic-acetic micro-organisms an influence which results in appreciable displacement of the balance of the two principal acids produced. If the acidity is high,



the formation of acetic acid increases, and *vice versa*, but it is not found possible to make acetic acid the main product.

T. H. POPE.

**Determination of potassium and alkalis in raisin wine and must.** L. SEMICHON, M. FLANZY, and (Mlle.) M. LAMAZOU-BETBEDER (Ann. Falsif., 1930, 23, 517—526).—The official French method based on the precipitation of potassium as the bitartrate gives high results, and in its place the following is recommended. The wine etc. (50 c.c.) is evaporated over a hot plate to the consistency of an extract; 25 c.c. of nitric acid and 2 drops of mercury are added, and heating is continued until the liquid clears. Dilution with 10 c.c. of boiling water and addition of excess of saturated baryta causes the precipitation of sulphates and phosphates. Carbon dioxide is then passed through for 2 min., followed by hydrogen sulphide to complete precipitation of the mercury and traces of iron. The solution is boiled, filtered, and made up to volume (100 c.c.). The potassium content of the filtrate is determined by the perchloric acid method. For sodium the alcohol filtrate from the potassium perchlorate can be converted into sulphate and weighed. The method should be carried out according to the detailed instructions given in the paper.

E. B. HUGHES.

**Iron and copper in white wines.** J. RIBÉREAU-GAYON (Ann. Falsif., 1930, 23, 535—544).—The wines contain (per litre) 5—50 mg. Fe and 0.5—3 mg. Cu in the form of salts. These small amounts of metal do not affect the taste, but they have an appreciable action on the clarity of the wine and also in catalysing oxidation reactions. The iron causes turbidity when in the ferric state and copper when in the cuprous state. Conditions allowing oxidation (*e.g.*, exposure to air, or aeration) favour the former and those favouring reduction produce the latter kind of turbidity. The presence of copper increases the susceptibility of the wine to turbidity by the iron. Both iron and copper catalyse oxidation of the sulphur dioxide in wine. Turbidity either from wine or copper does not readily appear in wine stored in casks, as aeration is then insufficient to oxidise the iron, but enough to maintain the copper in the cupric state.

E. B. HUGHES.

**Acidity of wines with regard to [French] legal definition.** L. FERRÉ (Ann. Falsif., 1931, 24, 75—80).—In definitions of the acidity of wine some standard  $p_H$  for the neutral point must be fixed, as the tannins present affect the end-point in titrations with alkali, using certain indicators. Litmus gives the best results.

E. B. HUGHES.

**Determination of methyl alcohol.**—See III. **Dextran fermentation of beet juice. Starch in barley etc.**—See XVII.

#### PATENTS.

**Manufacture of yeast producing high yields.** R. McD. ALLEN and F. E. TIMMER, Assrs. to VITAMIN FOOD CO., INC. (U.S.P. 1,775,800, 16.9.30. Appl., 14.12.25).—Yeast is grown in an intensely aerated and very dilute wort which contains molasses, ammonium sulphate, tricalcium phosphate, and, if desired, small amounts of potassium and magnesium sulphates.

C. RANKEN.

**Manufacture of denatured alcohol.** G. H. CONDUCT (U.S.P. 1,777,035, 30.9.30. Appl., 21.12.23).—The liquid containing alcohol is sprayed into a chamber heated to above the b.p. of the alcohol by a furnace supplied with solid organic fuel. The products of the incomplete combustion of the fuel are drawn into the chamber through a perforated bottom, and part with their content of substances of a denaturant nature to the volatilised alcohol, which is thereupon withdrawn to a condenser by means of a fan placed at the exit end of the condenser.

C. RANKEN.

**Manufacture of organic acids [by fermentation].** F. J. CAHN (B.P. 345,368, 2.1.30. U.S., 4.1.29).—Organic acids, such as citric acid, are produced by growing acid-producing fungus on slices of plants containing lævulose or carbohydrates yielding lævulose. The fermentative action is improved by powdering the plant sections with an alkaline material, and by breaking down superficially the cellular tissue of the sections by successive freezing and thawing. If necessary, the plant sections may be intermixed with inert materials, such as stoneware, Raschig rings, etc.

C. RANKEN.

**Increasing the potassium salt content of distiller's waste.** K. CUKER (U.S.P. 1,778,381, 14.10.30. Appl., 25.3.27. Czechoslov., 30.3.26).—The distiller's wash, which is clarified by centrifuging, is used instead of water to prepare the next molasses mash, and the process is repeated until the concentration of the salts in the centrifuged liquor is so high as to interfere with the fermentation.

C. RANKEN.

#### XIX.—FOODS.

**Physico-chemical constitution of spray-dried milk powder.** L. H. LAMPITT and J. H. BUSHILL (J.S.C.I., 1931, 50, 45—54 *tr.*).—A characteristic difference between full-cream roller-process milk powders and full-cream spray-process milk powders, which is accounted for by the difference in manufacture, is that in the former the fat is readily extractable in the cold by solvents, whereas in the latter only a small proportion of the fat can be so removed. It is shown that the fat of the spray-process powder becomes "free" when the powder has absorbed a certain minimum amount of moisture ("critical moisture content"), viz., 8.6—9.2% for ordinary full-cream powder (26% of fat). In the case of freshly made spray powders the fat becomes "free" at the lower critical limit only after a certain time (up to 4 days), whereas with older powders less time is required. This "freeing" of the fat is shown to be associated with crystallisation of the lactose of the milk powder. The change by which the fat becomes "free" is irreversible. A standard method for the determination of the "free" fat of milk powder is described.

E. B. HUGHES.

**Nutritional value of raw and pasteurised milk.** S. BARTLETT (J. Min. Agric., 1931, 38, 60—64).—The results obtained by Leighton and McKinlay in their report on an investigation in Lanarkshire schools (*cf.* "Milk Consumption and the Growth of School Children," Dept. Health, Scotland) have been recalculated, using larger groupings. The reclassification shows that the consumption of raw milk produces greater increase in weight and height than does pasteurised milk.

E. B. HUGHES.

**Fat of sow's milk.** O. LAXA (Ann. Falsif., 1931, 24, 87—88).—A complete analysis is given.

E. B. HUGHES.

**Determination of milk proteins.** IV. (A) Combined, (B) separate, determination of albumin and globulin. G. M. MOIR (Analyst, 1931, 56, 228—235).—(A) For the combined determination trichloroacetic acid is added, to a concentration of 4%, to the filtrate after removal of the casein (cf. B., 1931, 512), the mixture is heated for  $\frac{1}{2}$  hr. at the b.p., filtered, and the precipitate washed with 1% trichloroacetic acid. (B) After separation of the casein the filtrate may be neutralised to phenolphthalein and saturated with magnesium or sodium sulphate, when the globulin is filtered off, or alternatively, the casein and globulin may be precipitated together from the milk, after neutralisation, and the nitrogen due to casein deducted to give the figure for globulin. The total protein-nitrogen minus that due to casein and globulin will give albumin-nitrogen. T. McLACHLAN.

**The diphenylamine test for nitrates in milk as a means of detecting added water, and the effect of drenching cows with "nitre."** D. R. WOOD, E. T. ILLING, and A. E. FLETCHER (Analyst, 1931, 56, 248—249).—Lerrigo's test (cf. B., 1930, 837) is capable of detecting 5% of added water containing 0.5 pt. per 100,000 of nitrogen as nitrates. Maximum doses of "nitre" fed to cows over a period of a week do not cause nitrate to occur in unadulterated milk. Filter papers may contain nitrates, and in consequence give fictitious results. T. McLACHLAN.

**Manufacture and analysis of chocolates.** R. LECOQ (Ann. Falsif., 1931, 24, 11—22, 96—104).—Typical manufacturing formulæ and analyses of various types of chocolates are given. It is shown that reducing sugars are formed from the sucrose due to caramelisation according to the degree of heat-treatment of the chocolate in manufacture. The method of calculation from analysis of the approximate composition of chocolate, including added shell and germ, milk, starch, coffee, or nut (praline), is given. E. B. HUGHES.

**Investigation and evaluation of honey.** K. BRAUNSDORF (Z. Unters. Lebensm., 1930, 60, 575—588).—In the author's version of Gothe's test (B., 1914, 1217; 1915, 1024), 5 c.c. of fresh 20% honey solution and 1 c.c. of a fresh, filtered 1% solution of starch are maintained for 1 hr. at 40—45°, cooled in ice, and a drop of 0.1N-iodine solution is added. A blue colour corresponds with a diastatic power of 0—1 of Gothe's scale, violet to brown-red 2.5—8.3, brown to green-brown 10.5—13.9, olive >17.9. Forty genuine or adulterated German and other honeys of known history were tested in this way and by the Ley, Lund (tannin), Auzinger, Fiehe, and Fiehe-Kordatzki tests (cf. B., 1929, 146, 375, 955). It is concluded that genuine German bee-honeys have a diastatic power of >17.9, and yet may show traces of hydroxymethylfurfuraldehyde and give a doubtful Fiehe test. A value of 10—17.9 with a hydroxymethylfurfuraldehyde reaction and a normal Lund test indicate genuine honey which has been heated, but which, according to German regulations based on the diastatic power, is suitable only for cooking purposes. Values of <10.9 indicate adulteration. Fiehe's test

(*loc. cit.*) is improved by addition to the residue from an ether extract from 10 g. of honey, of 0.2 c.c. of a 1% solution of resorcinol in hydrochloric acid (*d* 1.19). The faint red precipitate indicating a positive reaction is associated with a positive hydroxymethylfurfuraldehyde test and a diastatic power of 10—20. Apparently anomalous results may be due to unequal degrees of heating of inner and outer layers of the sample. It is suggested that genuine honeys with abnormally low diastatic powers are analogous to genuine milks low in fat. J. GRANT.

**Determination of sand in vegetable feeding-stuffs.** I. RÜB (Chem.-Ztg., 1931, 55, 221—222).—5 G. of the air-dried sifted meal are treated with carbon tetrachloride for 1 hr. (with occasional stirring) in a Spaeth sedimentation cylinder. Foreign mineral matter detaches itself readily from the material and sinks to the bottom. The upper layer is decanted and the sediment rinsed with carbon tetrachloride into a platinum dish, evaporated, and gently ignited to burn off adherent fat before weighing. In the isolated case of rice meal the silicious epidermal hairs collect in the sediment, which must therefore be warmed (on the water-bath) with 20 c.c. of 3% caustic soda for 15 min., filtered, washed, and ignited. E. LEWKOWITSCH.

**Blood-albumin.** H. STADLINGER (Farben-Chem., 1931, 2, 125—127, 172—174).—Details are given of the preparation of blood serum from coagulated blood and the subsequent isolation of albumin of different grades (cf. B., 1928, 158), leaving a residue of blood meal. The products are tested for colour, odour, consistency, solubility, calcium and water contents. Distinction is drawn between blood-albumin and "defibrinated dried blood," an inferior product. The chief applications of blood-albumin are as plywood adhesive, and in the leather, paper, and textile industries for various coatings etc., replacing similar products in which casein, egg-albumin, etc. are used. "Blood meal" is used for fodder and manure. S. S. WOOLF.

**Detection of vitamin-A in medicinal food preparations.** H. MATTIS and E. NOLTE (Arch. Pharm., 1931, 269, 22—24).—Addition of approx. 0.017 g. of a lipid extract, prepared from the medicinal foods "Novo-Tropon" and "Ferripan," to a vitamin-A-free daily diet is sufficient to cure keratomalacia and restore normal growth in rats. H. E. F. NOTTON.

**Determination of methyl alcohol.**—See III. **Preservation of green feeding-stuffs.**—See XVI.

#### PATENTS.

**Production and sterile packing of bread and pastry.** C. MEYER (B.P. 345,885, 30.5.30. Ger., 31.5.29).—Bread is baked in a metal container and transferred to a preheated receptacle without access of air. The receptacle is then sealed and again heated to ensure sterility. E. B. HUGHES.

**Milk evaporator and deodoriser.** C. M. PETERSON, Assr. to WARD DRY MILK Co. (U.S.P. 1,778,959, 21.10.30. Appl., 28.5.26).—Milk, cream, buttermilk, etc. is forced under pressure through rotating nozzles each connected with a jacketed tube which may be heated. The liquid

is thus partly evaporated, the vapours being drawn off by means of a rotary blower. A vacuum may also be produced in the apparatus. It is claimed that deodorisation also takes place. E. B. HUGHES.

**Preservation of confectionery, fruit, and other edible substances.** A. F. MACKENZIE (B.P. 345,904, 3.7.30).—A couverture for sweetmeats contains a mixture of illipé butter, sugar and dry flour; colouring and flavouring matter, milk solids, and nuts may be added if desired. E. B. HUGHES.

**Manufacture of compositions for colouring or flavouring foodstuffs.** R. E. ELLIS. From H. KOHNSTAMM & Co., Inc. (B.P. 345,363, 30.12.29).—A mixture of approx. 20% of colouring matter, 30% of colour solvent or diffusion material such as glycerin, 48% of lactose or other sugars, and 2% of a colloidal suspension of a vegetable gum (gum arabic) in water is claimed. The solid preparation is finished in slab form. E. B. HUGHES.

**Egg products [for use in mayonnaise etc.].** A. E. WHITE. From EMULSOL CORP. (B.P. 346,031, 1.1.30).—See U.S.P. 1,744,575; B., 1930, 530.

**Ovens for the heat treatment of edibles.** A. S. JOHN (B.P. 346,479, 21.1.30).

**Drying apparatus [for milk etc.]. Centrifugal machine for milk. Filter device [for fish offal etc.]. Filtration process.**—See I. Yeast.—See XVIII. Alkaloids from cocoa. Extraction of organic substances.—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Ammonia content of cigar smoke.** D. E. HALEY, C. O. JENSEN, and O. OLSON (Plant Physiol., 1931, 6, 183—187).—Apparatus for the examination of the products from the intermittent smoking of cigars is described. In incompletely fermented tobacco samples, no relation between manuring and the ammonia content of the smoke was apparent. A. G. POLLARD.

**Determination of nicotine in solution and in insecticides.** A. SABATIE (Ann. Palsif., 1930, 23, 544—547).—A combination of three methods is suggested: (1) Steam-distillation of the solution made alkaline with magnesia or caustic soda and examination of each fraction of the distillate separately in a 5-dm. tube by means of a polarimeter, the proportion of nicotine being calculated by means of Biot's formula. (2) Titration with 0.1N-acid, using methyl-red as indicator. (3) Precipitation as the silicotungstate by Bertrand's method. The substitution of pyridine for nicotine gives results in (2) and (3) higher than in (1), and of ammonia a result in (2) higher than those in (1) and (3). E. B. HUGHES.

**Determination of morphine in opium.** C. G. VAN ARKEL and P. VAN DER WIELEN (Pharm. Weekblad, 1931, 68, 309—316).—The method of the Dutch Pharmacopœia is very unsatisfactory when gum has been added to the opium. The tartaric acid-alcohol method of van der Wielen gives much more uniform results, but when the opium content is high the results are too low.

By using larger quantities of the acid and alcohol, and by partial neutralisation with sodium hydroxide before evaporation, accurate results are obtained in all cases.

S. I. LEVY.

**Determination of morphine. II. Determination in presence of other opium alkaloids.** II. BAGGESGAARD-RASMUSSEN and F. REIMERS (Dansk Tids. Farm., 1931, 5, 21—34).—The official Danish preparation, tetrapon, which contains morphine, narcotine, codeine, and papaverine hydrochlorides, and similar mixtures (e.g., pantopon) may be analysed by a modification of the authors' method (A., 1931, 371). Tetrapon (0.4 g.) in water (20 c.c.) with 2N-sodium hydroxide (4 c.c.) is twice extracted with a mixture of chloroform (10 c.c.) and ether (30 c.c.) and once with chloroform (10 c.c.). The combined extracts contain the colouring matter, chryso-idine, and all the alkaloids, except morphine. Addition of ammonium chloride (1 g.) to the aqueous layer liberates the morphine, which is extracted with isopropyl alcohol-chloroform and determined as described previously. II. E. F. NOTTON.

**Essential oils of *Metrosideros*.** R. GARDNER (J.S.C.I., 1931, 50, 141—144 r).—The essential oils of the following species of *Metrosideros* (family *Myrtaceæ*), occurring in New Zealand, are described: *M. scandens*, *Parkinsonii*, *diffusa*, *Colensoi*, *excelsa*, *collina*, *umbellata*, *robusta*, and *perforata*. The oils in all cases contain, and in the first six species mentioned consist mainly of, a sesquiterpene similar in physical constants to cadinene and yielding cadinene dihydrochloride but not cadalene. The sesquiterpene from *M. scandens* is levorotatory but yields *d*-cadinene hydrochloride; all other species give *l*-cadinene hydrochloride. The oils of *M. umbellata*, *robusta*, and *perforata* contain also terpenes (mainly dipentene with less  $\alpha$ - and  $\beta$ -pinene) and some esters.

**Essential oil of leaves of *Chamæcyparis obtusa*, Sieb. et zucc., f. *formosana*, Hayata, or Arisan "Hinoki."** I. K. KAFUKU, T. NOZOE, and C. HATA (Bull. Chem. Soc. Japan, 1931, 6, 40—53. Cf. Uchida, B., 1928, 768).—The leaves on steam-distillation yielded 0.3% of oil having  $d_4^{25}$  0.8988,  $n_D^{25}$  1.4878,  $\alpha_D^{25}$   $-5.83^\circ$ , acid value 0.96, ester value 12.63, after acetylation 54.89. Constituents identified were  $\alpha$ -pinene,  $d$ -sabinene [ $\alpha$ ] $^{17}$   $+86.4^\circ$ , (?)  $\alpha$ -thujene, *p*-cymene,  $\alpha$ - and  $\gamma$ -terpinenes, dipentene, a substance, *chamene*, considered to be a new terpene, b.p. 86—88°/50 mm., 168—170°/760 mm.,  $d_4^{25}$  0.8228,  $n_D^{25}$  1.4686,  $\alpha_D^{25}$   $+35.0^\circ$ , an octenol,  $C_8H_{16}O$ ,  $d_4^{20}$  0.8454,  $n_D^{20}$  1.4441,  $\alpha_D^{20}$   $-10.62^\circ$ , *d*- $\Delta^1$ -terpinen-4-ol, *l*-linalool, acetic acid, an acid,  $C_{10}H_{16}O_2$ , b.p. 133—135°/2 mm.,  $d_4^{20}$  0.9544,  $n_D^{20}$  1.4732,  $\alpha_D^{20}$   $0^\circ$ , and (?) hinokic acid (cf. B., 1928, 768). *Chamene* gives a liquid hydrochloride, b.p. 80—90°/11 mm.,  $d_4^{25}$  1.0196,  $n_D^{25}$  1.4783. Treated with 30% sulphuric it gives *isochamene*,  $C_{10}H_{16}$ , b.p. 88—90°/50 mm.,  $d_4^{25}$  0.8222,  $n_D^{25}$  1.4726,  $\alpha_D^{25}$   $-0.27^\circ$  (which gives  $\alpha$ -terpinene nitrosite), whilst with alcoholic sulphuric acid *chamene* gives *disochamene* b.p. 155—156°/4 mm.,  $d_4^{20}$  0.9150,  $n_D^{20}$  1.5134,  $\alpha_D^{20}$   $-0.7^\circ$ . It is suggested that *chamene* is a cyclopentane derivative.

A. A. LEVI.

**Colloidal aluminium hydroxide.**—See VII. **Cigar-leaf tobacco.**—See XVI. **Medicinal foods.**—See XIX. **Chloroform poisoning.**—See XXIII.

## PATENTS.

**Production of pharmaceutical preparations from plants of the genus *Allium*.** "AGLIONAT" GES.M.B.H. (B.P. 345,600, 23.7.30. Ger., 5.2.30).—Ground plants of this genus, particularly garlic, are heated with water under pressure and a current of air or steam is passed through the hot pulp to remove the odour.

E. H. SHARPLES.

**Recovery and purification of alkaloids from cocoa products.** H. E. POTTS. From MONSANTO CHEM. WORKS (B.P. 345,250, 7.10.29).—To a suspension of the cocoa products a mineral acid is added, followed by an alkaline substance (alkaline-earth oxide or hydroxide), preferably in excess of the amount necessary to neutralise the acid. After separation of the liquid, theobromine is obtained therefrom either by crystallisation, extraction, neutralisation, or by removal of the alkaline earth with carbon dioxide, with or without the addition of a soluble carbonate. It may be purified either by acidification of a filtered, aqueous solution of its alkaline-earth salt or by converting this salt into an alkali salt by addition of alkali carbonate and filtering off the precipitated alkaline-earth salt before acidification. From the extracted cocoa product and the mother-liquors an alkaloid-free product of food and fertiliser value may be obtained by neutralisation of free alkaline-earth oxide with flue gases.

E. H. SHARPLES.

**Extraction of organic substances [of high mol. wt.].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 345,249, 12.9.29).—Alkaloids, bitter principles, sugars, saponins, etc. are extracted from dried vegetable or animal materials containing such substances with anhydrous, or practically anhydrous, liquid ammonia. Extractions of tobacco leaves, quillaia bark, sugar beet, lupin or coffee beans, dry Spanish flies (*Cantharis vesicatoria*), dry milk, etc. are given as examples.

E. H. SHARPLES.

Polarimeter.—See I.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Sensitive photographic plates for the red and infra-red.** G. HAFT and W. HANLE (Z. wiss. Phot., 1931, 28, 374—376).—Five Agfa plates and one Kodak plate have been tested by photographing the spectra of a carbon arc (with a step-wedge screen) and a neon Geissler tube. The general sensitivity (over the range 4500—9000 Å.) of the Agfa plates was considerably the greater; the Kodak plate was sensitive to 8200 Å. and the Agfa plates, on the average, to 8000 Å., one plate being sensitive to 8950 Å.

J. LEWKOWITSCH.

**Formation of photographic images on cathodes of alkali-metal photoelectric cells.** A. R. OLFIN and G. R. STILWELL (J. Opt. Soc. Amer., 1931, 21, 177—181; cf. A., 1930, 1230).—When very small amounts of sensitising materials are admitted to an illuminated surface of freshly distilled metal of a photoelectric cell cathode till a maximum current is recorded, the surface develops the power of recording clear negative photographic images of the source of light. If the sensitive stage is passed, more metal may be distilled on to the surface to restore it. The image is "fixed"

by a slight excess of sensitiser, and by continued alternating treatment a positive image may form. As sensitising agents sulphur vapour, oxygen and hydrogen in 9:1 ratio, hydrofluoric acid, and bromine have been found effective. While the image is forming, the photoelectric sensitivity falls by about 30%, but recovers when the image is fixed.

J. LEWKOWITSCH.

## PATENT.

[Taking pairs of complementary colour graduation value pictures for] colour photography. W. CHAPMAN (B.P. 346,010, 1.1.30).

## XXII.—EXPLOSIVES; MATCHES.

**Velocity of phenomena produced by detonation of solid explosives.** P. LAFFITTE and M. PATRY (Compt. rend., 1931, 192, 744—746. Cf. B., 1931, 224; Payman, B., 1926, 30).—The velocities of detonation and of the consequent shock-wave and luminous gases—these latter at different distances of from 1 to 67 cm. from the column of explosive—have been determined for three densities of loading of each of No. 1 dynamite and tetryl. The results clearly show the greater power of tetryl, and how the velocity of the luminous gases falls off more quickly than that of the shock wave, the distances at which separation of the two occurs increasing with increased density of loading.

C. A. SILBERRAD.

1:3:5-Trinitrobenzene.—See III. Cotton for nitration. Nitrated cellulose.—See V.

## PATENTS.

**Production of explosives.** W. FRIEDERICH (B.P. 345,859, 30.4.30. Ger., 10.10.29).—The cyclic keto-alcohols tetramethylolcyclopentanone and -hexanone and octamethylolcyclohexanediol, and the corresponding alcohols tetramethylolcyclopentanol and -hexanol and octamethylolcyclohexanediol, when nitrated, give explosives having a velocity of detonation of 8000 m./sec. or above and of great stability, their low m.p. enabling them to be cast.

W. J. WRIGHT.

**Heating compositions for blasting cartridges.** D. HODGE and W. ESCHBACH (B.P. 345,284, 11.11.29).—In blasting cartridges containing solid or liquid carbon dioxide, a composition, which is more combustible than the heating composition, is interposed between the latter and the match head. A suitable composition consists of a mixture of lead thiocyanate and potassium perchlorate.

W. J. WRIGHT.

Pulp for explosives.—See V.

## XXIII.—SANITATION; WATER PURIFICATION.

**Mechanism of the activated sludge process of sewage disposal.** E. C. C. BALY (J.S.C.I., 1931, 50, 22—26 T).—The isoelectric point of the colloids in sewage has been determined by the methods of flocculation and cataphoresis. In the absence of electrolytes the isoelectric point is at  $p_H$  4.6, in the presence of 0.17% NaCl it is at  $p_H$  6.5, and in the presence of 3% NaCl it is at  $p_H$  8.3. The most attractive theory of the activated sludge process—namely, the mutual coagulation of colloids of opposite charge—would thus seem to be negatived, since in fresh sewage with  $p_H$  7.4 the colloids

are electronegative as well as the activated sludge particles. No attention, however, has been paid to the electronegative charge on the bacteria and its variation with their activity. When 3% NaCl is added to fresh sewage and the mixture is fully aerated, the bacteria are flocculated by the electropositive colloids within four days. In fresh sewage containing 3% NaCl there exist two types of particle, namely, colloid particles with a larger electropositive charge and bacteria-colloid complexes with a smaller electropositive charge. The essential condition for the successful operation of the activated sludge process is the maximum bacterial activity of the sludge particles. This causes these particles to develop an increased electronegative charge, and it is suggested that the activated sludge process consists in the mutual coagulation of the bacteria-colloid complexes by the activated sludge particles, caused by the difference in charge of the two. It follows that an improvement would be effected by operating the process at  $p_H$  5.8–6, when the whole of the colloids would be electropositive. Considerable benefit would be gained in the preparation of the first quantity of activated sludge by adding 3% NaCl to the sewage, followed by aeration for 4 days, or, still better, by the addition of a cream of fine silt or clay to the saline sewage, followed by aeration for one hour.

**Standard methods for the examination of sewage and sewage sludge. Symposium. I.** J. J. HINMAN, JUN. II. S. E. COBURN. III. A. J. FISCHER. IV. L. H. ENSLOW. V. W. RUDOLFS and H. HEUKELEKIAN. VI. M. M. COHEN. VII. T. C. SCHAETZLE. VIII. E. J. THERIAULT. IX. C. C. HEMMON. X. C. K. CALVERT. XI. W. D. HATFIELD. XII. F. W. MOHLMAN. XIII. A. M. BUSWELL and G. E. SYMONS. XIV. M. LEVINE. XV. W. S. MAHLIE (Sewage Works' J., 1930, 3, 349–352, 353, 353–356, 356–357, 357–360, 360–363, 363–365, 365–367, 367–368, 368–370, 370–374, 374–378, 378–384, 384, 385–386).—III. A method for the determination of putrescible solids in grit samples is described.

VIII. In the determination of dissolved oxygen the use of permanganate is unnecessary in the absence of nitrites or iron. In presence of much organic matter permanganate gives lower results than Winkler's method. Permanganate gives untrustworthy results in sulphite waste liquors. More accurate results are obtained by using chlorine or hypochlorites, the excess of oxidant being removed with iodine. For the determination of biochemical oxygen demand buffered water is recommended.

XIII. A method for determining the quantity of settling solids is described.

XIV. A method for the determination of nitrate in sewage is described.

CHEMICAL ABSTRACTS.

**Function of ripe [sewage] sludge.** H. HEUKELEKIAN (Sewage Works' J., 1930, 3, 313–337).—The age of the sludge affects the rate of decomposition and the gasification of the solids added to it; decomposition should be recently complete. Regulation of  $p_H$  with ammonium hydrogen carbonate and carbonate and potassium hydrogen phosphate compares favourably with maintenance of  $p_H$  by repeated addition of lime.

CHEMICAL ABSTRACTS.

**Rôle of protozoa in [sewage purification by] activated sludge.** R. CRAMER (Ind. Eng. Chem., 1931, 23, 309–313).—In presence of sodium chlorate (0.3%) sewage clarifies in 2–4 days, producing sludge which is indistinguishable from well-conditioned activated sludge and a supernatant liquor free from suspended matter and bacteria. This fact was used as the basis of parallel laboratory experiments which showed that the essential factors in the clarification of sewage by the activated-sludge process are the presence of live protozoa, oxygen in solution, and aerobic bacterial life, and that the rate of clarification is considerably improved by increasing the temperature from 10° to 20°. C. JEPSON.

**Influence of seeding material on [sewage] sludge digestion.** C. E. KEEFER and H. KRATZ, JUN. (Eng. News-Rec., 1931, 106, 474–478).—Seeding sludges, varying in age from 10 days to 8 years, were given daily additions of fresh sludge in amounts equivalent to 2% and 6% of their dry matter content. As measured by the rate of gas evolution, a seeding sludge about 10 weeks old in which digestion was just commencing gave the best result in the early stages, but after 60 days' operation all sludges were giving similar results except the freshest one, which did not commence to digest till after 80 days. A rapid resolution of ether-soluble matter was observed in the first 10 days, but there was also a portion of this material which was only digested with difficulty. Approximately 600 c.c. of methane or 800 c.c. of total gas were obtained per g. of fresh organic matter added or 750–800 c.c. of methane and 1200–1300 c.c. of total gas per g. of organic matter destroyed. C. JEPSON.

**Reclamation of treated sewage.** R. F. GOUDRY (J. Amer. Water Works' Assoc., 1931, 23, 230–240).—In Southern California the lowering of the general level of the ground water by excessive pumping for potable and industrial purposes is causing serious infiltration of sea-water. By suitable treatment it is thought that reclaimed sewage may be used to meet such deficiencies and for many industrial purposes. An experimental plant has been installed at Los Angeles to investigate the possibility of the reclamation of sewage, to determine costs of installation and operation, and to work out refinements in the methods adopted. In this plant the effluent from an activated-sludge plant treating preclarified sewage is superchlorinated to remove phenols and odours, coagulated with ferric chloride, filtered through sand, and finally through activated charcoal before disposal on sand beds. Tests in and around the disposal area show that the 200,000 gals. reclaimed daily have no deleterious effect on the condition of the ground water, and chemical tests indicate that it is definitely superior thereto. C. JEPSON.

**Valuation of carbolic powder.** C. E. COULTHARD (Analyst, 1931, 56, 251).—The usual specification, that powders must contain not less than 15% of "cresylic acids," should be amended, since many powders, e.g., those containing spent gas-lime, may be more efficient, but are condemned under the present regulations. T. McLACHLAN.

**Testing of disinfectants.** E. K. RIDEAL and A. SCIVER (Analyst, 1931, 56, 249–250).—A criticism of the technique of the Patterson-Frederick (cf. B., 1931,

419) and of the Martin-Chick tests. The authors claim an accuracy of  $\pm 5\%$  for the Rideal-Walker test, and that it is equal, or superior, to any other method suggested. T. McLACHLAN.

**Elimination of taste and odour in the water supply of Lancaster, Pa.** E. D. RUTH (J. Amer. Water Works' Assoc., 1931, 23, 396—399).—Prior to April, 1928, the inadequacy of the purification plant treating water from the Conestoga River was responsible for very offensive tastes and the continuous presence of at least an odour of chlorine in the finished water. In that year the feeding of anhydrous ammonia (0.18 p.p.m.) to the raw water by means of a chlorinating machine enabled the post-chlorine dosage to be reduced from 0.45—3.0 p.p.m. to 0.18 p.p.m., with improved bacterial removal. Prechlorination was shown to have no beneficial result. Excessive vegetable growths in summer and autumn were destroyed by copper sulphate (maximum dose 1.0 p.p.m.), which was added with the coagulant, and the dead organic matter was oxidised by potassium permanganate (0.63—5.0 p.p.m.) during autumn to prevent the clogging of the sand filters. C. JEPSON.

**Superchlorination treatment [of water] for taste prevention at Toronto, Ontario.** N. J. HOWARD (J. Amer. Water Works' Assoc., 1931, 23, 387—395).—The difficulties encountered and overcome during the experimental stages of chlorination at Toronto are described. Since 1927 the whole of the supply has been treated at a cost of \$1.81/million gals., of which \$0.72 is due to the cost of superchlorination and dechlorination during periods of probable taste. In this case pollution is accompanied by a high ammonia content, and is subject to rapid variations, hence strict control is necessary to ensure an adequate residual of chlorine. The cost of treatment has been reduced by the use of 1-ton containers of chlorine, and the possibility of a further saving by a reduction in chlorine dosage and subsequent treatment with activated charcoal is being considered. Attention is drawn to the necessity for the study of the effect of temperature, degree and nature of pollution, and the time of contact for each individual water, to secure the best result. C. JEPSON.

**Preammoniation of the filtered water supply of Cleveland, Ohio.** J. W. ELLMS (J. Amer. Water Works' Assoc., 1931, 23, 400—407).—Preammoniation was recommended for the prevention of chlorophenolic tastes in water drawn from Lake Erie in preference to treatment by activated carbon. Anhydrous ammonia (0.096—0.2 p.p.m.) is added to the filtered water before chlorinating, and has successfully prevented objectionable tastes when up to 1.0 p.p.m. of phenol has been present. The cost of treatment is \$0.30 per million gals., and no adverse effect on the bacterial efficiency of the plant has been observed. C. JEPSON.

**Successful superchlorination and dechlorination for medicinal taste of a well supply, Jamaica, N.Y.** F. E. HALE (J. Amer. Water Works' Assoc., 1931, 23, 373—386).—Offensive tastes, due to the effect of chlorine on products introduced into a portion of the ground water by a leakage of petrol at a garage situated  $\frac{3}{4}$  mile away from the nearest supply point,

were prevented by superchlorination, and subsequent dechlorination by sulphur dioxide. The treatment enabled the whole supply to be used for potable purposes and increased the available normal volume by 40% and the peak-load volume by 80%, the cost of chemicals being 92 cents per million gals. C. JEPSON.

**Softening a well-water supply.** N. T. VEATCH, JUN., and B. L. ULRICH (J. Amer. Water Works' Assoc., 1931, 23, 272—275).—Well water at Manhattan, Kansas, with a total hardness of 463 p.p.m., of which only 15 p.p.m. are non-carbonate hardness, and an iron content of 12 p.p.m. is freed from iron by aeration, softened with lime, and then recarbonated with carbon dioxide obtained by the combustion of natural gas; the final water has a total hardness of 124 p.p.m.

C. JEPSON.

**Boiler feed-water.**—See I.

#### PATENTS.

**Treatment of sewage sludges.** H. G. GILLILAND (B.P. 345,194, 17.9.29).—Sludge of high organic matter content, obtained by the action of yeast on activated or precipitation tank sludges, is mixed with powdered carbonaceous material, an acid or alkali, and an oxide, hydroxide, or salt of calcium. The mixture is agitated at 26—93°, filtered while hot, and the resultant cake dried and crushed for use as a fertiliser. C. JEPSON.

**Appliances for purifying [removing water vapour and carbon monoxide from] respirable gases.** L. A. LEVY, D. W. WEST, and R. H. DAVIS (B.P. 345,672, 21.12.29 and 30.6.30).—The impure air is drawn through a canister containing successive layers of a dehydrating agent, *e.g.*, silica gel, of material for indicating by a colour change (observable through a window) when the dehydrating agent is exhausted, *e.g.*, pumice granules impregnated with an anhydrous cobalt or copper salt, and of material for the catalytic oxidation of the carbon monoxide. L. A. COLES.

**Removal of chlorine or hypochlorite compounds from water.** E. BERL (B.P. 344,363, 3.12.29).—The water is treated with lignin or material containing it, *e.g.*, wood which preferably has been treated with steam and partly freed from resin; when the chlorine content of the water is high, the latter should be treated with coke, fuller's earth, silica, etc. to remove any lignin chloride formed. L. A. COLES.

**Composition for repelling insects.** STANDARD OIL DEVELOPMENT Co., Assees. of D. H. GRANT (B.P. 345,993, 28.12.29. U.S., 29.12.28).—See U.S.P. 1,755,178; B., 1931, 46.

**Apparatus for adjusting and regulating the purifying and softening of water.** N.V. NECKAR WATERREINIGER MAATS., and A. H. M. TROUSSELOT (B.P. 346,134, 18.3.30).

**Apparatus for softening water.** C. P. EISENHAEUER (B.P. 346,287, 4.1.30. U.S., 11.3.29).

**[Pressure filter for] filtration of water.** FILTRATION & WATER SOFTENING PROPRIETARY, LTD. (B.P. 346,376, 28.10.29. Austral., 29.10.28).

**Filtration process. Dialysing liquids. Vaporisation of liquids.**—See I. **Fumigation. Gas-protection appliances.**—See VII

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JUNE 19 and 26, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

Some of the newer uses for silicon carbide. C. McMULLEN (Trans. Amer. Electrochem. Soc., 1931, 59, 199—204).—Tubes of bonded silicon carbide are now being used instead of metal tubes in heat-regenerator systems in view of their higher heat conductivity and better resistance to high temperatures. Other uses referred to include combustion chambers in "Carbo-radiant" furnaces and rotary metallurgical furnaces, boiler-furnace walls, ignition baffles of oil burners, resistors for heating elements and wireless apparatus, lighting arresters, and in the protection of power-transmission lines from high-voltage surges. Some of the more important properties of pure recrystallised carborundum and of bonded 80% SiC are tabulated.

H. J. T. ELLINGHAM.

Recent engineering applications of rubber. J. R. HOOVER and F. L. HAUSHALTER (Ind. Eng. Chem., 1931, 23, 462—469).—The importance of rubber as an engineering material is illustrated by its use for bearings, for absorption of noise and vibration, as a chemically resistant covering or lining for tanks, pipes, valves, etc., for elimination of the ice hazard in aviation, and as an abrasion-resistant surface layer for ball mills and conveyor belts. Reference is made to the successful development and applications of oil-resisting rubber and to the anode process for the manufacture of rubber articles and the rubber-coating of metals.

D. F. TWISS.

Fire extinction.—See II. Measurement of turbidity.—See XVII.

See also A., May, 591, Thermal insulation. Fractionating columns. 592, Hydrometer. Extraction apparatus. 593, Colorimeters, spectrophotometers, and nephelometers.

### PATENTS.

Furnace. R. WALKER (U.S.P. 1,781,616, 11.11.30. Appl., 14.11.28).—A furnace, suitable for burning sawdust etc. liable to be carried over to the heat-absorbing surfaces (e.g., boiler tubes), comprises primary and secondary combustion chambers separated by a depending arch and upstanding bridge; a secondary grate is also provided just beyond the bridge. Materials rising out of the primary combustion zone are deflected downwards by gas currents from ports in the arch; air is also admitted through the bridge. B. M. VENABLES.

Thermal treatment of pulverulent material. F. BARTLING (U.S.P. 1,781,659, 11.11.30. Appl., 14.2.29. Ger., 2.2.28).—Powdered material is subjected to heat treatment, e.g., destructive distillation, while

attached to a moving surface by means of static electrical charges.

B. M. VENABLES.

Processing of furnace dust. W. J. MCGURTY, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,780,833, 4.11.30. Appl., 24.4.28).—The gases from, e.g., a blast furnace are passed through a primary dust collector where the coarser dust is collected in a dry or damp state and are then passed through a wet scrubber. The fluid from the scrubber is thickened and the primary dust added to the thick pulp in a mixer, after which the sludge is dried and sintered by roasting or other means, the sinter being returned to the blast furnace.

B. M. VENABLES.

Heat removal by mercury. B. L. NEWKIRK, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,780,683, 4.11.30. Appl., 4.8.27).—A mercury boiler is constructed so that the heat is removed from the tubes mostly by a mixture of liquid and vapour of mercury having a low proportion (a few %) of liquid by vol. but not by wt., which absorbs heat much better than does mercury vapour. To this end the tubes are made long and of small diameter; they may also be flattened and provided with internal spirals or other means of preventing the mercury mist from settling out until the mixture reaches the desired place of separation, viz., the vapour drum. The tubes are led tangentially into the drum so that the droplets reach the wall by centrifugal force and are caught in a pool of mercury in the bottom, while the vapour leaves axially.

B. M. VENABLES.

Heat exchangers. F. B. DEHN. From YORK HEATING & VENTILATING CORP. (B.P. 346,197, 9.5.30).—An exchanger comprising a number of U-tubes in a casing is described.

B. M. VENABLES.

Tubular heat exchangers. E. F. A. D. BECK (B.P. 345,794, 7.3.30. Belg., 9.3.29).—The tubes, preferably carrying a liquid, of an exchanger are embedded in metallic masses providing a large surface for the outer fluid, usually a gas, and leaving only narrow passages for it, the shape being such that lodgment of dust and resistance to flow are minimised. Ribs may be provided parallel to the flow.

B. M. VENABLES.

Installations for carrying out strongly endothermic reactions. SOC. D'ETUDES ET RÉALISATION DITE ERÉAL, Assees. of E. CONNERADE (B.P. 345,629, 24.12.29. Belg., 27.12.28).—The reaction zone is placed on the axis within a gas producer in which one of the reacting gases is produced; the gas passes upwards through the producer, downwards through passages in the dividing wall, and upwards through the reaction zone. Other, external, condensing and heat-exchanging devices

\* The remainder of this set of Abstracts will appear in next week's issue.



are provided. Possible applications are in the reduction of roasted zinc ores and for the production of nitrogenous compounds.

B. M. VENABLES.

**Refrigeration.** W. H. CARRIER, Assr. to CARRIER ENG. CORP. (U.S.P. 1,781,051, 11.11.30. Appl., 15.10.26).—Methylene chloride, preferably refined so that it is free from fractions of different b.p., is utilised at a pressure not much above 1 atm. (preferably under partial vacuum). It is sprayed so as to form a film over un-submerged evaporating surfaces and the vapour is drawn off, condensed, and re-used together with the un-vaporised liquid.

B. M. VENABLES.

**Centrifugal mills.** E. BARTHELMESS (B.P. 346,011, 1.1.30. Ger., 2.5.29).—A centrifugal cup or other rotor which may be unbalanced is permitted to rotate about its centre of gravity by giving the upper bearing (that nearest the cup) restrained freedom in all horizontal directions, by permitting spherical movement of the lower bearing, and providing a flexible coupling to the motor.

B. M. VENABLES.

**Apparatus for grinding and sifting.** E. BARTHELMESS (B.P. 345,996, 30.12.29. Ger., 11.10.29).—In a machine where the grinding is effected by ascending and rotating currents of air produced by a centrifugal rotor, means are provided for adjusting the annular air exit and hence the size of the finished material.

B. M. VENABLES.

**Multiple rubbing, grinding, and mixing rolling mill for chocolate, soap, paint, etc.** SIMON A.-G. (B.P. 346,166, 9.4.30. Ger., 10.4.29).—The mill comprises a number of rolls in series rotating alternately at high and low speeds, but with a progressive increase in speed in the direction of travel of the material, e.g., the odd numbers may rotate at 144—160 and the even at 72—80 r.p.m.

B. M. VENABLES.

**Dry separation of solid materials.** A. T. MASTERMAN (B.P. 345,997, 30.12.29).—A device of the kind in which heavy particles fall farther away from an air jet than light particles is supplemented by devices intended to effect separation of the middling containing large light and small heavy particles. The falling particles are caught and rendered nearly stationary by a baffle and then allowed to slide down inclined surfaces which may be roughened to any desired degree, the heavy particles acquiring more momentum being collected in side compartments and the lighter in central compartments.

B. M. VENABLES.

**Separation of granular, powdered, and like materials.** W. D. WILLIAMS (B.P. 346,002, 1.1.30).—The apparatus is suitable for estimating the amount of "flour" in such materials as Portland cement by elutriation with air. It comprises a  $\Pi$ -tube with suitable branches for connexion to gauges and a suction device. The limb to which the sample is fed is provided with bulges to maintain the air current turbulent.

B. M. VENABLES.

**Disc feeder and mixer.** L. C. BONNOT, Assr. to BONNOT Co. (U.S.P. 1,781,097, 11.11.30. Appl., 22.7.29).—A number of constituents are delivered from separate hoppers through separate spouts with adjustable outlets on to a single rotating disc which effects the

feeding and from which they drop to a lower disc of larger diameter where they are mixed. On the upper disc a scraper is provided for each constituent, but on the lower a single scraper suffices.

B. M. VENABLES.

**Mixing machine [for dough etc.].** W. F. DEHUFF (U.S.P. 1,781,321, 11.11.30. Appl., 4.5.29).—A variable-speed gear of the expanding-V pulley type is applied to a mixer.

B. M. VENABLES.

**Worm presses for treatment of moist materials.** F. KRUPP GRUSONWERK A.-G. (B.P. 345,820, 29.3.30. Ger., 13.5.29).—A method of restricting, in an adjustable manner, the annular outlet of an archimedean press is described.

B. M. VENABLES.

**Apparatus for treating [laminated] materials under pressure [and while under vacuum].** H. GRIFFITHS (B.P. 345,772, 18.2. and 30.10.30).—Laminated materials such as safety glass are interleaved with flexible chambers in a press which is run into a vacuum chamber. The squeezing is produced by admission to the flexible chambers of a pressure fluid which may, if desired, be hot.

B. M. VENABLES.

**Press for extracting liquids.** H. G. SCHWARZ (U.S.P. 1,781,250, 11.11.30. Appl., 5.4.27).—An archimedean press having perforations for exit of liquid in both the cylindrical casing and the conical hollow shaft of the worm is described. The annular outlet for solids is obstructed more or less by an adjustable tapering collar. Applications cited are the extraction of oils from fish and fish residues, and of juices from various fruits.

B. M. VENABLES.

**Filter presses.** SOC. CHEM. IND. IN BASLE (B.P. 346,191, 1.5.30. Ger., 1.5.29).—A filter press is provided with hot plates in each frame on the line of division of the cake.

B. M. VENABLES.

**Filtering apparatus.** SOC. EN NOM COLLECTIF E. & M. LAMORT FILS (B.P. 345,761, 12.2.30. Fr., 22.2.29).—A filter for large quantities of liquid of the type which is cleaned by reverse flow is described. The filter is in the form of a cylindrical shell which is smaller than the surrounding casing. At opposite ends of a diameter fins, attached to the filter, make fluid-tight joints with the casing; the inlet and dirt-outlet ports are diametrically opposite in the casing and the outlet for filtrate is axial. In normal filtering the fins are placed opposite the middle of the opposing ports, the dirt outlet being closed by a valve; for cleansing purposes the axial outlet is closed, the dirt outlet opened, and the filter rotated so that the fins cause the fluid to enter at one side and leave by the other, carrying the dirt from one half with it.

B. M. VENABLES.

**Filter bed.** A. LENDERINK (U.S.P. 1,780,791, 4.11.30. Appl., 8.3.29).—A form of construction of a bed for a filter, but not for the filter medium, suitable for fluids such as sewage, is described.

B. M. VENABLES.

**Treating the filter cake produced from continuous filters.** B. H. RYLEY (U.S.P. 1,781,652, 11.11.30. Appl., 4.5.28).—Each leaf is jarred by a hammer in order to compact the cake.

B. M. VENABLES.

**Centrifugal machines for separating milk or the like.** ECREMEUSES MELOTTE, SOC. ANON. (B.P.

345,834, 11.4.30. Belg., 11.4.29).—The supply tank and casing for the gearing of a centrifugal separator are cast in one piece with rounded corners, the gearing being placed in one corner of the tank. Means are provided for rapidly removing the casing of the bowl.

B. M. VENABLES.

**Centrifugal separator bowls.** AKTIEB. SEPARATOR (B.P. 345,849, 23.4.30. Swed., 24.4.29. Addn. to B.P. 316,181; B., 1930, 307).—A centrifuge constructed on the principles described in the prior patent is provided with outlets for the products at opposite ends of the bowl, at least one of the outlets being adjustable, *e.g.*, by a removable weir-plate. B. M. VENABLES.

**Evaporators.** G. & J. WEIR, LTD., and J. G. WEIR (B.P. 345,810, 21.3.30).—The spray-separating baffle in the vapour space of an evaporator is provided with a gutter and drainpipe to lead the collected liquid to an innocuous place, and in combination with this is placed, below the baffle, a perforated diaphragm, which is substantially horizontal, but is higher in the middle.

B. M. VENABLES.

**Steam distillation.** E. H. LESLIE and E. M. BAKER (U.S.P. 1,780,977, 11.11.30. Appl., 11.5.23).—The apparatus comprises a fractionating tower with condenser for total or reflux condensation at the top, filling in the tower, means for admitting the original hot liquid (*e.g.*, petroleum oil) to any desired stage, and means for drawing off separate liquid fractions from a number of stages. Below the tower are means for the flash-vaporisation of the residual liquid, which is re-admitted as vapour between two layers of filling, steam being admitted into the lowest pool of all.

B. M. VENABLES.

**Apparatus for separating liquids into constituents of low b.p. and those of high b.p. by distillation and rectification.** A. MESSER (U.S.P. 1,780,563, 4.11.30. Appl., 9.11.25. Ger., 15.11.24).—In a bubbling tower the downflow of liquid is arranged alternately at the centre and circumference of the trays, and the bubbling caps are distributed at a uniform distance apart.

B. M. VENABLES.

**Fractionation apparatus.** W. Z. FRIEND, ASSR. to BALTIMORE GAS ENG. CORP. (U.S.P. 1,780,818, 4.11.30. Appl., 17.9.28).—A fractionating tower is provided with a manhole on the axis of the casing and with corresponding detachable plates in the centre of each tray so that access may be had for cleaning.

B. M. VENABLES.

**Atomisation of liquids.** J. A. REAVELL (B.P. 345,714, 13.1.30).—The liquid is admitted on to a rotary atomising disc by means of an annular passage surrounding the driving shaft and, if desired, supporting the lower bearing for the latter. The disc is corrugated and the feed passage may be Venturi-shaped; the initial inlets to the disc may be tangential to give the liquid a preliminary spin in the direction of travel of the disc.

B. M. VENABLES.

**Liquid and gas contact apparatus.** F. H. WAGNER, ASSR. to BARTLETT HAYWARD Co. (U.S.P. 1,780,255, 4.11.30. Appl., 8.3.28).—The apparatus comprises a tower having a number of perforated diaphragms over which liquid is spread by discs (on a common shaft)

running in apertures in the centre of the diaphragms. The pressure of the upflowing gas should be sufficient to prevent any substantial downflow of liquid through the perforations, annular gutters and siphon pipes being provided for that purpose. B. M. VENABLES.

**[Laboratory] gas-producing apparatus.** G. R. H. FORD (B.P. 346,421, 31.12.29).—For provision of a supply of gas, such as hydrogen sulphide, a generator and a reservoir are mounted at opposite ends of a platform which can be tilted at any desired angle. The principle may be adapted to gas production by electrolysis.

R. H. GRIFFITH.

**Liquefaction of gases.** L. B. STRONG, ASSR. to F. G. CAMPBELL (U.S.P. 1,780,250, 4.11.30. Appl., 27.3.30).—The gas to be liquefied, *e.g.*, air, is compressed, cooled to ordinary temperature, and expanded adiabatically in a heat-insulated engine, *e.g.*, a turbine, the work from which may be used in the original compression. The high pressure is so chosen that the entropy of the gas at high pressure and ordinary temperature is equal to or less than its entropy at the lower pressure after expansion and its b.p. at that pressure, under which conditions the expanded gas will be in the form of saturated vapour. Another gas of lower critical temperature, *e.g.*, hydrogen, is compressed, cooled, and isothermally expanded in a series of turbines or engines alternating with heat-exchanging coils upon which the saturated air is condensed, the latent heat of the air now in the hydrogen being recovered by heat exchange between the in- and out-going streams of hydrogen, which is wholly returned to its compressor. The process may be applied to the separation of helium from natural gas.

B. M. VENABLES.

**[Controlling the flow of gas through] gas-analysis apparatus.** AKTIEBOLAGET CARBA, ASSECS. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 345,665, 14.12.29. Swed., 17.12.28).—The actual analysing apparatus is placed in a shorter by-pass of a longer one, the latter permitting an adequate current of gas to be brought close to the apparatus irrespective of the small quantity actually analysed.

B. M. VENABLES.

**Viscosimeter.** E. M. SYMMES, ASSR. to HERCULES POWDER Co. (U.S.P. 1,780,952, 11.11.30. Appl., 27.9.27).—A viscosimeter of the falling-ball type is adapted to opaque liquids by the use of oscillating radio-valve circuits to detect the fall of the ball. B. M. VENABLES.

**[Wax-resin balls for] determining the sp. gr. of liquids [*e.g.*, battery acid].** BRITANNIA BATTERIES, LTD. (B.P. 345,846, 17.4.30. Ger., 23.4.29).—It is stated that gas bubbles do not adhere to density-indicating floats when they are made of artificial wax and resin. Claim is made for compositions containing equal parts of artificial resin and artificial wax with colouring matter and a variable amount of barium sulphate according to the gravity desired. B. M. VENABLES.

**Friction material for brakes, clutches, etc.** A. E. WHITE. From UNION ASBESTOS & RUBBER Co. (B.P. 345,991, 24.12.29).—The lining comprises an asbestos base with wires or strips of lead or lead alloy embodied in it and a resinous binder. Besides its known function as a lubricant the lead seems to act as

a chemical preventative of the formation of abrasive materials such as slags or carbides ordinarily caused by the friction.

B. M. VENABLES.

**Apparatus for producing artificial fog.** HANSEATISCHE APPARATEBAU-GES. VORM. L. VON BREMEN & CO. M.B.H. (B.P. 345,215, 17.12.29. Ger., 17.12.28).—An apparatus for the production of acid fog by the method described in B.P. 298,980 (B., 1930, 127) is provided with automatic valves for the emission of spray and admission of reagent controlled by corrugated tube diaphragms, similar devices being used instead of stuffing boxes.

B. M. VENABLES.

**Dialysing membranes.**—See V. **Furnaces for sulphate production etc.**—See VII. **Inextensible belting.**—See XV.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Composition of coal. Soluble constituents of coal and their degree of coalification.** C. COCKRAM and R. V. WHEELER (J.C.S., 1931, 854–860).—Samples of coal from the Pittsburgh seam (80.5–89% C) and the Barnsley seam (80.6–84.5% C) have been subjected to solvent analysis (cf. B., 1927, 401). Whilst the Pittsburgh coals exhibit no progressive variation in the proportion of the complete  $\gamma$  fraction, there is a gradual increase in the ratio of the combined  $\gamma_2$  and  $\gamma_3$  fractions (resinic compounds) to the  $\gamma_1$  fraction (free hydrocarbons) with increasing carbon content. Taking the "Festbitumen" and "Ölbitumen" of Fischer, Broche, and Strauch (B., 1925, 233) as corresponding with the combined  $\gamma_2$  and  $\gamma_3$  fractions and the  $\gamma_1$  fraction, respectively, the relative proportions of these in the German coals vary with the carbon content ("rank") in the opposite direction to that observed for the Pittsburgh coals. In the Barnsley coals there appears to be no relationship between their "rank" and their content of free hydrocarbons. The necessity for caution when attempting to compare coals that have been formed under widely different conditions is emphasised.

A. B. MANNING.

**Unit coal studies on some Virginia coals.** F. H. FISH and J. A. ADDLESTONE (Ind. Eng. Chem. [Anal.], 1931, 3, 155–158).—The unit B.Th.U. values for the Virginia coals under investigation are in best agreement when calculated by the Fieldner modification of the Parr formula (Trans. Amer. Inst. Min. Met. Eng., 1930, 78, 597). When, however, a correction is applied for the carbon dioxide, which is present in considerable amount in some cases, the original Parr formula gives the better agreement. For 28 samples taken from the same seam the average deviation from the mean is  $\pm 75$  B.Th.U. by the Parr formula and  $\pm 86$  B.Th.U. by the Fieldner modification. For other coals studied, the average difference between the untreated, float, and sink portions of each coal is 82 B.Th.U. (Parr) and 96 B.Th.U. (Fieldner).

E. S. HEDGES.

**Determination of the hygroscopic properties of coal.** R. VONDRÁČEK and L. MOSENDZ (Coll. Czech. Chem. Comm., 1931, 3, 81–92).—The static and dynamic methods of determining the hygroscopicity of coal are described. By consideration of previous results obtained by the static method with different types of

coal the equation  $A^n - (A - a)^n = nkp$ , where  $A$  is the initial humidity and  $a$  the humidity at vapour pressure  $p$ , and  $n$  and  $k$  are constants characteristic of the type of coal, has been derived. Experiments on the rate of dehydration of lignite at the ordinary temperature over sulphuric acid in stationary and moving air and *in vacuo* are described. The results accord with the theoretical equation  $(H - x)^{-m} - H^{-m} = mkt$ , where  $H$  is the total moisture content,  $x$  the loss during time  $t$ , and  $m$  and  $k$  are constants. The value of  $m$  is independent of the method of desiccation and increases from about 1.1 at 25° to 1.9 at 60°, falling again to 0.75 at 100°;  $k$  increases continuously with rise of temperature and varies with the method of desiccation.

H. F. GILLBE.

**Physics of coal carbonisation.** S. P. BURKE, T. E. W. SCHUMANN, and V. F. PARRY (Fuel, 1931, 10, 148–171).—The temperature distributions in (a) a flat slab, (b) a cylinder, (c) a cylindrical annulus, and (d) a sphere, the outer surfaces of which are suddenly heated to, and maintained at, a fixed higher temperature, have been calculated as functions of the time and the thermal diffusivity of the material. The times required to attain a definite distribution of temperature in two geometrically similar bodies are proportional to the squares of their corresponding linear dimensions. It is deduced theoretically that this "law of squares" still holds even when the thermal constants vary with the temperature, and when thermal reactions involving the liberation or absorption of heat occur in the material, provided that such reactions may be regarded as taking place instantaneously. Experiments on crushed coal and coke in cylindrical retorts of different sizes have shown that the laws of conduction for crushed materials deviate by less than 2–3% from those for solid materials. The thermal diffusivity of crushed coal (approx. 16-mesh) was 0.010 sq. in./min. (15–340°), and of monolithic coke 0.037 sq. in./min. (15–540°). The initial part of the curve showing the variation of the temperature at the centre of a charge of coal with time is modified by the presence of moisture, heat being transferred through the charge by convection of water vapour as well as by conduction. Endothermic reactions probably occur during the decomposition of coal in the plastic range, and both endothermic and exothermic reactions subsequently occur on heating the coke. These reactions alter the shape of the heating curve and thus affect to some degree the time of carbonisation. Despite the effect of convection and the influence of the time element on the chemical reactions occurring in coal, the "law of squares" is found to hold good for the coking process. If  $F$  is the carbonising time (in min.) for a cylindrical retort of unit radius for any particular wall temperature, then the carbonising time for a cylindrical retort of radius  $R$  (in.) is  $FR^2$ , that for a flat retort of semi-width  $R$  is  $2FR^2$ , and that for a cylindrical annular retort is  $2FR^2$ , where  $2R$  is the difference of the outer and inner radii. The coking time of Pittsburgh coal as a function of the retort wall temperature, initial temperature of the coal, and dimensions of the retort has been studied, experiments having been carried out in cylindrical retorts ranging from  $< 2$  in. to  $> 13$  in. in diam. The curve showing the coking time as a function of the

temperature lies between the theoretical curves for coal and coke, respectively, approaching the former asymptotically at the lower temperatures and the latter at the higher temperatures.

A. B. MANNING.

**Process of combustion of powdered coal.** A. GREBEL (Compt. rend., 1931, 192, 567–569).—The ignition of coal etc. in presence of a trace of moisture is due to ignition of hydrogen evolved through the action of heat on the carbon and the moisture. An outline of the subsequent process of combustion follows, and is illustrated by a series of curves based on Fink's results.

C. A. SILBERRAD.

**Behaviour of carbonised fuels in an open fire grate.** G. MILNER, J. H. DYDE, and H. J. HODSMAN (J.S.C.I., 1931, 50, 113–120 r).—The laboratory properties of carbonised fuels have been correlated with their behaviour in an open, vertical-fronted fire grate. The radiation depends jointly on the area radiating,  $A$ , its emissivity,  $e$ , and its temperature,  $T$ , i.e., on the product  $eAT^4$ . The area factor depends on grate design and geometrical configuration of the fuel. Dense compact fuels, tending to pack closely, do not give so high an output of heat as spongy, bulky fuels of irregular form. The emissivity factor is lessened by the presence of flame, resulting either from the combustion of the volatile matter of the fuel or the reduction of carbon dioxide in the upper part of the fire. The latter also lowers the temperature of the fuel bed, thus tending to reduce the output of heat. A similar chilling effect is produced by moisture, particularly in the early stages of lighting. Ash reduces the rate of output of heat, but has only a slight influence on thermal efficiency. Ignition temperatures serve as a useful guide as to ease of lighting. Under the experimental conditions the radiant efficiencies lie between 20% and 37%, but when the fire reaches a steady state about 40% may be emitted. It seems possible to predict these figures approximately from a knowledge of the reactivity and bulk density of a coke. It is concluded that the desirable properties of a solid fuel are sometimes mutually incompatible, and therefore an ideal fuel cannot be realised.

**"Melting" of coal during coke formation.** E. AUDIBERT (Fuel, 1931, 10, 189–190).—A reply to Davies and Wheeler (B., 1931, 428). The question as to whether coal melts completely or only partly during coke formation is regarded as still open.

A. B. MANNING.

**Application of antioxygenic action to fire extinction: negative catalysis of the ignition of coal.** C. DUFRASSE and R. HORCLOIS (Compt. rend., 1931, 192, 564–566).—Addition of 5% of carbon tetrachloride vapour or of 1% of phosphoryl chloride vapour to air used for combustion of charcoal has practically the same effect as total deprivation of air. Several other halogen-containing compounds, diethylamine, and sulphur dioxide act more or less similarly. It is concluded that such substances act catalytically.

C. A. SILBERRAD.

**Activity of incrustated coke and the production of malleable pig iron low in carbon.** E. PRIVOVARSKY [with F. KRAMER] (Giesserei, 1930, 17, 1149–1152; Chem. Zentr., 1931, i, 843).—The reactivity of coke towards carbon dioxide at 900–1100° is increased by

treatment with milk of lime. The production in the cupola furnace of iron containing less than 1.6% C is described.

A. A. ELDRIDGE.

**Sodium thiocyanate as a medium for drying [coal] gas.** K. KELLER and H. NORDT (Ber. Ges. Kohlentechn., 1931, 3, 460–464).—The procedure of gas-drying with sodium thiocyanate is similar to that of methods using other hygroscopic salts, but the material is one which can be manufactured at a coke-oven plant. If gas saturated with water vapour at 20° is washed at this temperature with a saturated solution containing 508 g. NaCNS per litre, the dewpoint falls to 7.3°. If gas-washing is carried on at 25°, using the solution saturated at this temperature, the dewpoint of the dried gas is 8.4°. A series of determinations of dewpoints, using more dilute solutions, is given.

C. IRWIN.

**Utility of solutions of nickel and copper salts for the removal of hydrogen cyanide from coke-oven gas.** W. GLUUD and W. RIESE (Ber. Ges. Kohlentechn., 1931, 3, 437–451).—It was desired to ascertain whether hydrogen cyanide could be separated from coke-oven gas efficiently as complex copper or nickel cyanides and the cyanide conveniently recovered. The reaction is complicated by the simultaneous formation of thiocyanate, depending on the oxygen content of the gas and on other factors. The principal reactions of crude coke-oven gas with a solution of a nickel salt are the initial formation of nickel sulphide followed by  $\text{NiS} + 2\text{NH}_4\text{CN} = (\text{NH}_4)_2\text{S} + \text{Ni}(\text{CN})_2$ ;  $\text{Ni}(\text{CN})_2 + 2(\text{NH}_4)\text{CN} = (\text{NH}_4)_2[\text{Ni}(\text{CN})_4]$ . This last salt is not decomposed by carbon dioxide or hydrogen sulphide. The simultaneous formation of thiocyanate is greatly promoted by ammonia. It is therefore necessary to use gas free from ammonia and to replace this by another alkali. The soda or potash double salts can be brought up to concentrations suitable for crystallisation without ill effect on the cyanide removal. There are several methods of decomposing the double salts, but none appears likely to be economical, and this is the principal objection to the process. A double copper salt  $\text{K}_2[\text{Cu}(\text{CN})_4]$  can be prepared in a similar way, but is not so stable to carbon dioxide as the nickel salt. With increasing concentration the washing efficiency on coke-oven gas deteriorates and its use does not appear practical. The recovery of hydrogen cyanide from the copper salt offers the same difficulties as with nickel.

C. IRWIN.

**Production of water-gas in horizontal chamber ovens.** A. STEDING (Gas- u. Wasserfach, 1931, 74, 357–361; cf. Gill, B., 1930, 848).—Three series of experiments are described in which steam was passed through the charge in a suitably modified Otto coke-oven setting. The steam was introduced (a) through the floor of the oven, (b) through the walls, and (c) through tubes projecting into the charge from above, respectively. With each of these arrangements a good-quality water-gas could be produced. Moreover, the introduction of steam lowered the nitrogen content of the gas by diminishing the in-leakage of flue gases. The temperature of the coke when discharged was considerably lower with steaming than without.

A. B. MANNING.

**Kinetics of the thermal decomposition of methane.** T. S. WHEELER (Fuel, 1931, 10, 175—181. Cf. Bone and Coward, B., 1908, 886; Wheeler and Wood, B., 1929, 88; 1931, 193; Holliday and Exell, A., 1929, 773).—The available experimental data have been critically examined. They indicate that the decomposition of methane in contact with silica, porcelain, and chrome iron is heterogeneous, and is retarded by the hydrogen produced. The experimental results can be reproduced by an equation which indicates that the retardation is a function of both the hydrogen and the methane pressures.

A. B. MANNING.

**Nature and properties of coal tar.** E. V. EVANS and H. PICKARD (S. Metropolitan Gas Co., Chem. Dept., 1931, 41 pp.).—The consistency of a tar (seconds Hutchinson or time of flow in the Redwood No. 2 viscosimeter) is related to the temperature ( $^{\circ}\text{F.}$ ) by the formula  $CT^n = a$ , where  $n$  and  $a$  are constants. The addition of naphthalene to a "synthetic" tar markedly lowered the consistency, the addition of 5%, e.g., changing the values of  $n$  and  $a$  from 5.7 and  $712 \times 10^{11}$  to 5.1 and  $213 \times 10^{10}$ , respectively. A similar lowering of the consistency was produced by the addition of naphthylamine, benzene, or aniline, whereas the effect of adding naphthol or phenol was much less marked. By plotting log temperature ( $^{\circ}\text{F.}$ ) against log consistency for these mixtures, and for mixtures of pitch with the tar, a series of straight lines was obtained which when produced passed approximately through a common point (focus). A viscosimeter suitable for determining the consistency of pitch at raised temperatures has been designed, based on the measurement of the time of flow of the viscous liquid down an inclined cylinder. The logarithmic consistency curves of mixtures of tar and pitch obtained by using this instrument again took the form of a series of straight lines emanating from a common focus. Asphaltic bitumens differed from coal-tar products in the slope of the corresponding lines, the temperature coefficient of consistency being less for bitumens than for tar products, and in the position of the focus. Addition of a filler, e.g., lamp-black, to the tar or pitch increased the consistency, the new log consistency curve lying parallel to the original and passing through a different focus. Removal of the "free carbon" insoluble in pyridine had a negligible effect on the consistency of a pitch, whereas removal of the "free carbon" soluble in pyridine but insoluble in benzene produced a pronounced lowering in the consistency. Finely-divided coal has been shown to be a suitable filler for modifying the consistency of a tar (cf. B.P. 316,897; B., 1929, 803). The consistency of pitch at ordinary temperatures has been measured by means of a modified penetrometer, in which the needle is replaced by a rod having a rounded end. Within practical limits of accuracy, pitch behaved as a true fluid at these temperatures, the velocity-load curves being straight lines passing through the origin. Asphaltic bitumen, on the other hand, exhibited anomalous consistency effects in its velocity-load relationship. Dispersions of coal in tar exhibited corresponding anomalous consistency effects, and in this respect, therefore, the physical properties of the asphaltic bitumen have been reproduced in a tar.

A. B. MANNING.

**Low-temperature tars.** G. T. MORGAN (Fuel, 1931, 10, 183—189; cf. Morgan, Pratt, and Ross, B., 1929, 156).—The methods and results of examining some low-temperature tars are described. The presence of the following substances in the fractions and/or the aqueous liquor of a "primary" tar produced by the carbonisation of Dalton Main coal in a Pehrson retort at  $450^{\circ}$  has been demonstrated: benzene, toluene, *m*- and *p*-xylenes, naphthalene, 2-methylnaphthalene, phenol, *o*- and *p*-cresols, pyrocatechol, resorcinol, and quinol. No pyridine was found. The "primary" tar had a lower sp. gr. (1.008) than that of a tar produced from the same coal at  $625^{\circ}$  (1.02), and contained more neutral oil, less bases, less phenols, and less paraffin wax than the latter.

A. B. MANNING.

**Phenol content of tars and oils derived from coal.** J. L. WILTSHIRE (J.S.C.I., 1931, 50, 125—128 r).—The methods available for the determination of phenol in tars are discussed, and one of them is shown to give satisfactory results without undue elaboration. This method is applied to a series of products derived from coal: low-temperature tar from Fuel Research Board vertical retorts, 1.0% of phenol (Warwick coal) and 1.5% of phenol (Shafton coal); hydrogenated Shafton low-temperature tar, 4.9% of phenol; liquid product from hydrogenation of Shafton coal, 2.6% of phenol.

**Ageing phenomena in creosote oils.** W. GRUND-MANN (Chem.-Ztg., 1931, 55, 267).—The viscosities of creosote oils and of their alcoholic solutions increase on keeping, the change being accelerated by repeated cooling at  $-30^{\circ}$  to  $-40^{\circ}$ . Similar changes occur also in their electrical conductivities, so that creosotes of different age and from different distillations rarely have identical physical properties.

F. R. ENNOS.

**Chemical aspect of [oil-well] drilling muds.** A. DUCKHAM (J. Inst. Petroleum Tech., 1931, 17, 153—182).—The mud, used as a lubricant in rotary drilling and as a vehicle for removing rock cuttings, may either be prepared from clays found near the well or imported and may require "doctoring" to give it the required weight, viscosity, and colloidity. A heavy mud may be necessary to prevent "blow outs," and for this purpose barytes or hematite may be added, weights up to 120 lb./cub. ft. being occasionally used, although 80 lb./cub. ft. is the more usual quantity. A chart which enables the amount of barytes required to be added to a given mud to produce the desired weight is given. Viscosity is determined in the Stormer viscosimeter by rotating a cylinder in the mud. No ready means of determining colloidity has been elaborated. Muds are freed from cuttings by straining or by such apparatus as the Dorr classifier. Colloidity is controlled by the addition of such materials as bentonite, "aquagel," or soda. The addition of soda gives a suitable viscosity to heavy muds, but excess causes "ropiness" and separation. Before adding soda the alkalinity of the mud and its water content should be determined. The ingress of salt water may spoil a mud. Mud which has become "gassed" may sometimes be freed from gas by pumping it against a plate.

T. A. SMITH.

**Desulphurisation of shale oils.** T. KOERN (Tehn. Ajakiri, 1930, 9, 166—168; Chem. Zentr., 1931, i, 1046).—When passed over a heated catalyst (bog-iron ore, or various metallic oxides deposited on carriers as nitrates and subsequently reduced with hydrogen or water-gas) the hydrocarbon (1.06% S) gave a distillate of which the first fractions were low in sulphur. The catalyst was rapidly inactivated and regeneration was difficult. When the oil is heated under pressure with hydrogen in presence of the powdered catalyst the sulphur content may be reduced to 0.06%. Regeneration is easily effected.

A. A. ELDRIDGE.

**Hydrogenation and desulphurisation of Estonian shale oils.** J. HÜSSE (Tehn. Ajakiri, 1930, 9, 162—165; Chem. Zentr., 1931, i, 1046).—Desulphurisation is effected in the liquid phase by heating under pressure with hydrogen in presence of a catalyst (bog-iron ore, minette, or nickel-chromium). The oil so treated gives lower values for density, viscosity, phenol content, and solubility in sulphuric acid.

A. A. ELDRIDGE.

**Reactions of olefines with sulphuric acid.** W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petroleum Tech., 1931, 17, 185—187).—The reaction of a number of olefines in "aromatic-free" heavy petroleum spirit (1 pt. of olefine to 9 pts. of spirit) with sulphuric acid of various concentrations (81—99.5%) has been determined, the bromine values (Hanus) before and after treatment being determined. The olefines become resistant to sulphuric acid as their mol. wt. increases, thus agreeing with the behaviour of these substances towards permanganate as stated by Howes and Nash (B., 1930, 596). The difference in behaviour, however, is marked only at the lower concentrations (up to 90%) of acid. At higher concentrations the bromine values are reduced to very low figures with all the olefines examined.

T. A. SMITH.

**Effect of light on determination of ethylene.** J. L. OBERSEIDER and J. H. BOYD, JUN. (Ind. Eng. Chem. [Anal.], 1931, 3, 123).—Inaccuracies occur in the determination of ethylene in gaseous mixtures of paraffin and olefine hydrocarbons separated from cracked oils, when the absorption in bromine water is carried out in daylight or artificial light. Correct results are obtained when a blackened absorption pipette is used. E. S. HEDGES.

**Removal of hydrogen sulphide and hydrogen cyanide from benzol.** K. KELLER (Ber. Ges. Kohl-entech., 1931, 3, 429—436).—The claim in G.P. 76,348, that benzol used for the dephenolisation of ammoniacal liquor and containing 1% of phenol was quickly freed from hydrogen sulphide by keeping even in the dark and in absence of air, was investigated. It was found that the presence of ammonia rather than that of phenol is the most likely explanation. Benzol either pure or crude is quickly freed from hydrogen sulphide by air treatment in presence of ammonia and moisture, the sulphide being oxidised to thiosulphate. At the same time hydrogen cyanide is also removed. Such a treatment could usefully be given to benzol intended for phenol extraction before use, in a closed washer with agitation, and troublesome corrosion thereby be avoided. Crude benzol containing 1.5 g.  $H_2S$  and 0.3 g.  $HCN$  per

litre will require 35 litres of 5% ammonia solution per in.<sup>3</sup> The ammonia can be recovered. Experimental work is reported in detail.

C. IRWIN.

**Silicon carbide. Applications of rubber.**—See I. Ammonium sulphate crystals. Conversion of cyanide into ferricyanide.—See VII. Tar oils for timber.—See IX. Fruit tree carbolineums.—See XVI.

See also A., May, 574, Combustion of pulverised fuel. 576, Reaction  $H_2 + CO_2 \rightarrow CO + H_2O$ . 630, Pyridine bases from tar. 638, Detection of carbon disulphide.

## PATENTS.

**Treatment of [anthracite] coal.** E. V. COLLINS, JUN., ASSR. to DELAWARE, LACKAWANNA, & WESTERN COAL Co. (U.S.P. 1,781,102—3, 11.11.30. Appl., 14.2.30).—(A) The appearance of anthracite which deteriorates on storage is restored by treatment with an aqueous solution containing 20% of ammonium persulphate; this may be applied by spraying or by agitating the coal in the liquid. (B) The same result is obtained by washing anthracite with a solution containing 1.5% of titanium trichloride in 10% hydrochloric acid.

R. H. GRIFFITH.

**Heating of low-temperature carbonisation retorts.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,533, 28.2.30).—A plant for the low-temperature distillation of coal, shale, etc. is built into a boiler setting so that heating gases pass first over one part of the boiler, then over the surface of the carbonisation retort, and finally over another portion of the boiler. The position of the retort is chosen so that it is heated to any desired temperature. High thermal efficiency is claimed.

R. H. GRIFFITH.

**Treatment of coal and other carbonaceous material.** A. E. WHITE. From THERMOLIZED COAL CORP. (B.P. 346,383, 7.12.29).—Crushed coal, or similar carbonaceous material, is treated with a stream of waste gas, and then subjected to low-temperature carbonisation. The resulting semi-coke is cooled in an inert atmosphere and finely pulverised so as to be suitable for furnace firing. The powdered material thus obtained is preferable to coal dust in that it is less liable to spontaneous ignition and can be burned with a shorter and more uniform flame. [Stat. ref.]

R. H. GRIFFITH.

**Distillation of coals.** W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,781,614, 11.11.30. Appl., 21.12.25).—Carbonisation of finely-divided coal is effected by the combustion of part of the material. The process is carried out in a stream of air which moves at such a speed that a selected proportion of the carbonised particles is carried away together with gases and vapours which are simultaneously produced. The calorific value of the gas-solid mixture is appreciably higher than that of the gas alone, and provides a useful fuel from coals very low in volatile matter.

R. H. GRIFFITH.

**Production of coke.** A. H. WHITE, ASSR. to REGENTS OF THE UNIV. OF MICHIGAN (U.S.P. 1,782,556, 25.11.30. Appl., 26.10.25).—Broken coal is allowed to fall freely

through an empty vertical retort, and is carbonised by heat radiated from the walls, which are at 700° or above. The time of treatment is very short, but the coke obtained has appreciably less volatile matter than the original coal, and can be pulverised to give an excellent dust fuel. The properties of the coke depend on the size of the raw material, the length of the retort, and the temperature to which it is heated; particles too large to pass 12-mesh are not suitable for treatment. Steam or hydrogen may be added to the retort, and the gas produced is of very uniform quality.

R. H. GRIFFITH.

**Improving explosive mixtures for internal-combustion engines etc.** A. DUCLOUX (U.S.P. 1,777,554, 7.10.30. Appl., 3.5.27. Fr., 7.5.26).—A carburetted fuel is passed through a confined space containing a heated mixture of approx. 10% of a mineral containing thorium, a relatively small percentage of an oxide of a radioactive metal, and a relatively large percentage of an inert substance. The heating may be done conveniently by the exhaust gases. A preferred mixture consists of monazite or thorite 10%, thoria 2%, asbestos 40%, and talc or chlorite (which may include powdered iron, nickel, or manganese) 48%.

H. S. GARLICK.

**Gas producer.** H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,781,767, 18.11.30. Appl., 10.12.23).—In a small gas producer (< 1 ft. in diam.), special precautions are introduced to prevent heat losses from the relatively large surface available. Moist air is drawn into the system in such a way that it passes three times over cylindrical jackets which surround the fuel bed. The grate can be reached only by such indirect passage, so that radiant heat lost from the generator is largely returned to the apparatus.

R. H. GRIFFITH.

**Gas producer.** A. K. BRADLEY, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,782,677, 25.11.30. Appl., 7.6.28).—The shell of a gas producer is mounted to rotate at a constant speed, the vessel being closed by a fixed water-sealed cover and by a water-sealed grate which rotates independently of the producer. The speed of the lower part is variable, and within it is an ash plough which can either be fixed so as to move with the shell, or be carried round solely by the weight of ashes and fuel pressing on it.

R. H. GRIFFITH.

**Generation of combustible gas and coke.** H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,781,766, 18.11.30. Appl., 9.9.22).—A vertical vessel for the carbonisation of coal narrows rapidly below the coal inlet, and then widens to a grate, the centre of which rotates. Below the fire bars is a gastight chamber (where ash and clinker collect) provided with a steam inlet to a hollow standard carrying the rotary section. At the narrowest portion of the retort, and also towards the base, are numerous openings from which gas can pass to, or air be introduced from, a heat exchanger. Producer gas, water-gas, and low-temperature coal gas can be obtained by different methods of operation, and the required mixture is collected from a common top offtake.

R. H. GRIFFITH.

**Water-gas generators.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 345,411, 3.2.30).—Air for

the "blow" period is supplied by tuyères in different amounts and at different levels to a deep bed of fuel contained in the generator, so that the maximum air supply is at the point nearest to that of the exit of the "blow" gases (which is at the bottom of the generator). During the "run" period steam is passed in at the bottom, the resulting water-gas being passed out at the top. Regenerators, to recover the heat of the "blow" gases, for preheating the steam are supplied.

C. B. MARSON.

**Use of heavy oil in the manufacture of carburetted water-gas.** HUMPHREYS & GLASGOW, LTD., Asscs. of J. A. PERRY (B.P. 345,577, 6.6.30).—A water-gas set comprises a generator, a carburetter unobstructed by heat-absorbing material such as chequer brick, a superheater, and a wash-box. The fuel bed in the generator is blasted with air, the resulting "blow" gases passing to the carburetter where, after being burned with secondary air, thereby storing heat in the walls, they are passed through the superheater to the stack. At the same time a shallow fuel bed in the carburetter is blasted with air, the blow gases also being passed through the superheater to the stack. When sufficient heat has been stored in the generator fuel bed, the carburetter, and the superheater, the air blast is terminated and steam is admitted either to the base or top of the regenerator, the resulting water-gas passing to the top of the carburetter, where it is carburetted by heavy oil, also admitted at the top. The oil is vaporised by the heat from the wall and from the fuel bed. The coke from the oil vaporised above the fuel bed is deposited thereon by the downward passage of the water-gas through the carburetter. From the carburetter the water-gas and oil vapour pass to the superheater, where further cracking takes place, and then through the wash-box to storage.

C. B. MARSON.

**Production of gas from garbage.** W. C. RATH (U.S.P. 1,777,449, 7.10.30. Appl., 19.5.23).—The waste materials are heated in a retort to about 400°, and the moisture and other volatile products driven off are passed through a bed of incandescent carbon, whereby an enriched water-gas is obtained. After cooling and scrubbing, a gas of high calorific value and free from sulphur is obtained; the residue in the retort is suitable as fertiliser.

C. JEPSON.

**Removal of sulphur from industrial gases by a cyclic process with simultaneous extraction of pure sulphur.** S. HUNYADY and K. KOLLER (B.P. 345,594, 2.7.30).—The gases to be purified are brought into intimate contact with an aqueous suspension of finely-divided manganic oxide. The sulphur-manganese sulphide mixture is collected on a filter and the manganese sulphide converted into manganic hydroxide and sulphur by the action of atmospheric oxygen. The manganic hydroxide-sulphur mixture is treated with dry air, after which the sulphur is removed by a solvent such as trichloroethylene; the residual manganic oxide is employed for further gas purification.

C. B. MARSON.

**Removing ammonia and hydrogen sulphide from gases.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 346,452, 13.1.30).—The process for removal by



means of a solution of a polythionate is carried out in apparatus constructed of aluminium or an aluminium alloy. If other metals are employed they must be coated with aluminium. R. H. GRIFFITH.

**Tar separator.** G. W. WATTS, ASSR. to STANDARD OIL CO. OF INDIANA (U.S.P. 1,781,618, 11.11.30. Appl., 24.6.25).—A centrifugal separator, for treating tars containing water, is constructed so that the material is fed tangentially to the top of an annular space between two vertical, cylindrical shells. The process is operated under reduced pressure, and the liquids draining from the inner and outer walls are separately collected.

R. H. GRIFFITH.

**Refining of tars.** COMP. TECHNIQUE DES PÉTROLES (B.P. 346,415, 11.12.29. U.S., 11.12.28).—The higher-boiling fractions of low-temperature tars are worked up, particularly for the recovery of lubricating oils and of waxes, by non-destructive distillation with steam or under reduced pressure. A pretreatment to remove acids and bases is advantageous, and crystalline waxes are separated from the distillates by filter-pressing, preferably after dilution with a low-boiling oil. Higher-melting amorphous waxes are subsequently obtained by dilution and chilling, and the residue is divided into fractions of varying viscosity. Final purification may be carried out by acid and alkali washing, and by further distillation.

R. H. GRIFFITH.

**Manufacture of asphaltic material.** G. ABSON, ASSR. to CHICAGO PAVING LABORATORY, INC. (U.S.P. 1,782,186, 18.11.30. Appl., 25.10.29).—The preparation of asphalt by air-blowing of petroleum still residues is greatly accelerated by the presence of a small quantity of a metallic salt which is not volatile at the temperature of operation and does not cause cracking of the material. The product has a higher penetration number than that obtained under the same conditions without the addition of the inorganic compound.

R. H. GRIFFITH.

**Oil still.** W. A. JONES, ASSR. to BABCOCK & WILCOX Co. (U.S.P. 1,776,437, 23.9.30. Appl., 23.5.25).—The still consists of longitudinal drums connected at either end to lower transverse drums, these, in turn, being joined by a steeply inclined bank of tubes which is traversed by the furnace gases. Oil circulates along the longitudinal drums, through a pipe of wide cross-section, to the lower of the transverse drums. It then passes by means of the heated bank of tubes to the upper transverse drum and returns to the longitudinal drum. The last-named drum is connected by means of curved pipes (to permit of differential expansion) to an upper longitudinal drum where vapours can be disengaged. One end of the apparatus is fixed, whilst the other is free to permit of expansion. A number of such stills may be arranged in series.

T. A. SMITH.

**Apparatus for distilling liquid hydrocarbons.** N. E. MERRILL (U.S.P. 1,778,269, 14.10.30. Appl., 30.1.26).—Oil is led through a preheater into a heating chamber mounted in a furnace and consisting of a conical wall closed at the bottom with a removable plate directly exposed to the heat. This heating chamber forms the bottom section of a vertical shell still and communicates with a tube up which the vapours and liquid pass. The vapours are led off from the top of the shell, while

the liquid flows from the top of the tube and downwardly over superimposed baffle plates between the inner wall of the shell and the tube, the residue being drawn off from the bottom. H. S. GARLICK.

**Distillation of hydrocarbon oils.** W. II. BAHKE, ASSR. to STANDARD OIL CO. (U.S.P. 1,778,445, 14.10.30. Appl., 9.8.28).—Oil is passed through a heating coil wherein it is superheated to a predetermined temperature. The inlet of the coil is provided with a pipe through which a regulated amount of steam is admitted. The outlet of the coil leads into an intermediate point of a fractionating column provided above and below the point of entry with suitable fractionating devices. A perforated steam coil enters the lower end of the column where is situated an outlet for the withdrawal of the heavy stripped oil. From its upper end a vapour outlet leads to a partial condenser followed by a water condenser.

H. S. GARLICK.

**Apparatus for cracking oil.** G. EGLOFF, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,766,986, 24.6.30. Appl., 23.5.21. Renewed 17.12.29).—Oil is passed under pressure through a heating coil in a furnace, into an enlarged vapour chamber provided with a vertical partition which forms a relatively small compartment at one side. Vapours leave the chamber and pass up through the bottom of a dephlegmator and condensing system. Reflux condensate is returned to the small compartment and thence to the heating coil. The residue is continually drawn off from the vapour chamber to maintain its level below the upper portion of the partition, thereby maintaining the reflux condensate separate from the residuum, but heated prior to its introduction into the heating coil. H. S. GARLICK.

**Distillation of mineral oil under high vacuum.** A. E. FEW, JUN., ASSR. to SUN OIL CO. (U.S.P. 1,778,565, 14.10.30. Appl., 27.2.26).—A pump with throttling device, for preventing the admission of air while oil is supplied to a still operating under high vacuum, is described.

H. S. GARLICK.

**Distillate petroleum liquid.** J. B. RATHER and L. C. BEARD, JUN., ASSRS. to STANDARD OIL CO. OF NEW YORK (U.S.P. 1,776,598, 23.9.30. Appl., 26.11.26).—The development of colour, odour, or gum in light petroleum distillates, *e.g.*, kerosene and gasoline, is prevented by passing a finely-divided stream thereof downwardly through a container, filled with baffle pieces, up which is passed a copious stream of inert gas, thereby removing the air content of the distillate, which is drawn off from the bottom of the container and stored in hermetically sealed vessels. H. S. GARLICK.

**Treatment of hydrocarbons.** L. B. CHERRY, ASSR. to C. & C. DEVELOPING CO. (U.S.P. 1,779,356, 21.10.30. Appl., 10.3.22).—Cracking stock is introduced into a low-pressure, furnace-heated, horizontal still through perforations in a coil arranged therein. Vapours pass from the still through one or more vertical reaction tubes where they are subjected to the action of high-frequency oscillatory discharges sufficient to maintain a violet glow in each tube, which in addition is heated by an individual suitable furnace. The vapour outlet from the cracking tubes delivers the hot gaseous products to a fractionating condenser from which the condensate is returned to the

still by gravity, and the uncondensed vapours pass on to a final condenser. Suitable pressure-equalising means are provided for the apparatus. H. S. GARLICK.

**Treatment of hydrocarbon oils.** W. S. HADAWAY, JUN., Assr. to TEXAS Co. (U.S.P. 1,776,023, 16.9.30. Appl., 16.5.27).—In a cracking process, the bulk of the heat necessary to raise the oil to cracking temperature is supplied during passage through a furnace-heated high-pressure coil, and a further relatively small amount of heat is supplied by passing the hot oil through an electrically-heated tube in which the electric heat input is regulated to maintain a predetermined outlet temperature. H. S. GARLICK.

**Apparatus for heat-treating hydrocarbon oils.** H. C. WADE, Assr. to HYDROGENATING PROCESS CORP. (U.S.P. 1,777,708, 7.10.30. Appl., 2.5.28).—Economy of fuel and increased capacity of heat-treating apparatus are obtained by providing a heating kiln of normal dimensions with several series of coils, the coils of each series lying contiguously and having the same axis and diameter. One series of coils embraces a second series so that both have substantially the same axis, the lineal capacity of both being substantially the same. Each coil has a discharge outlet at its base. H. S. GARLICK.

**Decomposition of relatively high-b.p. oils into relatively low-b.p. oils.** A. L. STROUT, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,776,985, 30.9.30. Appl., 25.4.27).—Oil is heated to cracking temperature in a tube furnace and passed to a reaction and vaporising chamber. The vapours from this chamber are taken to a dephlegmator and the residue passes to a further vaporising chamber in which the pressure is reduced and part of the oil is vaporised. These vapours are scrubbed with the incoming stock, which is then used as refluxing material in the dephlegmator before passing to the cracking coil. T. A. SMITH.

**Neutralisation of washed light oil.** J. J. LAWTON, Assr. to SEMET-SOLVAY Co. (U.S.P. 1,779,944, 28.10.30. Appl., 31.12.27).—Crude light oil from the destructive distillation of coal is washed with sulphuric acid and the resultant sludge separated. The washed oil is treated with ammonia and subjected to steam distillation in the presence of the neutralising liquid to separate low-boiling oils, thereby breaking any emulsions and preventing decomposition and volatilisation of any sulphur compounds present. H. S. GARLICK.

**Manufacture of hydrocarbons of high b.p., in particular viscous hydrocarbons from hydrocarbons of low b.p.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,334, 18.12.29).—Low-b.p. hydrocarbons are condensed by heat treatment in the presence of a previously prepared double compound of ethylene and aluminium chloride dissolved in a hydrocarbon solvent, while passing gaseous olefines or mixtures containing them into the reaction mixture. The double compound may be made by passing, at 40–60°, ethylene alone or mixed with inert gases into a solvent, free from unsaturated compounds (e.g., paraffin oil), in which the aluminium chloride is suspended. H. S. GARLICK.

**Manufacture of conversion products of higher paraffin hydrocarbons [lubricants].** A. CARPMAEL.

From I. G. FARBENIND. A.-G. (B.P. 343,948, 11.9.29).—Di- and poly-halogenated paraffins above  $C_8$  are heated (e.g., at 190–220°) with aqueous caustic alkalis or alkali carbonates (excluding hydrogen carbonates) or alkaline earths, preferably under pressure, to give viscous light-coloured oils, which are probably unsaturated monohydric alcohols and may still contain halogen. C. HOLLINS.

**Reclamation of lubricating oils.** L. D. GRISBAUM, Assr. to RAILWAY SERVICE & SUPPLY CORP. (U.S.P. 1,777,722, 7.10.30. Appl., 30.4.27).—The used oil is mixed with a chemical treating solution (e.g., caustic soda or potash) and heated in a sealed container to approx. 115°. The developed pressure is further increased by a superimposed air pressure sufficient to prevent ebullition. Heating is stopped and the pressure maintained during cooling. H. S. GARLICK.

**[Lubricating or insulating] oil purification process.** F. M. CLARK and A. T. HARDING, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,776,550, 23.9.30. Appl., 18.12.29).—Unsaturated substances are extracted in the preparation or recovery of such oils, by means of a polyhydric alcohol (glycerol or ethylene glycol); after separation the oil is distilled to remove traces of alcohol. T. A. SMITH.

**Purification of [insulating] oils.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of F. M. CLARK (B.P. 345,708, 9.1.30. U.S., 10.1.29).—Glycerin or other equivalent polyhydric alcohol is maintained in contact with the oil, but out of contact with the insulating and electrical members of transformers, switches, etc. J. S. G. THOMAS.

**Oil-purifying process.** L. D. JONES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,778,831, 21.10.30. Appl., 29.4.24).—Oil is continuously withdrawn from a steam turbine or similar circulating system and treated with cooled water to bring the temperature of the resulting mixture to that at which precipitation of sludge and impurities soluble in hot oil occurs. The water and impurities are separated from the oil by centrifugal means, and then the oil is heated and any traces of water remaining are removed by a further centrifugal treatment. H. S. GARLICK.

**Refining of hydrocarbons.** N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of R. I. LEWIS (B.P. 345,596, 7.7.30. U.S., 15.7.29).—Pressure distillate containing sulphur is treated first with dilute acid, e.g., sulphuric acid ( $d$  1.23), at atmospheric temperature, and thereafter with acid of  $d$  1.84 at a temperature substantially below atmospheric. If desirable, a further treatment with acid ( $d$  1.84) at a raised temperature may be given. H. S. GARLICK.

**Purification of hydrocarbons.** W. H. LOW, Assr. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,777,619, 7.10.30. Appl., 6.9.27).—A substantially dry cracked naphtha or gasoline stock containing mercaptans or other sulphur compounds is rendered "sweet" to the "doctor test" by filtration through a filter-bed containing sufficient comminuted dry lead sulphide. The lead sulphide may be intermittently revived by treatment with a solution of an alkaline sulphide. H. S. GARLICK.

**Purification of hydrocarbons.** M. MERCURIO, Assee. of J. F. A. BRUZAC (B.P. 346,152—3, 2.4.30. Fr., [A] 30.10.29, [B] 22.6.29).—(A) Crude hydrocarbons, containing sulphur compounds which are not separable by distillation, are purified by treatment with oxygen or ozone, preferably at ordinary temperatures, followed by washing with a solution of alkali. (B) Distillates obtained from heavy hydrocarbons by heating them in contact with inert surfaces at temperatures below 500° are purified as described above. [Stat. refs.]

R. H. GRIFFITH.

**Refined viscous hydrocarbon oil.** T. H. ROGERS, Assr. to STANDARD OIL Co. (U.S.P. 1,774,845, 2.9.30. Appl., 19.8.26).—Acid formation and increase in emulsibility in use of highly refined viscous hydrocarbon oils is prevented by adding thereto up to 0.1% of an aromatic compound in which an amino- or hydroxyl group is attached to the ring. Suitable additions are: diphenylamine 0.4—0.1%, gallic acid 0.002%, dibutyl-resorcinol 0.05%.

II. S. GARLICK.

**Testing the purity of solid or liquid hydrocarbons.** A. ESAU (B.P. 345,464, 5.3.30. Ger., 13.3.29).—The material to be tested is subjected to the action of short electric waves of frequency of the order of  $10^8$  Hertz and the resultant changes of temperature produced are measured.

H. S. GARLICK.

**[Anti-knock] motor fuels.** H. D. ELKINGTON. From BENZOL-VERBAND GES.M.B.H. (B.P. 345,587, 20.6.30).—The mixture of aromatic amines obtained by the direct nitration and reduction of crude hydrocarbon mixtures containing benzene and its homologues, obtained in the dry distillation of coal, is added to a benzene motor fuel.

H. S. GARLICK.

**Absorbent oil treater.** J. L. LOONEY (U.S.P. 1,777,005, 30.9.30. Appl., 13.8.28).—An apparatus for the chemical treatment of oils to remove sulphur and sludge consists of a horizontal cylinder fitted with intake and offtake manifolds. The former delivers into a number of pipes which convey oil to the lower part of the cylinder. The offtake is connected to the upper part of the cylinder. Openings for running off sludge are fitted at the oil-chemical interface. T. A. SMITH.

**Burners for pulverulent materials.** H. NIELSEN and B. LAING (B.P. 346,826, 16.1.30).

**Quenching and conveying apparatus for coke or charred substances.** SOUTH METROPOLITAN GAS Co., and C. C. CARPENTER (B.P. 347,424, 25.3.30).

**Means for controlling or operating water-gas apparatus.** A. BREISIG (B.P. 346,711, 15.1.30. Austr., 15.1.29).

**Furnace. Thermal treatment of material. Steam distillation.**—See I. Antioxidants [for oils].—See III. Wax-impregnated fibres.—See V. Water-proof fibres. Mine-ventilating tubing.—See VI. Hydrogen.—See VII. Road coverings. Paving compositions.—See IX.

### III.—ORGANIC INTERMEDIATES.

**Determination of ethylene.**—See II. [Furan derivatives from] agricultural wastes.—See XVI. Tests for methyl alcohol.—See XVIII. Cresol

soap solutions.—See XX. Alkyl *p*-hydroxybenzoates as disinfectants.—See XXIII.

See also A., May, 574, Furfuraldehyde. 578, Reduction of carboxylic acids. 612, Anthracene derivatives. 623, *p*-2-Carboxybenzoylbenzenesulphonic acids. 630, Pyridine bases from tar. 638, Detection of carbon disulphide. 656, Acetic fermentation. Propionic group of bacteria.

#### PATENTS.

**Production of organic compounds containing oxygen [acetic acid from methyl alcohol and carbon monoxide].** BRIT. CELANESE, LTD., and H. F. OXLEY (B.P. 343,947, 22.8.29).—There is added to the catalyst (*e.g.*, phosphoric acids or acid phosphates) as promoter iron, manganese, vanadium, chromium, cobalt, or nickel, or a compound thereof; *e.g.*, 0.5—1% of iron or manganese formate is added to pyrophosphoric acid to increase the yield of acetic acid from methyl alcohol and carbon monoxide at 290—315°/120—160 atm.

C. HOLLINS.

**Manufacture of oily polymerisation products from butadiene or a homologue thereof.** I. G. FARBENIND. A.-G. (B.P. 343,116, 16.10.29. Ger., 16.10.28).—Vapour of butadiene (*etc.*) is passed through a tube at 400—500°, in presence, if desired, of a catalyst, *e.g.*, iron. Butadiene gives vinylcyclohexenes, *b.p.* 110—135°; isoprene yields mainly dipentene, *b.p.* 165—180°; from  $\alpha\gamma$ -dimethylbutadiene there is obtained an oil, *b.p.* 195—210°/atm., 90—100°/13 mm.

C. HOLLINS.

**Antioxidant or age-resister [for rubber, oils, *etc.*].** A. M. CLIFFORD, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,779,390, 21.10.30. Appl., 27.7.29).—On heating piperidine and  $\alpha$ - or  $\beta$ -naphthol for several hours at 260—290° under pressure, products are obtained, *viz.*, 1- and 2-piperidynaphthalenes, respectively, which are effective antioxidants for rubber and for other oxidisable organic substances such as transformer oils. For the production of this type of antioxidant substituted naphthols also may be used in the reaction with piperidine.

D. F. TWISS.

**Manufacture of wetting, cleansing, and dispersing agents.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 343,098, 7.8.29).—Open-chain or monocyclic aliphatic ketones above  $C_7$ , free from aryl and carboxyl groups, are sulphonated. Oleone, obtained by heating oleic acid and iron filings at 340°, is treated with 23% oleum until the product is water-soluble and is not saponified by heating with 10% hydrochloric acid; or is treated with chlorosulphonic acid in carbon tetrachloride at 20—25°. The ketone from palmitic acid or from tall oil gives a similar product. C. HOLLINS.

**Manufacture of mercaptobenzthiazoles.** GOOD-YEAR TIRE & RUBBER Co., Assees. of J. TEPPEMA and L. B. SEBRELL (B.P. 343,013, 8.8.29. U.S., 26.11.28).—A mixture of carbon disulphide and an alkali salt of an *o*-aminothiophenol is treated with hydrochloric or sulphuric acid while heating under reflux. C. HOLLINS.

**Manufacture of (A) 1:4:4'-trihydroxy-2:2'-dianthraquinonyl 3:1'-oxide [2:6:9-trihydroxy-3:4:7:8-diphthaloyldiphenylene oxide] and (B) 1:4:1':4'-tetrahydroxy-2:2'-dianthraquinonyl.**

A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 343,090—1, 13.11.29).—Substance *A* of B.P. 10,074 of 1903 (B., 1904, 367) is 1:4:1':4'-tetrahydroxy-2:2'-dianthraquinonyl, whilst substance *B* of that specification is the corresponding furan derivative formed by loss of 2 hydrogen atoms. (B) Substance *A* is conveniently obtained free from substance *B* by warming quinzarin with piperidine in alcohol at 60–70°. (A) This product is converted smoothly into the furan derivative by heating in nitronaphthalene or other high-boiling nitro-compound. C. HOLLINS.

**Lubricants.**—See II. [Acetic acid from] black liquor.—See V. Elastic-plastic body.—See XIV.

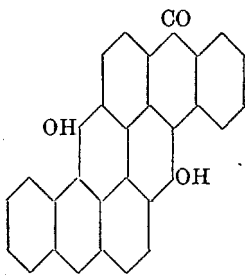
#### IV.—DYESTUFFS.

**Determining dyes in foods.**—See XIX.

See also A., May, 563, Action of electrolytes on substantive dyes. 618, Combined dye radicals. 627, Condensation of ketones with resorcinol. Fluoran derivatives. 632, Triphenylmethane dyes. Dyes from acenaphthaquinone. 636, Azo dyes from arsanilic acids. 644, Colouring matter of milk.

#### PATENTS.

**Manufacture of derivatives of pyranthrone [vat dyes].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 343,100, 9.8.29).—The dihydroxypyranthrone (annexed formula) or the monohydroxy-compound formed by its reduction, prepared by acid condensation of 2:2'-dialdehydo- or 2:2'-tetrahalogenodimethyl-1:1'-dianthraquinonyl in presence of a reducing agent, is alkylated and, if desired, halogenated. Dimethoxy- (golden-orange), diethoxy-, and monomethoxy- (red-brown) derivatives are described. C. HOLLINS.



**Manufacture of [acid] dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 342,667 and 342,706, 30.9.29).—4-Halogeno-1-aminoanthraquinone-2-sulphonic acids are condensed (A) with aromatic diamines (except *o*-arylenediamines) which may carry a single *N*-alkyl, -aralkyl, or -cycloalkyl substituent (*p*-phenylenediamine itself and its nuclear derivatives being excluded), or (B) with aromatic diamines (except *o*-arylenediamines), the product being subsequently acylated, especially by means of  $\omega$ -halogenoacyl halides. Green-blue to blue wool dyes of good levelling power are obtained. Examples are the condensation of 4-bromo-1-aminoanthraquinone-2-sulphonic acid with *m*-phenylenediamine (green-blue), acetylated (pure blue), chloroacetylated (reddish-blue), dichloroacetylated (blue), or  $\beta$ -chloropropionylated (blue); with benzidine (green-blue), acetylated (green-blue); with tolidine (blue); with 1:5- or 1:8-naphthylenediamine (blue or grey-blue); with 4:4'-diaminodiphenyl sulphide (blue); with *p*-aminoethylaniline (greenish-blue), acetylated (reddish-blue); with *p*-amino-*N*-cyclohexylaniline (greenish-blue); with *p*-phenylenediamine, acetylated (green-blue), chloroacetylated (blue), or con-

densed with ethyl chloroformate (blue); with *p*-phenylenediaminethiosulphonic acid, acetylated (blue); with *m*-aminoethylaniline, chloroacetylated (blue); with *p*-aminomethylaniline, chloroacetylated (blue).

C. HOLLINS.

**Manufacture of [mono-]azo dyes and their application.** SOC. CHEM. IND. IN BASLE (B.P. 343,016, 8.10.29. Switz., 8.10.28).—Diaz compounds are coupled in the 3-position with 4-hydroxy-1-naphthoic esters to give, according to the first component used, light-fast acid, mordant, lake, or cotton dyes, or pigments for acetate silk or lacquers. Examples of diazo components are: aniline (red), *p*-aminoacetanilide (bluish-red), *m*-aminobenzyl alcohol (red-orange), sulphanilic acid (red-orange), 4-chloro-*o*-aminophenol-6-sulphonic acid (chrome-red-violet), and  $\beta$ -naphthylamine-1-sulphonic acid (red barium lake). C. HOLLINS.

**Manufacture of [mono-]azo dyes [for acetate silk, lacquer pigments, etc.].** SOC. CHEM. IND. IN BASLE (B.P. 343,006, 2.11.29. Switz., 2.11.28).—A non-sulphonated *o*-aminophenol *o*-arylsulphonate is diazotised and coupled with a non-sulphonated phenol or arylamine of the benzene series or an aniline-formaldehyde-bisulphite compound, the dye is hydrolysed and then alkylated or acylated, to give pigments suitable for acetate silk dyeing and for colouring zapon varnishes. Examples are: *o*-aminophenyl toluene-*p*-sulphonate  $\rightarrow$  aniline, hydrolysed, and acetylated (greenish-yellow), or  $\rightarrow$  phenol, hydrolysed, and ethylated (greenish-yellow). C. HOLLINS.

**Manufacture of azo dyes [ice colours and pigments].** W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 343,419, 10.4.30. Addn. to B.P. 336,938 and 339,620; B., 1931, 151, 339).—2:3-Hydroxynaphthoic 3-chloro-4:6-dimethoxyanilide and 2-chloro-4:5-dimethoxyanilide are used as coupling components for pigments or ice colours, especially with diazotised *m*-xylydine, 4:6-dichloro-*m*-toluidine, or *o*-nitroaniline. C. HOLLINS.

**Manufacture of azo dyes insoluble in water [ice colours and pigments].** I. G. FARBERIND. A.-G. (B.P. 343,164, 15.11.29. Ger., 16.11.28).—Arylamides of 3-hydroxycarbazole-2-carboxylic acid are coupled in substance or on the fibre with diazo components free from solubilising groups. Examples are: anilide with diazotised 5-nitro-*o*-anisidine (brown), 3-aminocarbazole (red-brown), or tetrazotised dianisidine (blue-black);  $\beta$ -naphthylamide with tetrazotised dianisidine (dark blue), diazotised *o*-phenetidine  $\rightarrow$   $\alpha$ -naphthylamine (deep black);  $\beta$ -naphthylamide with diazotised 4-nitro-*o*-anisidine (brown); *p*-anisidine with diazotised 4-nitro-*o*-anisidine (black-brown); *o*-anisidine with diazotised 5-nitro-*o*-anisidine (brown). C. HOLLINS.

**Manufacture of [azo] dyes containing metal and the application thereof [in varnishes].** SOC. CHEM. IND. IN BASLE (B.P. 343,014, 7.10.29. Switz., 5.10.28).—Non-sulphonated, non-carboxylated monoazo dyes of the type: an *o*-aminophenol  $\rightarrow$  a phenol or an arylamine, are treated in presence of alkali but in absence of hyposulphites with hydroxides of one or more metals (preferably mordant metals) or with the products of the interaction of such hydroxides with alcohols and alkali.

Examples are: 2-amino- $\alpha$ -naphthol  $\rightarrow$   $\beta$ -naphthol with chromium hydroxide and aqueous potassium hydroxide to give a violet pigment soluble in varnish media; 4-chloro-*o*-aminophenol  $\rightarrow$  resorcinol with chromium hydroxide, alkali, and glycerol (brown-red varnish pigment); etc. The pigments are specially suitable for zapon varnishes. C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Factors affecting the development of mildew in wool.** R. BURGESS (J. Soc. Dyers and Col., 1931, 47, 96—99).—Mainly a review of previous investigations (B., 1930, 96, 1062) from a technical viewpoint. Commercially scoured worsted cloth containing about 0.5% of alkali (calc. as sodium carbonate) will not "mildew" at 23° unless its moisture regain is about 24%, which corresponds to the regain in an atmosphere of 94% R.H. Unscoured yarn, raw wool in the grease, and sized wools mildew at a lower R.H., whereas well washed wool mildews only slowly even in a moisture-saturated atmosphere. The presence of vegetable oils, but not mineral oils, in wool promotes mildew growth. Disintegration of wool by micro-organisms can be followed by means of the Rimington-Pauly test (cf. Rimington, B., 1930, 1021). Sodium silicofluoride is the most suitable mildew preventative for wool. The stoving of wool gives temporary protection, but over-chlorination (in the non-shrink finish) favours the growth of mildew. A. J. HALL.

**Identification of fungi causing mildew in cotton goods. The genus *Aspergillus*.** II. G. SMITH (J. Text. Inst., 1931, 22,  $\tau$  110—116).—An analysis is made of the whole series of strains belonging to the above group isolated from mildewed cloths and yarns.

B. P. RIDGE.

**Permeability of fabrics to air.** M. C. MARSH (J. Text. Inst., 1931, 22,  $\tau$  56—63).—The apparatus and methods used are fully described, and comparisons are made between the permeabilities of knitted and woven cotton, woollen, artificial silk, linen, and silk fabrics. Large variations from point to point are found for most fabrics.

B. P. RIDGE.

**Production of cellulose by means of chlorine. III. Chlorination.** II. J. KAWAMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 62—64 B; cf. B., 1931, 478).—Samples of bagasse were chlorinated for various times over the temperature range 22—27°, using different proportions of chlorine to material, and the ratio of chlorine present as hydrochloric acid to combined chlorine and the yield of cellulose were determined. When the temperature and time of reaction are increased, the amount of chlorine taking part is increased, whilst, as previously observed by Waentig, the ratio of hydrochloric acid formed to combined chlorine remains practically constant. When a much larger amount of chlorine is used for a long time, this ratio is increased and probably oxidation of the non-lignin or of the lignin substance is promoted.

B. P. RIDGE.

**Cellulose acetate.** H. REEDY (Kunstseide, 1930, 12, 420—422; Chem. Zentr., 1931, i, 596).—Acetyl-sulphuric acid is considered to be formed first, and

then from it sulphoacetic acid. The formation of sulphoacetates is unlikely to be prevented by acetylation with acetylsulphuric acid, in which free sulphuric acid and sulphoacetic acid are in equilibrium.

A. A. ELDRIDGE.

**Production of viscose silk of high tensile strength.** H. SCHMIDT (Chem.-Ztg., 1931, 55, 265—267, 286—288).—The latest processes as described in patent literature are outlined, with special reference to the composition of the spinning baths and methods of stretching the thread.

F. R. ENNOS.

**Chemical analysis of rayons. I. Chemical properties of some commercial rayon yarns.** B. P. RIDGE, II. L. PARSONS, and M. CORNER (J. Text. Inst., 1931, 22,  $\tau$  117—140).—The following properties have been measured for standard (first quality) varieties of viscose, Lilienfeld, cuprammonium, acetate, and nitrocellulose rayons of recent manufacture, mostly of British origin: copper number, loss of weight on boiling with alkali, methylene-blue absorption, fluidity in cuprammonium hydroxide solution, ash content and ash alkalinity, material extractable by solvents, sulphur, copper, and iron contents, and, for acetate rayons, acetic acid content. The first four are dependent mainly on the extent of chemical modification of the cellulose itself, whilst the remainder are indicative of the nature and amount of the non-cellulose impurities. The chemical properties of these materials resemble those of chemically attacked cotton cellulose rather than those of the purified but otherwise unchanged material. None of the chemical properties measured can be used to characterise all the varieties examined. Copper number and loss of weight fail to distinguish between viscose, Lilienfeld, and cuprammonium yarns made from wood pulp; methylene-blue absorption is lowest for acetate rayon, but is otherwise very variable; and fluidity values are low and approximately the same for Lilienfeld and cuprammonium rayons made from cotton linters and high for acetate varieties, though here they are of little significance because these are not composed wholly of cellulose. All the tests applied show the chemical inferiority of nitrocellulose rayon.

B. P. RIDGE.

**Durability of purified wood fibres. Accelerated ageing tests of various types of papermaking fibres.** G. A. RICHTER (Ind. Eng. Chem., 1931, 23, 371—380).—Papers made from rag stock, sulphite wood pulp, and purified wood pulp of high  $\alpha$ -cellulose content, respectively, have been tested. Exposure to a stream of air at 100° for 72 hrs. causes appreciable reduction of physical strength of sulphite papers, rag and purified wood retaining a high percentage of their original strength. Oxygen accelerates the decomposition of paper, exposure at 60° and 200 lb./in.<sup>2</sup> giving similar results to the ageing at 100° in air. Decomposition is, in general, accelerated by the presence of moisture. Highly-hydrated pulps prepared from the purified wood fibres do not deteriorate to any greater extent than those beaten normally, whereas highly-beaten sulphite pulps decompose readily. Rag and purified wood papers are no more resistant to the action of acid vapours than sulphite papers. Extensive

tables showing the effect of ageing on the  $\alpha$ -cellulose content, tensile strength, tearing strength, and folding endurance are given. T. T. POTTS.

**Correlation of bursting strength and tensile strength of paper.** J. STRACHAN (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 86—100).—The formulæ advanced by Dawe, Bergmann, Carson, and Dalén for the connexion between bursting strength and tensile strength are discussed and criticised. Deviation of calculated tensile strength from observed values is shown to be an indication of the degree of homogeneity of the sheet, and a factor of uniformity,  $v = Br/2T$ , where  $r$  is the radius of the test disc used for the burst, is claimed to yield satisfactory results. The factor  $v$  is shown to depend on fibre length and cohesion.

T. T. POTTS.

**Variability in results in paper strength testing.** C. V. OLIVER (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 53—74).—Numerical examples of the variations occurring in the testing of paper for bursting, tearing, and tensile strengths are given. It is shown that the errors vary according to the type of paper being examined, and that different instruments designed for a given test, *e.g.*, the Mullen and Ashcroft bursting testers, do not yield results which bear a constant relation to one another. The effects of varying conditions on the accuracy of results are discussed.

T. T. POTTS.

**Colouring of paper.** N. L. MATHEWS (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 144—156).—A lecture, giving an account of the various natural and synthetic colouring matters used in the paper industry, with notes on their application.

T. T. POTTS.

**Soda recovery [in the paper mill].** J. A. WALKER (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 187—198).—An account is given of practical experience in the conduct of plant for the recovery of soda from the spent liquors of esparto digestion.

T. T. POTTS.

**Sampling of cotton for determination of fibre properties.** III. Size and reliability of a satisfactory sample. R. S. KOSHAL and A. J. TURNER (J. Text. Inst., 1931, 22, T 17—55).

**Fabric wear testing.** I. H. CRAWSHAW, W. E. MORTON, and K. C. BROWN (J. Text. Inst., 1931, 22, T 64—76).

**Random and systematic selections of warp specimens in cloth sampling.** A. J. TURNER (J. Text. Inst., 1931, 22, T 77—97).

See also A., May, 606, Cellulose butyrate, and nitrate. Red beech. 655, Cellulose-fermenting organism.

#### PATENTS.

**Manufacture of soluble ethers from vegetable materials.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 343,147, 14.11.29).—Straw, sea-weed, esparto grass, etc., containing fibres which are encrusted but not turned into wood, are alkylated, and especially benzylated, by the methods used for cellulose ethers.

C. HOLLINS.

**Manufacture of cellulose acetate.** KODAK, LTD., Assees. of C. J. STAUD and C. S. WEBBER (B.P. 345,235,

10.12.29. U.S., 22.12.28).—Cellulosic material is treated at 0° with a saturated solution of nitrogen peroxide in a lower aliphatic acid, preferably acetic acid, before being acetylated in the usual manner. F. R. ENNOS.

**Treatment of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 345,408, 29.1.30. U.S., 7.2.29).—Cellulose esters are fractionally precipitated from solution, *e.g.*, by addition of a non-solvent or by cooling, yielding fractions differing in physical properties.

F. R. ENNOS.

**Production of artificial filaments, films, and like products from viscose.** H. DREYFUS (B.P. 344,474, 5.11.29).—Viscose is coagulated in the required form as a cellulose metal xanthate in a bath free from mineral acid and composed of either an organic liquid (alcohol) or an aqueous solution of a metallic salt; the product is continuously reconverted into cellulose by means of mineral acids, acid esters of mineral acids, or organo-mineral acids in low or high concentration, together with glycerin, sugars, sulphonated oils, etc., if desired.

F. R. ENNOS.

**Manufacture of artificial filaments, threads, ribbons, films, and other artificial products.** H. DREYFUS (B.P. 344,510, 7.12.29).—Materials of modified lustre are produced by spinning solutions of cellulose or of cellulose derivatives containing insoluble organic fibrous material (cotton, linen, straw, wheat husks, wool, or artificial fibrous materials) in a finely-divided state.

F. R. ENNOS.

**Spinning of artificial threads or filaments.** CELLULOSE ACETATE SILK Co., LTD., and H. C. CURTIS (B.P. 344,591, 10.1.30).—To obtain a temporary increase of pressure at the jet when starting the spinning operation, communication between the jet and the supply pipe is closed, and the solution pumped into a pressure chamber in or connected with the supply pipe; when a sufficiently high pressure is reached the pressure chamber is connected with the jet.

F. R. ENNOS.

**Dry-spinning of artificial silk.** "CHATILLON" Soc. ANON. ITAL. PER LA SETA ARTIFICIALE, and E. ORIOLI (B.P. 344,385, 25.11.29).—The spinning cell is provided with a pipe open at each end, which is heated by either an internal pipe or an external jacket. The rising current of gases in the pipe is caused to impinge on the filament from one side only of the cell through the upper aperture, which has its axis at right angles to the path of the filament.

F. R. ENNOS.

**Treatment of artificial fibres.** L. A. PALEY (U.S.P. 1,779,103, 21.10.30. Appl., 17.12.26).—Freshly spun fibre cakes, after insertion of a specially constructed holder, are grouped end to end in the form of a closed cylinder on a tube having a number of openings through which water, hot dilute sodium carbonate, or air is passed under pressure to the interior and through the walls of the cakes, whereby the latter are washed, freed from impurities, and dried; the holder also facilitates unwinding of the fibre cake therefrom after treatment.

F. R. ENNOS.

**Digestors for treating fibrous materials.** J. J. DE LA ROZA (B.P. 346,119, 6.3.30).—The digestor is constructed so that the material may be charged, digested, and discharged while contained in baskets or

other containers; the manhole is of about the same diameter as the cylindrical body portion.

B. M. VENABLES.

**Manufacture of pulp.** J. NEUMANN (U.S.P. 1,778,199, 14.10.30. Appl., 14.6.26. Renewed 4.1.30).—Cellulosic raw material is digested with a solution of sodium silicate, either hot or cold, to which a solution of sodium sulphide is afterwards added. F. R. ENNOS.

**Manufacture of cellulose pulp.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 345,208, 12.12.29. Ger., 13.12.28).—At any desired stage before working up into cellulose solutions or compounds, the pulp is broken down by intensive chemical treatment such as boiling with acids, strong bleaching, or oxidation in the presence of alkali, so that it has a copper viscosity (method of determination described) of less than 6. It is then freed from hemicellulose by dilute alkali until the  $\alpha$ -cellulose content is at least 90% (preferably >93%). F. R. ENNOS.

**Manufacture of transparent sheet material.** S. D. WARREN CO., and W. M. DRIESEN (B.P. 344,471, 29.10.29).—A paper web composed of transparent fibres is saturated with a solution of a cellulosic film material (nitrocellulose) in a mixture of a volatile solvent (alcohol and volatile esters) and a stable solvent (lanoline, amyl phthalate), and the volatile solvent is removed by evaporation. F. R. ENNOS.

**Stencil sheet.** E. E. NOVOTNY (U.S.P. 1,776,368, 23.9.30. Appl., 11.7.28).—A porous base (Yoshino paper) is coated with a cellulose ester solution (pyroxylin lacquer) containing ethyl oleate and carnauba wax. F. R. ENNOS.

**Manufacture of impregnated sheets.** H. C. CHEETHAM, Assr. to BAKELITE CORP. (U.S.P. 1,776,885, 30.9.30. Appl., 16.11.26).—A reactive resin, *e.g.*, of the phenol-formaldehyde type, together with a suitable plasticiser (ethyl tartrate) is heated until it loses its initial fusibility and solubility; after fine grinding, the plasticised resinoid is mixed with fibre in the presence of water and laid into sheets, which are subsequently dried and hot-pressed. F. R. ENNOS.

**Hektographic materials and blankets.** W. W. TRIGGS. From DITTO INC. (B.P. 345,062, 25.2.30).—The coating materials comprise a glue and glycerin composition, containing approx. 0.5% of titania.

H. ROYAL-DAWSON.

**Manufacture of dialysing and pervaporating membranes.** P. A. KOBER (U.S.P. 1,779,942, 28.10.30. Appl., 26.11.26).—A cellulose derivative (nitrocellulose) and a protein substance (gelatin) are dissolved in a common solvent (acetic or other aliphatic carboxylic acid) and the membrane, which is formed in the required shape by removal of the solvent, is treated with water. (Cf. B., 1917, 1038.) F. R. ENNOS.

**[Wax-]impregnated fibrous materials.** H. FRIEDLANDER, and SPICERS, LTD. (B.P. 345,453, 26.2.30. Addn. to B.P. 236,224; B., 1926, 986).—Instead of hard wax the impregnating material used consists of montan wax pitch, brown-coal wax pitch, or similar pitchy residues derived from wax-containing materials. F. R. ENNOS.

**Production of designs [on paper, cloth, etc.].** C. MAIER and W. SWAYSLAND (U.S.P. 1,778,397, 14.10.30. Appl., 18.8.28).—A visible design is formed on white paper etc., the surface of which has been impregnated with a series of tiny spots of lead sulphate, by treating certain of the spots with a solution of a soluble sulphide; the paper (etc.) may be used again by reconvertng the lead sulphide spots into lead sulphate by means of hydrogen peroxide. Half-tones are thus obtainable, and spots of other colours produced with different solutions. F. R. ENNOS.

**Device for evaporation of the solvents in the manufacture of papers and films from cellulose esters and ethers.** A. GIEBMANN (B.P. 344,645, 24.2.30. Fr., 23.2.29).—The casting cylinder or other device on which the films etc. are formed is enclosed in a chamber subdivided into several compartments, preferably four, each containing one or more heating devices such as radiators; as the amount of solvent to be evaporated in the successive compartments decreases, the radiators are arranged nearer to the casting surface of the cylinder, which finally passes through a compartment containing water, heated or not, to remove the last traces of solvents and provide the film surface with water vapour, to avoid formation of static electricity. F. R. ENNOS.

**Treatment of black liquor [from soda-pulp process].** L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,779,226, 21.10.30. Appl., 3.1.21. Renewed 5.7.28).—Black liquor is treated with sufficient acid or acid salt yielding a causticisable sodium salt to precipitate most of the organic matter or in addition to liberate the acetic and other volatile acids. After removal of the organic matter, the liquor is causticised before or after concentration and is returned to the digester for re-use; the acetic acid in the liquor is recovered either as sodium acetate during concentration, or as free acid by distillation of the acid liquor. F. R. ENNOS.

**[Apparatus for] stretching of artificial threads.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 347,454, 7.4.30. Ger., 29.4.30).

**Waterproofing of paper.**—See VI. Spent wood liquors.—See XII. Rubber compositions.—See XIV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Detection of traces of active chlorine in [bleached] textiles.** A. SCHMIDT (Z. angew. Chem., 1931, 44, 278).—The production of a blue colour when bleached textile fibres are brought into contact with an iodide-starch mixture is not proof of the presence of hypochlorite or free chlorine (cf. Schwarze, B., 1931, 291), neither of which is likely to exist for any period in contact with organic materials. It may, however, result from the presence of relatively stable compounds of the chloroamine type, which are frequently produced during the bleaching of linen and cotton; they may be removed most readily by treatment of the material with bisulphite or thiosulphate. H. F. GILLBE.

**Measurement of the colour of textile fabrics.**  
**VII. Relations between concentration of dyestuff**



and colour of unexposed and exposed fabrics. P. W. CUNLIFFE and (Miss) P. N. LAMBERT (J. Soc. Dyers and Col., 1931, 47, 73—78).—A continuation of previous work (B., 1930, 657), with special reference to the relations between "concentration" and "colour quality," between "concentration" and "brightness factor" (luminosity), and between the "brightness factor" of exposed and unexposed dyeings, these relations being illustrated by means of numerous graphs. The "colour quality" curves of Acetyl Rose 2GL and Coomassie Violet R dyed on worsted indicate that in dyeings of concentration exceeding 4% and 2%, respectively the colour becomes darker or less pure. The linear relationship discovered previously (J. Soc. Dyers and Col., 1929, 45, 306) between luminosity ("brightness factor") of a dyed material and the logarithm of the concentration of the dye has been examined and confirmed over a much wider range of concentrations with a number of dyes; the earlier results have also been confirmed for concentrations less than the optimum. Owing to the change in "brightness factor" observed during the fading of several dyes it is concluded that no method is permissible for calculating the degree of fading which assumes that a dyeing will fade to a colour equal in brightness to that of the original undyed material (cf. Haller and Ziersch, B., 1930, 369).

A. J. HALL.

**Fur dyes : their oxidation and identification on the fibre.** R. B. FORSTER and C. SOYKA (J. Soc. Dyers and Col., 1931, 47, 99—109).—Unoxidised bases may be extracted by means of ligroin or ether, the evaporated extract being further extracted with 0.1N-hydrochloric acid for separation of fats from the bases, or by means of that acid after first degreasing the fur; these methods are satisfactory for rabbit, skunk, sable, beaver, and sheep skins. Full details are given of a number of colorimetric methods for identifying the bases in the resulting extract, and the reactions of a number of bases are tabulated. Bandrowski's base, formed by oxidation of *p*-phenylenediamine, reacts very similarly to *p*-phenylenediamine in the Indamine and hypochlorite tests, but it can be distinguished by reducing with zinc and hydrochloric acid and then exposing a drop of the resulting colourless solution on filter paper to ammonia; the spot becomes brownish and then brownish-violet with a pink rim. A satisfactory method is described for determining *p*-phenylenediamine in dilute solution by titration with nitrous acid; in this reaction only one amino-group is diazotised, the second group forming an inner diazoamino-compound. *p*-Phenylenediamine oxidises with hydrogen peroxide more easily than the *m*-compound in the absence of a mordant, but the reverse is true when a mordant is present. *p*-Phenylenediamine has been identified in the reduction products of Bandrowski's base.

A. J. HALL.

**Mildew in wool. Colouring of paper.**—See V. **Determining dyes in foods.**—See XIX.

See also A., May, 572—3, **Kinetics of chlorine bleaching.**

## PATENTS.

**Finishing [bleaching or dyeing] of yarns or fabrics.** F. J. STOKES (U.S.P. 1,779,831, 28.10.30. Appl.,

22.6.25).—The usual treatments for preparing yarns and fabrics for dyeing are avoided by placing the textile material (in the standard package or form in which it is manufactured) in a centrifuge situated within a closed tank, creating a vacuum, admitting dye liquor until the material is completely immersed, applying air pressure to assist penetration of dye liquor into the textile material, and removing excess of dye liquor by means of the centrifuge.

A. J. HALL.

**Printing of cotton [with vat dyes].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,102, 14.8.29).—Relatively stable printing preparations are made by reducing a vat dye, in the absence of caustic alkali, with a reducing agent (hyposulphite, titanous chloride, etc.) and a weak alkali (ammonia, potassium carbonate) in presence of water and glycerin or other water-soluble alcohol (e.g., glycol, diglycol, thiodiglycol, thiodiglycerol, glycerol hydroxyethyl ether). The leuco-compound is present in insoluble form in stable suspension, which may be concentrated by evaporation. Hydrotropic substances, e.g., urea, sodium benzenesulphonate, phthalic anhydride, sodium dimethylaniline-*m*-sulphonate or naphthalene-2-sulphonate, may be added. The preparations are printed in the usual way with thickening agent and formaldehydesulphoxylate, steamed damp, and developed with dichromate and acetic acid.

C. HOLLINS.

**Dry cleaning.** L. E. JACKSON (U.S.P. 1,778,948, 21.10.30. Appl., 8.4.29).—The development of rancidity in textile materials which have been dry-cleaned (due to their retention of free fatty acids from the dry-cleaning liquor) may be avoided by adding periodically to the liquor sufficient of an alkali to neutralise acid substances present in the materials being cleaned.

A. J. HALL.

**Laundrying methods.** R. A. PHAIR, Assr. to H. KOHNSTAMM & Co., INC. (U.S.P. 1,777,857—S, 7.10.30. Appl., [A] 11.6.23, [B] 7.10.24).—In a laundrying process which comprises treatment of the goods with a liquor containing an alkali, the removal of this alkali in the final rinsing is facilitated by adding (A) ammonium salts, e.g., ammonium chloride and sulphate, or (B) ammonium sulphite, to the rinsing water.

A. J. HALL.

**Treatment [delustring] of textile [cellulose ester and ether] materials.** BRIT. CELANESE, LTD. (B.P. 345,509, 31.3.30. U.S., 12.4.29. Addn. to B.P. 343,698; B., 1931, 486).—The fabrics etc. are treated as described in the prior patent except that they are suspended in the delustring liquor in the form of hanks in such a way that they are completely immersed.

A. J. HALL.

**Manufacture of spangled, diamanté, and like fabrics, leather, and like flexible materials.** M. I. KELLER (B.P. 344,585, 7.1.30).—Fabric is coated with a caoutchouc adhesive, then dusted or sprayed with spangles, pressed, and excess of spangles removed, e.g., by suction.

A. J. HALL.

**Treatment of textile materials.** BRIT. CELANESE, LTD. (B.P. 345,022, 28.1.30. U.S., 6.2.29).—Fabric having a cockled appearance is produced by weaving

with mixed yarns containing fibres of cellulose acetate or other organic derivative of cellulose and fibres of another material and then treating the fabric with a reagent which causes unequal shrinkage of the two types of fibres. Alternatively, the fabric is woven from similar mixed yarns already treated with the shrinking agent. When the mixed yarn consists of cotton and cellulose acetate silk and caustic soda is used for effecting shrinkage, the alkali should be used at about  $0^{\circ}$  so that hydrolysis of the silk is retarded. A. J. HALL.

**Treatment [with ozone] of wool, fur, and other animal fibres, or textile fabrics containing them.** S. W. WILKINSON (B.P. 345,406, 27.1.30).—To prevent over-ozonisation with consequent deterioration of the fibres when treated as in B.P. 317,133 and 242,027 (B., 1929, 848; 1926, 11), they are pretreated with gaseous or a solution of formaldehyde, with or without the presence of alkaline or acid solutions or gases.

H. ROYAL-DAWSON.

**Treatment [sizing] of textile materials.** BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and P. F. C. SOWTER (B.P. 344,528, 27.8.29).—The materials are sized with aqueous solutions of water-soluble hydroxy- or carboxy-alkyl ethers of cellulose or degraded cellulose.

F. R. ENNOS.

**Waterproofing of fibrous products.** F. QUECK (U.S.P. 1,777,447, 7.10.30. Appl., 3.9.29).—Wood pulp is mixed with an emulsion of unsaponifiable substances such as asphalt or a blown asphalt (*e.g.*, Parolite and Stanolite), the emulsion being prepared with the aid of a soap, particularly a rosin soap, and then an acid or acid salt is added to render the asphalt insoluble; fillers such as starch, lampblack, or calcium sulphate may also be added.

A. J. HALL.

**Waterproofing compositions [for paper].** C. S. SHELTON (U.S.P. 1,778,964, 21.10.30. Appl., 18.4.25).—Compositions consisting of a mixture of one or more metal oxides (zinc oxide and/or lead oxide), benzene, light petroleum, and dammar gum or sodium silicate solution are claimed.

A. J. HALL.

**Impregnated and coated fabric.** G. BAEKELAND, ASSR. to BAKELITE CORP. (U.S.P. 1,776,879, 30.9.30. Appl., 28.1.26).—Rubber-coated fabrics such as are used in the manufacture of motor tyres, pressure hose, belting, etc. are manufactured from fabrics which are initially impregnated with a varnish which yields a flexible film of the phenol-methylene resinoid type; the bond between the subsequently applied coat of rubber and the fabric is thereby improved and the resulting products have increased tensile strength.

A. J. HALL.

(A) Aircraft covering. (B) Flexible impermeable fabric. I. M. JACOBSON and S. TRUSCOTT (U.S.P. 1,779,846—7, 28.10.30. Appl., 5.3.26).—The base fabric (cotton cloth) is coated (A) with a mixture of dried rubber with carbohydrates (glucose), polyhydric alcohols or their derivatives (glycerol, triacetin), or gelatin, which has been milled and dissolved in an organic solvent (benzene), the rubber being afterwards vulcanised; or (B) with cuprammonium cellulose and, after coagulation of the latter with acid, is washed and plasticised by immersion in glycerin.

F. R. ENNOS.

**Coating of [balloon] fabrics.** W. C. CALVERT, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,779,388, 21.10.30. Appl., 2.12.27).—Flexible and durable fabric which is non-permeable to gases is prepared by coating a fabric successively with solutions consisting of 1 litre of water and (1) 60% rubber latex 200 c.c.; (2) 60% rubber latex 100 c.c., gelatin 30 g., and glycerin 60 g.; (3) 60% rubber latex 50 c.c., gelatin 60 g., and glycerin 120 g.; (4) gelatin 40 g., glycerin 80 g., and alum 2.4 g., drying between each coat, then sponging with water and applying a layer of gas-resistant Cellophane.

A. J. HALL.

**[Fire- and mildew-]resistant [textile] material [for mine-ventilating tubing].** F. A. McDERMOTT and F. J. FUNK, ASSRS. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,779,258, 21.10.30. Appl., 3.4.25).—The fabric is impregnated with a solution of zinc salicylate (or similar water-soluble antiseptic), dried, and then coated with a rubber composition containing paraffin, pine oil, zinc salicylate, and barytes; the whole is then vulcanised.

A. J. HALL.

**Treatment of sacking and the like to prevent rotting of the same.** F. H. COTTON, and WAKELEY BROTHERS & CO., LTD. (B.P. 345,468, 6.3.30).—Sacking material is impregnated with a 2% solution of copper sulphate tinted with a basic dye, particularly Brilliant Green Crystals Y, and then treated with a solution containing sodium silicate and common yellow soap, whereby copper stearate and silicate are precipitated as protective agents within the fibres.

A. J. HALL.

**Liquid treatment of yarn in hank or skein form.** W. GERBER (B.P. 347,284, 27.1.30).

**Treatment [packaging] of textile strands [to facilitate bleaching, dyeing, etc.].** E. J. ABBOTT (B.P. 346,895, 6.2.30).

**Jigger machines for the dyeing or like treatment of fabrics.** MASCHINENFABR. BENNINGER A.-G. (B.P. 347,059, 26.6.30. Ger., 20.7.29).

**Machines for treating textile fabrics [to produce moiré effects].** A. MILHOMME (B.P. 347,034, 23.5.30. U.S., 4.4.30).

**[Shrinkage-preventing] treatment of textile fabrics [during laundering].** TOOTAL BROADHURST LEE CO., LTD., and K. S. LAURIE (B.P. 346,782, 16.1.30).

**Wetting etc. agents.**—See III. Designs on paper etc.—See V. Coloured rubber.—See XIV. Protecting leathers etc.—See XV. Photographic bleaching-out process.—See XXI.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Theory of the lead-chamber [sulphuric acid] process.** "Blue acid"  $[\text{SO}_5\text{NH}_2]$ . E. BERL and H. H. SAENGER (Z. angew. Chem., 1931, 44, 291).—Blue acid, formed by reduction of nitrosylsulphuric acid by sulphur dioxide, plays a part in most theories of the lead-chamber process. It can be produced as a blue coloration by cathodic reduction of concentrated sulphuric acid containing  $\text{SO}_5\text{NH}_2$  at an *E.M.F.* of 3–4 volts, and by passing nitrogen peroxide and water

vapour into liquid sulphur dioxide at  $-70^{\circ}$ . At ordinary temperatures it rapidly decomposes:  $\text{SO}_5\text{NH}_2 = \text{H}_2\text{SO}_4 + \text{NO}$ . This latter reaction has been shown experimentally to be reversed at pressures of the order of 250 atm., when up to 77% combination as blue acid has been obtained.

C. IRWIN.

**Preparation of hydrogen cyanide by oxidation of ammonium thiocyanate or thiocyanic acid with nitric acid.** W. GLUUD and K. KELLER (Ber. Ges. Kohlentechn., 1931, 3, 395—419).—The principal reaction is  $\text{RCNS} + 2\text{HNO}_3 = \text{HCN} + 2\text{NO} + \text{RHSO}_4$ . A 20% solution of thiocyanate is run into a stirring vessel with nitric acid, the latter being kept in excess; the temperature must be approx.  $100^{\circ}$ . The gases are scrubbed with water at  $80^{\circ}$ , which produces nitrous acid, and are then passed into an alkaline absorbing solution. The remaining nitric oxide is regenerated for further use. This process was originally worked out in England, using sodium or calcium thiocyanate. The authors in applying it to the ammonium salt find that for satisfactory yields in this case a minimum concentration of 1.5 g. of acid ( $d$  1.14) per 100 c.c. must be maintained in the liquor. Losses of nitric oxide occurred and formation of ammonium nitrite with subsequent decomposition was frequent. On this account the oxidation of thiocyanic acid formed by decomposition of the ammonium salt with the sulphuric acid produced was investigated. The solution of the free thiocyanic acid decomposes in a few hours. Decomposition is retarded by dilution and by cooling with ice. It must therefore be oxidised as soon as it has been prepared. The process proposed is to treat a 25% ammonium thiocyanate solution with 78% sulphuric acid and oxidise with excess of nitric acid ( $d$  1.20). After saturation of the residual liquor with ammonia "Leuna saltpetre" is obtained. The decomposition is carried out in a stoneware or ebonite-lined apparatus. Alternatively, a 17% solution of thiocyanic acid is to be oxidised with nitric acid in a similar apparatus. Laboratory experiments are described in detail and yields and water, steam, and power consumptions calculated.

C. IRWIN.

**Preparation of ammonium sulphate in coarse tabular crystals.** W. GLUUD, W. KLEMPF, and H. RITTER (Ber. Ges. Kohlentechn., 1931, 3, 371—384).—It has been shown that the presence of iron as an impurity is responsible for the production of ammonium sulphate in the fine acicular form usually met with in the coke-oven product. The variation of crystalline form with variation of temperature, method of evaporation, and speed of agitation was studied in the laboratory. Little difference in the product was observed except that with very low speeds of agitation a powdery crystal was obtained as from stationary crystallisation. Further tests with mother-liquor from a coke-oven ammonium sulphate plant showed that the crystal form was not due to the presence of tar or tar products or of arsenic. Iron, however, which is present in the ferrous state, and is not precipitated by excess of ammonia, is important. If it was oxidised with hydrogen peroxide and then removed by precipitation the salt was obtained as coarse tabular crystals. On

adding ferrous ammonium sulphate to the purified liquor, crystals of the fine needle type were obtained. The iron may equally be removed as sulphide. Ferric iron (which is reduced under coke-oven conditions), chromium, and aluminium are all harmful, giving a paste of fine crystals. The action of ferrous iron appears to be due to adsorption which prevents crystal growth except in one direction. A process was worked out in the laboratory and on a semi-works' scale for the elimination of iron by atmospheric oxidation, keeping the saturator alkaline. On the larger scale the use of air was afterwards supplemented by hydrogen sulphide treatment, using untreated gas. The precipitated iron was removed by a coke filter or centrifuge, and the liquor passed to a vacuum evaporator. It was found possible to work without the use of air-blowing, and the iron sulphide was easily separated. It was essential to maintain 0.09% free  $\text{NH}_3$  in the liquor. Air oxidation requires 0.3—0.4% free  $\text{NH}_3$  and a temperature of  $60$ — $80^{\circ}$ , and the ferric hydroxide is not so easy to separate. The crystals of salt obtained, though of the desired form, were fine owing to the rate of evaporation exceeding the rate of growth of the crystals. This was altered by changing the evaporator design, after which about 80% was obtained above 0.5 mm. in diam. The application of this process to the direct, indirect, and semi-direct sulphate processes is described and costs are discussed.

C. IRWIN.

**Production of superphosphate from Khibinsk apatite.** S. I. VOLFKOVICH, L. E. BERLIN, and L. B. GRINSPAN (Udobr. Urozhai, 1930, 2, 300—312).—Enriched apatite obtained from nepheline apatite rock by fine grinding and sieving or flotation is used. When 88% of the rock is milled to less than 0.1 mm. the material (40%  $\text{P}_2\text{O}_5$ ) gives an acid phosphate with 18% of soluble  $\text{P}_2\text{O}_5$ .

CHEMICAL ABSTRACTS.

**Conversion of cyanide solution into potassium ferrocyanide and the oxidation of the latter to ferricyanide by compressed air and electrolysis.** W. GLUUD [with W. KLEMPF, K. KELLER, F. BRODKORB, and C. DIECKMANN] (Ber. Ges. Kohlentechn., 1931, 3, 385—394).—The problem investigated was the utilisation of hydrogen cyanide present in a gas in large quantities. This compound was first converted into calcium cyanide and the slightly alkaline solution at  $80^{\circ}$  stirred, with gradual addition of concentrated ferrous sulphate slightly in excess of the theoretical. It was filtered from calcium sulphate and the filtrate treated with potassium carbonate solution with heating. After again filtering, the filtrate was evaporated *in vacuo* and the calcium carbonate used for further cyanide absorption. The yield of ferrocyanide in the laboratory was 97%. The oxidation of ferrocyanide is reversed by the potassium hydroxide liberated in the reaction, and means must be used to prevent alkalinity developing. Chlorine is not suitable owing to the difficulty of separating the potassium ferricyanide from chloride. Other reagents are expensive, and the authors propose the use of air with carbon dioxide at high pressure, which process is, however, not quantitative unless supplemented by other methods. Experiments conducted in a small autoclave at 50 atm. showed that

sulphuric acid was less effective than weaker acids or carbon dioxide. The optimum temperature is 100°, and little variation in results occurred between 20 atm. and 50 atm. pressure. Using oxygen and carbon dioxide, 10 atm. is sufficient, and the theoretical quantity only of carbon dioxide is necessary for 90–95% oxidation. A 20% calcium ferrocyanide solution is most convenient for treatment, and good agitation is necessary. In order to complete oxidation, the treated solution is electrolysed at 50° between nickel electrodes at a current density of 0.005–0.1 amp./cm.<sup>2</sup>, and an *E.M.F.* of 7–12 volts. Solid calcium oxide is added to the anode solution to neutralise the free acid formed. The current efficiency is about 50%. The solution is then treated with the equivalent of potassium carbonate, filtered, and evaporated *in vacuo*. The whole process can be carried out with a yield of 90–92%.

C. IRWIN.

**Complex cyanides of nickel, copper, and chromium.** W. GLUUD and W. RIESE (Ber. Ges. Kohlentechn., 1931, 3, 452–459).—Titration with potassium iodide and silver nitrate indicates that a ferrocyanide is decomposed by ammonia with formation of cyanide and complex cyanides containing ammonia. A similar action occurs with potassium nickel cyanide. In this case the final products are KCN and  $[\text{Ni}(\text{NH}_3)_4](\text{CN})_2$ , all the cyanide being capable of titration with silver nitrate. The nickel salt does not, however, react with ferrous cyanide to form ferrocyanide. Copper acts similarly. With ammonium polysulphide, on the other hand, no cyanide is formed, but the final products are nickelous sulphide and ammonium thiocyanate. Copper and chromium react similarly, but less readily. With ferrous sulphate a white compound,  $\text{Fe}[\text{Ni}(\text{CN})_4]$  is produced, or, in alkaline solution, ferrous hydroxide. The copper and especially the chromium complex cyanides appear to produce some ferrocyanide with ferrous sulphate.

C. IRWIN.

**Determination of polysulphide and preparation of calcium sulfuratum solum (Vlemingx' solution).** J. BÜCHI (Pharm. Acta Helv., 1930, 5, 124–132; Chem. Zentr., 1931, i, 978).—A solution of boric acid (1 g.) in water (60 c.c.) is boiled, 0.2 g. of potassium cyanide and 1 c.c. of the solution being successively added; after 10 min. the liquid is cooled and diluted to 100 c.c. A portion (50 c.c.) is treated with concentrated hydrochloric acid (1–2 c.c.) and excess of bromine water, the excess being removed with phenol. After 10 min. potassium iodide (1 g.) is added, and after a further 15 min. the iodine is titrated with thiosulphate.

A. A. ELDRIDGE.

**Solubility of Bordeaux mixture.** G. L. HOCKENROS (Phytopath., 1931, 21, 231–234).—The method of Callan and Henderson (A., 1930, 53) is applied to the examination of Bordeaux mixtures. Any turbidity produced during the colour test by the presence of calcium salts is avoided by acidifying the test solution with hydrochloric acid, making alkaline with ammonia, and adding saponin solution to peptise colloidal matter, prior to the addition of the diethyldithiocarbamate reagent. More metallic copper dissolves in boiled-out distilled water than in aerated water. The proportion of dis-

solved copper present in a number of Bordeaux mixtures is determined. The minimum solubility (0.00009%) is reached in a 4 : 1.08 : 50 preparation, *i.e.*, a  $\text{Cu} : \text{Ca}(\text{OH})_2$  ratio of 1 : 0.27. Addition of 0.5% of sugar to the Bordeaux mixture raised the copper solubility to 0.078%.

A. G. POLLARD.

**Determination of sulphate in chrome liquors.** O. MACCHIA (Ind. chimica, 1930, 5, 1346–1351; Chem. Zentr., 1931, i, 818–819).—The liquid (20 c.c.) is gently warmed with acetic acid (20 c.c.) and tartaric acid (3.2–3.5 g.), and then treated with hydrazine or hydroxylamine hydrochloride until effervescence is no longer produced. After dilution with water (150 c.c.) the solution is boiled and the sulphate is precipitated with barium chloride.

A. A. ELDRIDGE.

**Chlorometry. Definition of chlorometric value.** H. LESTRA (Bull. Sci. pharmacol., 1930, 37, 300–308; Chem. Zentr., 1931, i, 973).—The chlorometric value is unchanged when hydrochloric acid or chlorides are present. The chlorometric value (English) expresses in g. of chlorine the oxidising power of 100 g. of hypochlorite.

A. A. ELDRIDGE.

**Removal of hydrogen sulphide and cyanide from benzol, and of cyanide from coke-oven gas.** See II. Soda recovery in paper mills.—See V. Basic slag.—See XVI.

See also A., May, 555, System  $\text{AlCl}_3$ – $\text{NaCl}$ . 562, Silica gel. 577, Sodium and lead arsenates. 581, Aluminium hydroxide. 582, Cerium sulphide. 583, Rhenium. 588, Micro-determination of calcium and magnesium. Tartar emetic. 638, Detection of carbon disulphide.

## PATENTS.

**Manufacture of nitric acid.** LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 345,591, 27.6.30).—The vapours containing the last traces of nitric acid are passed through irrigating absorption towers and then subjected to electrical precipitation in a single- or multi-stage apparatus arranged between these towers.

H. ROYAL-DAWSON.

**Preparation of concentrated nitric acid.** N. CARO and A. R. FRANK (B.P. 344,826, 6.12.29. Cf. B.P. 342,068; B., 1931, 392).—Aluminium or an alloy containing it is used in the construction of pressure vessels etc. for use in the process of the prior patent. Such vessels may be (a) provided with a ceramic lining, (b) packed with aluminium rings etc., and (c) applied direct to supporting walls or have an intervening gas space filled with a compressed non-reacting gas.

L. A. COLES.

**Treatment of [residual] ammoniacal liquors [from the ammonia-soda process].** D. R. MEANS, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,781,987, 18.11.30. Appl., 20.9.26).—The liquor, after decomposition of any ammonium carbonate present by treatment with steam in the upper section of a tower, is conducted from an intermediate point in the tower to the lower end of a cylindrical tank in which it is treated with unground quicklime containing, *e.g.*, 5–12% of unburnt limestone. The lime, which is fed in at the top of the tank, is swept down a series of trays by a rotating

agitator, and residual unburnt stone is removed from the bottom. The treated liquor is returned from the top of the tank to the tower for recovery of the ammonia.

L. A. COLES.

**Furnaces for production of sulphates and hydrochloric acid.** H. FRISCHER (B.P. 344,801, 10.12.29).—In furnaces for this purpose, the bottom, walls, pipes, stirring and scraping devices, and other such parts as come in contact with molten sulphates resulting from the interaction of chlorides and sulphuric acid are made from silicon carbide or materials containing it.

H. ROYAL-DAWSON.

**Treatment of [production of alkali aluminates or pure alumina from] bauxite, alunite, and like aluminous material.** SULPHATES PROPRIETARY, LTD. (B.P. 344,959, 21.12.29. Austral., 22.12.28).—A mixture of the material with an alkali hydrogen sulphate or with an alkali sulphate and sulphuric acid ( $\text{Na}_2\text{O} : \text{Al}_2\text{O}_3 = 1.1-1.5:1$  mol.), worked up into briquettes or nodules, is heated in a vertical shaft furnace in the presence of a countercurrent of a reducing gas (water-gas), and the product, after removal from the furnace, is treated in thin layers with an oxidising gas and then lixiviated to extract the alkali aluminate.

L. A. COLES.

**Manufacture of high-grade zinc oxide.** F. E. PIERCE (U.S.P. 1,781,702, 18.11.30. Appl., 9.7.21. Renewed 30.3.28).—Zinc vapour produced by smelting zinc ores or the crude oxide in a reverberatory furnace is burned at 1090–1290° by admixture with preheated air in a combustion chamber connected with the furnace; the oxide, which leaves the chamber at 600–700°, is drawn by a fan through a flue or trail in which it is cooled to about 260° by the admission at intervals of cold air, and thence passes successively through a cyclone or centrifugal separator to remove impurities and gritty particles of oxide, a bag house, and a furnace in which it is reheated for about  $\frac{1}{2}$  hr. at 550–600°.

L. A. COLES.

**Manufacture of finely-divided antimony trioxide.** DEUTS. SCHMELZ-U. RAFFINIERWERKE A.-G. (B.P. 345,034, 6.2.30).—The upper surface of a stationary charge of antimony, or of a mixture of ores which on roasting will give oxides of antimony, is heated to about 1000° and the metal is burned or the ore roasted in air diluted with inert gases (carbon dioxide, nitrogen, or combustion gases).

H. ROYAL-DAWSON.

**Refining of heavy spar.** METALLGES. A.-G. (B.P. 345,186, 21.7.30. Ger., 28.8.29).—The spar, after dressing if necessary, is calcined at 1000–1200° under oxidising conditions, cooled dry, treated with hot (preferably hydrochloric) acid, and washed with water and finally with dilute ammonia solution. Alternatively, the ore is calcined to burn away any bitumen and then treated directly with the acid. L. A. COLES.

**Production of hydrogen.** F. T. SNYDER (U.S.P. 1,781,935, 18.11.30. Appl., 16.12.25. Can., 12.3.25).—Natural gases, gas mixtures containing hydrocarbon vapours, etc., and a current of air are passed alternately, but in opposite directions, through a furnace filled with refractory chequerwork so that the carbon deposited

during the cracking stage is burned during the combustion stage to maintain the chequerwork at about 980°.

L. A. COLES.

**Apparatus for decomposing water into hydrogen and oxygen gases.** S. BESSHO and T. SAKAKIBARA (B.P. 345,149, 14.5.30. Jap., 28.9.29).—Water maintained at a constant level by a float regulator in a vertical cylindrical chamber passes up a narrow annular space between two concentric axially-disposed tubes, and is vaporised therein by an electrically-heated nichrome wire in the inner tube; the vapour passes into a compartment above the cylinder in which it is decomposed by an electrically-heated platinum wire stretched around the chamber in the form of a polygon.

L. A. COLES.

**Manufacture of hydrogen peroxide.** J. B. PIERCE, JUN., ASSR. to BARIUM REDUCTION CORP. (U.S.P. 1,781,859, 18.11.30. Appl., 14.12.27).—Strontium peroxide, prepared, *e.g.*, as described in U.S.P. 1,325,043 (cf. B.P. 130,840; B., 1919, 719 A), is treated with an equivalent weight of cold, dilute sulphuric acid and a small quantity (0.2–0.1% of the wt. of  $\text{H}_2\text{SO}_4$ ) of an acid yielding a soluble strontium salt, and the strontium sulphate is removed by filtration. L. A. COLES.

**Recovery of sulphur from pyrites.** R. F. BACON (U.S.P. 1,782,225–6, 18.11.30. Appl., [A] 6.4.27, [B] 13.4.27).—(A) The pyrites is heated in a rotating furnace at 600–700° in the presence of steam, the sulphur is deposited in a condenser maintained above 100°, and the residual steam, after preheating, is returned to the furnace. Sufficient sulphur dioxide to interact with any hydrogen sulphide present is also introduced into the condenser. (B) Part of the sulphur is recovered from the pyrites as described in (A), and the remainder is removed as sulphur dioxide by heating the residues in an air blast in a separate furnace. The gaseous products from the two sets of furnaces may pass into a common condenser so that the hydrogen sulphide from the one set reacts with the sulphur dioxide from the other, or the hydrogen sulphide may be stored for treatment with sulphur dioxide as required.

L. A. COLES.

**Preparation of chlorine dioxide.** W. BECHER (B.P. 345,190, 26.8.30. Holl., 26.8.29).—The gas is prepared by the action below 65° of concentrated sulphuric acid on prepared lumps comprising chlorates and non-reacting material, *e.g.*, gypsum; a catalyst (oxide of manganese, vanadium, iron, or osmium) and a carbonate may be added to the lumps, and a non-reacting gas, which may be saturated with an organic reducing agent (formaldehyde), may be passed through the reacting mixture.

L. A. COLES.

**Apparatus for compressing carbonic acid snow.** MASCHINENFABR. ESSLINGEN, and J. STOFFELS (B.P. 347,050, 12.6.30. Ger., 20.9.29. Addn. to B.P. 334,682).

**Installations for endothermic reactions.** Sp. gr. of battery acid. Gas producer. Liquefaction of gases.—See I. Ammonia etc. from gases. Sulphur from gases.—See II. Lithopone. Titanium pigments.—See XIII. Manures etc.—See XVI. Silicyl compounds.—See XX.

## VIII.—GLASS; CERAMICS.

**Variations caused in the heating curves of glass by heat treatment.** A. Q. TOOL and C. G. EICHLIN (J. Amer. Ceram. Soc., 1931, 14, 276—308).—Heating curves have been obtained for a series of glasses which represented the progression of the change taking place during the annealing of a glass. A particular glass representative of its type (chilled) gave a heating curve which rose rapidly between 350° and 550° and then declined to a lower level at a somewhat higher temperature. These phenomena are due to an exothermic effect followed by either an endothermic effect or a rapid change in sp. heat and conductivity. Another glass (annealed) showed no exothermic effect, but exhibited a marked decline at the higher temperature, which is consequently definitely attributed to an endothermic effect. The determination and interpretation of the heating curve is discussed at length, especially in connexion with annealing. Among the many points which are noted, the importance of the following facts in practical annealing is emphasised: Properties such as density, refractive index, etc. may be varied 1% by suitable treatment in the lower part of the annealing range, and such manipulation may be utilised to correct differences in property resulting from slight variation between the compositions of different melts. Incidentally great caution is needed, in determining the effect of a change in certain constituents on a particular property, that the same annealing schedule is followed. To produce a homogeneous product temperature gradients should not exist during the annealing process.

J. A. SUGDEN.

**Expansion measurements of several glasses by means of a self-registering apparatus.** W. M. COHN (J. Amer. Ceram. Soc., 1931, 14, 265—275).—The apparatus (which is described) records photographically the expansion relative to vitreous silica, and reveals clearly certain inflexion points in the temperature-expansion curve which are liable to be undetected by visual observation. Although one glass (a special apparatus glass) showed no inflexion point and another (a tube glass) two points, most glasses showed only one. The annealing range of a glass is defined by the softening and inflexion points. Complete analyses of the glasses are given.

J. A. SUGDEN.

**Electrochemistry applied to glass.** A. SILVERMAN (Trans. Amer. Electrochem. Soc., 1931, 59, 183—191).—Applications of electrochemistry in the glass industry include chromium-plating of moulds, plating on glass for decorative purposes, and conductivity determinations as a measure of the viscosity of glass. The results of investigations on electrolytic conduction and ionic migration through glass are reviewed.

H. J. T. ELLINGHAM.

**Comparison of bodies containing blended feldspars and one-mine feldspar of similar composition.** C. W. PARMELEE and C. R. AMBERG (J. Amer. Ceram. Soc., 1931, 14, 309—312).—An electrical porcelain body was made up with single and blended feldspars of similar composition and fired at cones 7—13. Measurement of the volume shrinkage and modulus of rupture showed that single and blended feldspars were not the same in effect. This is due to variations in the soda-

lime ratio of the plagioclase and the degree of weathering. It is recommended that blending be restricted to feldspars from the same mine.

J. A. SUGDEN.

**Tshasov-Jar clays.** K. KÖHLER (Trans. Ceram. Res. Inst., Moscow, 1929, No. 20, 88—150).—The characteristics of the clay from the point of view of its use in the ceramic industry have been investigated. A rational classification of clays is proposed.

CHEMICAL ABSTRACTS.

**Use in ceramic industry of the sandy Tshasov-Jar clay called "balyki."** P. N. KAVOKIN (Trans. Ceram. Res. Inst., Moscow, 1929, No. 20, 172—181).—The use of this clay, previously discarded, for the manufacture of stoneware and firebrick is described.

CHEMICAL ABSTRACTS.

**Influence of high temperature on Prosyanyaya kaolin.** V. V. YURGANOV and M. V. ZUSMANOVICH (Trans. Ceram. Res. Inst., Moscow, 1929, No. 21, 28—55).—At 880° metakaolin is formed; at 980° kaolin decomposes into free silica and the sparingly soluble form of clay, and shows the first sign of formation of one or more aluminium silicates. At 1050—1100° there is little change except further combination of free silica and alumina. No sillimanite was found. At 1200° the sparingly soluble residue,  $5\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ , increases; at 1320° its composition is  $4\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ . Kaolin burned at 1400—1470° ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ), when examined in thin layers, exhibits crystals.

CHEMICAL ABSTRACTS.

**Weathering of plastic fireclays.** S. M. PHELPS and C. G. DENNEY (J. Amer. Ceram. Soc., 1931, 14, 319—324).—Extensive tests were made on clays stored in the laboratory, on the same clays after 2—3 years' weathering on the roof of a building, and on commercially weathered clays. The properties studied were water of plasticity, plasticity, drying and firing shrinkages, refractoriness, loss on ignition, transverse strength, sieve analysis, and rate of settling. Weathering appeared to have no appreciable effect. It is concluded that either there is no satisfactory method of measuring the changes or that the weathering effect is over-estimated and that it can be reproduced to a large degree by finer grinding and treatment with electrolytes.

J. A. SUGDEN.

**Currents and temperatures in glass tanks.** I. G. GEHLHOFF, W. SCHNEEKLOTH, and M. THOMAS (J. Soc. Glass Tech., 1931, 15, 12—29 T).

Silicon carbide.—See I.

See also A., May, 546, Glass for ultra-violet rays. 579, Coloration of silicate glasses. 593, Filtering discs. 595, Minerals of the clay group.

PATENTS.

**Making sheet glass.** MISSISSIPPI GLASS CO., Assees. of M. L. BEGEMAN (B.P. 346,927, 5.3.30. U.S., 21.5.29).

**Apparatus for working fused silica.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of G. A. MILLAR (B.P. 346,940, 13.3.30. U.S., 13.3.29).

**Preparation [drying etc.] of ceramic articles.** W. and M. LENGERSDORFF (B.P. 347,020, 9.5.30. Addn. to B.P. 328,777).

**Treatment of laminated materials.**—See I. [Adhesive] compositions.—See XIII.

## IX.—BUILDING MATERIALS.

**Suitability of tar oils for the impregnation of mine timber.** W. ENGELS (Chem.-Ztg., 1931, 55, 285—286, 307—308).—A comparison is made between tar oils and a number of preparations consisting of salt solutions possessing germicidal action. Besides having certain disadvantages such as objectionable odour, action on the skin, and risk of fire, tar oils, which depend for their action mainly on their tar acid content, afford protection to the wood for only a limited period compared with the salt solutions. F. R. ENNOS.

## PATENTS.

**Rotary cement kiln.** M. VOGEL-JORGENSEN (B.P. 345,731, 21.1.30).—The total inside area of a rotary dry cement kiln is at least 120 times as large as the internal cross-sectional area of the kiln at normal diameter, and for a wet kiln this figure excludes the drying section of the kiln. The length of the kiln may be adjusted, maintaining the normal diameter in the preheating and calcining zones, which preferably are fitted with some form of agitator. The sintering zone may be enlarged and the preheating and calcining sections split up into a number of tubes to effect better contact of the material with the hot gases.

C. A. KING.

**Production of cementitious material.** G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 345,145, 24.12.29. Cf. B.P. 330,278; B., 1930, 770).—A puzzuolanic cement is produced by mixing 25% of calcium (and/or magnesium) oxide with 75% of the product obtained by heating an aluminium silicate, *e.g.*, slate, clays, or shale, at about 750°. Calcium sulphate (5—15%) may be incorporated as an accelerator. C. A. KING.

**Composition for waterproofing cement.** F. W. HUBER (U.S.P. 1,772,999, 12.8.30. Appl., 7.8.28).—High early strength and proofing of cement is attained by mixing the cement batch with an emulsion containing an oil (petroleum) as the external phase and an accelerating solution of an alkali or alkaline-earth salt, or an acid which will produce such a salt by reaction.

C. A. KING.

**Surface treatment of concrete or other materials pervious to moisture.** E. O. COWPER (B.P. 345,671, 18.12.29 and 24.5.30).—A mixture of a dilute emulsion of rubber latex (with preservative, if desired) and dilute sodium silicate solution is sprayed on to concrete, brick, etc. and penetrates before the latex coagulates. Approximately equal quantities of emulsion and solution are used, the degree of dilution being never less than 2:1 in the case of either ingredient (latex or silicate).

C. A. KING.

**Manufacture of porous building materials.** A. V. CARLSON (U.S.P. 1,782,460, 25.11.30. Appl., 6.8.26. Swed., 19.8.25).—A mixture comprising acidic material (pumice, argillaceous schist, hydraulic residues, cements, etc.), sufficient unslaked lime to give a foundation mass, and material for evolving gases (powdered metals or carbides, and, if desired, alkalis) is hydrated by the addition of water or steam, and the product is moulded and then hardened by treatment with hot air etc.

L. A. COLES.

**Colouring of slate granules.** H. C. FISHER, Assr. to PHILIP CAREY MANUFG. CO. (U.S.P. 1,782,648, 25.11.30. Appl., 29.4.25).—The granules are wetted with a solution containing compounds which form hydroxides and then oxides on roasting, *e.g.*, with a solution containing ferrous sulphate (6 mols.) and sodium dichromate (1 mol.), which gives a brown colour on roasting. The colour may be modified by varying the proportion of the compounds and the oxidising or reducing properties of the roasting gases.

L. A. COLES.

**Making of cold tar road coverings.** D. J. PICKÉE (B.P. 346,025, 30.12.29. Ger., 23.8.29).—Tarry substances are prepared for cold application to road surfaces by admixture with hydrated lime. The lime is preferably used in aqueous solution or suspension, and its weight should be 10—12% of that of the asphaltic material. The viscosity of the mixture may be reduced by adding a solvent such as benzene. Dry lime may also be added to the tar, and the necessary water then stirred in or added on the road surface.

R. H. GRIFFITH.

**Cold bituminous paving composition.** O. H. BERGER, Assr. to W. P. McDONALD CONSTRUCTION CO. (U.S.P. 1,776,763, 23.9.30. Appl., 14.7.28).—A hard asphalt containing 60% or more of bitumen, *e.g.*, raw Cuban asphalt, is mixed with a "fresh oil" (petroleum containing a fair proportion of volatile oil) and then with a mineral base. Finally 3—5% of water and "fresh oil" together are incorporated until the mixture is light brown in colour.

C. A. KING.

**Plant for manufacture of artificial wood.** O. KAYSER (B.P. 345,191, 10.6.30).—A conveyor belt is led under the lower part of a filter drum, and a closed conduit supplies wood pulp, preferably tangentially, between the filter and the band.

C. A. KING.

**Preservation of wood.** D. STEINIERZ, Assr. to GRUBENHOLZIMPRÄGNERUNG GES.M.B.H. (U.S.P. 1,777,235, 30.9.30. Appl., 27.6.29. Ger., 3.7.28).—Wood is impregnated with a water-soluble substance and then with a separate solution which will reduce the solubility of the first salt. Thus a 2% solution of sodium fluosilicate or an aqueous solution of sodium 2-naphthalenesulphonate may be followed by a 4% solution of sodium chloride.

C. A. KING.

**[Rubber] floor coverings.** N. KENYON, and SPEN RUBBER WORKS, LTD. (B.P. 344,956, 19.12.29).—In a floor covering comprising a layer of rubber united by vulcanisation with a backing consisting of a vulcanised mixture of rubber and wood flour, integral portions of the backing extend through the rubber layer and form part of the tread surface.

D. F. TWISS.

**Furnace. Separation of granular etc. materials.**—See I.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Influence of oxygen and sulphur on the malleability, hot-shortness, and other properties of pure iron.** E. W. FELL (Arch. Eisenhüttenw., 1930—1, 4, 393—400; Stahl u. Eisen, 1931, 51, 386—387).—Armco iron containing up to 0.07% O can be readily hot-forged



after melting *in vacuo*, but the presence of 0.01–0.02% S considerably reduces the malleability at a red heat; with 0.03% O and 0.03% S the metal is definitely hot-short. After melting in an oil-fired furnace, however, iron relatively rich in sulphur and oxygen forges well if the treatment is commenced at a high temperature, *e.g.*, 1200°. Alternate bending tests at 1100–1150° of square rods on the anvil showed the marked superiority of vacuum-melted metal over metal melted in an oil-fired furnace. Hot-shortness above 800° is shown by iron containing more than 0.03% O and 0.02% S; thus metal with 0.054% O and 0.01% S is distinctly hot-short in the bending test although it forges well. Notched-bar impact tests confirmed the deleterious effect of a combined oxygen and sulphur content above about 0.04%. The depth of penetration of the carbide layer in the cementation test is highest with pure iron and decreases with increasing oxygen content.

A. R. POWELL.

**“Through improvement” of constructional steels with reference to the effect of cross-sectional area and alloying constituents.** H. KALLEN and H. SCHRADER (Arch. Eisenhüttenw., 1930—1, 4, 383–392; Stahl u. Eisen, 1931, 51, 387).—The term “through improvement” (Durchvergütung) is applied to that process of treating a steel which produces a structure such that the yield point is constant throughout the cross-section of the specimen. The “state of improvement” (Vergütungszustand) of a steel is characterised by the relation between the yield point and tensile strength, and is determined by the proportion that the area of improved structure bears to the total area of the cross-section of the specimen. Numerous technical steels in specimens 20–200 mm. in diam. have been tested to determine their “state of improvement” and the degree of “through improvement,” and from the results obtained the various technical steel alloys are divided into five classes. Alloy steels with a high nickel and chromium content are in the most satisfactory condition whatever their cross-sectional area, and plain carbon steels are usually the least uniform in their mechanical properties.

A. R. POWELL.

**Indirect determination of silicon in 48–52% ferrosilicons.** G. T. DOUGHERTY (Ind. Eng. Chem. [Anal.], 1931, 3, 158–159).—Practical details are given of a rapid method of determining silicon in ferrosilicons by difference instead of directly.

E. S. HEDGES.

**Cause of the “iron disintegration” of blast-furnace slags.** A. GUTTMANN and F. GILLE (Arch. Eisenhüttenw., 1930—1, 4, 401–410; Stahl u. Eisen, 1931, 51, 432–433).—The so-called “iron disintegration” of slags is due to the presence of finely-divided iron and manganese sulphides in the slag; these sulphides in contact with moist air become hydrated and increase in volume by about 38%, thus causing the slag to break up into scales and crusts on the surface. This action occurs only when the slag contains at least 1.5% FeO and 0.5% S and when it has been left in contact with the molten iron in the settler or mixer for a long time. To prevent such slags from disintegrating they may be heated in air at above 1000° or, better, treated with sand before they solidify.

A. R. POWELL.

**Rapid volumetric determination of manganese in ores and alloys.** I. MAJDEL (Arh. Hemiju, 1931, 5, 45–49).—The finely-powdered material (1 g.) is fused with 6 g. of sodium peroxide in an iron crucible, the cold mass extracted with warm water, and the solution, without filtering, boiled for 5–10 min. with, if necessary, the addition of a few drops of alcohol to destroy manganate and permanganate; 50 c.c. of 0.1N-oxalic acid and 50 c.c. of 1:5 sulphuric acid are added to the hot liquid to dissolve the ferric hydroxide and manganese dioxide, followed by 10 c.c. of phosphoric acid (*d* 1.7) to decolorise the iron salt, and the excess of oxalic acid is titrated with permanganate.

A. R. POWELL.

**Removal of antimony from white metals.** R. THEWS (Metallbörse, 1930, 20, 1545–1546; Chem. Zentr., 1930, ii, 2434).—Removal by addition of sodium, aluminium, or zinc, which form alloys or mixtures with the antimony, is discussed. Iron and magnesium are unsuitable.

A. A. ELDRIDGE.

**Rapid test of corrosion-resistance.** L. W. HAASE (Chem. Fabr., 1931, 169–170, 184–185).—Existing methods are criticised, mainly because they take no account of the oxygen content of the corrosive fluid and do not continuously renew the fluid. In the electro-metric method described the fluid, at constant head, is fed in succession through two vessels, each of which contains the material under test and a platinum electrode; the liquid entering the second vessel is previously saturated with oxygen. The electrical apparatus is so arranged that each pair of electrodes is connected either to the measuring instrument or across a high resistance; it is thus possible to compare the corrosion-resistance of a material towards two different liquids or of two different materials towards one liquid. With a suitable choice of the velocity of flow of the liquid comparative results may usually be obtained within 24 hrs. The rate of formation of a protective layer may also be determined. The applicability of the method to materials of the same chemical composition, but different in structure, *e.g.*, alloys before and after heat treatment, and the interpretation of the results obtained, are described.

H. F. GILLBE.

**Corrosion of the tinplate container by food products.** T. N. MORRIS and J. M. BRYAN (Dept. Sci. Ind. Res., Food Invest., Spec. Rept., 1931, No. 40, 85 pp.).—The previous literature on corrosion is reviewed and the apparatus used in this investigation is described. The condition of the metal surface is shown to be important. A large number of tests are described showing the effect of  $p_H$  and of oxygen on the corrosion of tin, iron, and tinplate, from which it is concluded that low acidity leads to greatest corrosion of tin, and the presence of air especially at high  $p_H$  induces corrosion of iron. Sugars, colloids, and tin salts are good inhibitors of corrosion, but traces of sulphur compounds act as accelerators. The discoloration of foodstuffs by tin and iron salts is discussed and conditions favourable to the production of hydrogen “swells” and perforations are described with suggested remedial measures. The report closes with an appendix on the examination of canned foods and an extensive bibliography.

E. B. HUGHES.

**Studying the stress-strain relation in the notched-bar impact test.** T. KAWAI (Sci. Rep. Tôhoku, 1930, 19, 727—743).—A Charpy machine is used to apply a small impact repeatedly to a test-piece and the bending at each impact is measured until the test-piece is broken. From these observations the energy-bending relation in the test is calculated and the stress-bending relation obtained therefrom by evaluating the tangent at each point on the curve representing the former relationship.

W. E. DOWNEY.

**Effect of cold-working on the density and electrical resistance of metals.** T. UEDA (Sci. Rep. Tôhoku, 1930, 19, 473—498).—The change in the density and electrical resistance of Swedish steel, copper, and brass, when stretched by a tensile-testing machine have been measured. The density of steels always decreased with an increasing tension, but a discontinuous decrease occurred in low-carbon steels at the yield point. The sp. electrical resistance always increased with the tension, at the yield point a discontinuous increase in the case of low-carbon steels and a decrease of resistance in that of medium- and high-carbon steels being observed. In the case of copper and brass the decrease of density and increase of sp. electrical resistance increased in magnitude with tension; in this latter case, however, discontinuity was not observed at the yield points as in the case of carbon steels.

W. E. DOWNEY.

**Electrolytic refining of aluminium and production of aluminium coatings in an aluminium chloride-sodium chloride bath.** W. A. PLOTNIKOV, N. S. FORTUNATOV, and W. P. MASCHOWETZ (Z. Elektrochem., 1931, 37, 83—88).—Electrolysis of mixed aluminium and ammonium chlorides at 170° yields a crystalline deposit of the metal, which, however, reacts with the bath. Aluminium containing Fe 0.043—0.060%, Si 0.023%, and Na 6.015% may be obtained by electrolysis in a bath containing 1.5—2 mols. of sodium chloride per mol. of aluminium chloride at 160—180° with an aluminium cathode. The deposit is crystalline and spongy, and is not improved by variation of the temperature nor by the use of a rotating cathode. Addition of alkaline-earth chlorides merely raises the m.p. and reduces the conductivity of the bath without altering the nature of the deposit; no improvement results from replacement of sodium chloride by potassium chloride, or from addition of a variety of other substances. The presence of lead chloride aids the formation of a coherent deposit, which, however, always contains lead. On copper and iron cathodes coherent deposits are readily obtainable; a 0.04—0.05-mm. coating may be obtained by electrolysis at 160° with a current density of 1—1.5 amp./dm.<sup>2</sup> The chemical inertness of the refined aluminium and the high corrosion resistance of the aluminium coatings are noted. Corrosion in moist air ceases completely when a layer of aluminium 0.003 mm. thick has become oxidised.

H. F. GILLBE.

**Chromium deposits directly on aluminium.** H. K. WORK and C. J. SLUNDER (Trans. Amer. Electrochem. Soc., 1931, 59, 175—181).—Chromium can be satisfactorily deposited directly on aluminium and its alloys from a plating bath of the ordinary type if the surface has been suitably cleaned and etched. The

nature of these preliminary processes to be used under various conditions is described. The best current density for chromium deposition depends on the nature of the work, but for thin deposits from a solution at 45° usually exceeds 21.5 amp./dm.<sup>2</sup> At lower temperatures the deposit is darker in colour, but is more easily buffed to a high lustre. For thick deposits, however, good adhesion is obtained by working at 60° and 40 amp./dm.<sup>2</sup> The thin deposits have a fairly good resistance to outdoor exposure and to the salt spray, and thicker plates afford considerable protection against strong alkaline solutions. Deposits 0.025—0.1 mm. thick on an alloy containing 4% Cu, 0.5% Mn, and 0.5% Mg showed good resistance to abrasion, and pistons so plated have behaved well in tests.

H. J. T. ELLINGHAM.

**Independence of the hardness of electrolytic metals of their content of hydrogen.** GUICHARD, CLAUSMANN, BILLON, and LANTHONY (Compt. rend., 1931, 192, 623—625; cf. Hugues, B., 1926, 159).—Samples of electrolytic iron and cobalt, respectively, lose all their hydrogen when heated at 250° and 450°, with no and very slight loss of hardness. Heating to higher temperatures causes a steady decrease in hardness. It is concluded that the hardness of electrolytic metal is in no way due to the presence of hydrogen, but results solely from their very fine structure.

C. A. SILBERRAD.

**Progress of the hot nickel[-plating] solution.** O. P. WATTS (Trans. Amer. Electrochem. Soc., 1931, 59, 193—197).—Information regarding composition and *pH* value of baths, temperature, current density, material for tanks and linings, nature of work plated, and quantity of solution maintained has been supplied by a number of firms, mostly in the U.S.A., which have adopted the hot nickel-plating process recommended by the author in 1916. Most of the baths consist of nickel sulphate and chloride with addition of boric acid, but the *pH* values reported range from 2.2 to 6.5, temperatures from 32° to 71°, and current densities from 10 to 100 amp./ft.<sup>2</sup> Comments by platers on the respective merits of hot and cold baths are also reported.

H. J. T. ELLINGHAM.

**Electrodeposition of cobalt-nickel alloys. II.** S. GLASSTONE and J. C. SPEAKMAN (Trans. Faraday Soc., 1931, 27, 29—35; cf. B., 1930, 1074).—The investigation of the compositions of alloys deposited from well-buffered solutions of definite hydrogen-ion concentration containing various mixtures of cobalt and nickel sulphates at a series of current densities has been extended to 50° and 95°. The relative tendency for cobalt and nickel to deposit is independent of the hydrogen-ion concentration. The alloys deposited at very low current densities contain decreasing proportions of cobalt as the temperature is raised, and this proportion is, in general, less than that in the solution for depositions at 90°. With increasing current density the proportion of cobalt in the alloy increases rapidly up to a maximum, independent of the current density. This maximum decreases with rise in temperature. The influence of cobalt on the deposition of nickel at 50° and at 90° is very similar to that of iron at 15° and 50°, respectively (cf. A., 1928, 851). The theoretical basis of the results

is discussed in terms of the deposition potentials and overvoltages of the metals concerned. O. J. WALKER.

**Silicon carbide. Applications of rubber.**—See I. **Low-carbon pig iron.**—See II.

See also A., May, 556, **Brasses. Systems Cu-Ag, Cd-Ag, Al-Ag, and Si-Al. Ternary silver alloys. 557, Fe-Si-C and Fe-W-C systems. 561, Colloidal silver and gold. 569, Ternary alloys. 570, Dissolution and deposition of metals. Brass. 574—5, Corrosion. 583, Rhenium. 584, Reduction of iron oxide. Reactions of iron etc. with oxides of iron. 586, Analysis of iron and steel. 588, Determination and separation of lead and bismuth. 589 and 590, Determinations of molybdenum, zirconium, and iridium. 590, Detection of gold, palladium, and silver.**

#### PATENTS.

**Drum furnace for melting metals.** C. BRACKELSBURG (B.P. 344,980, 31.12.29).—The interior of a revolving drum is divided by an annular passage into a relatively long melting chamber of conical formation and into a second chamber, preferably inclined to the axis of the drum, for mixing and superheating. A pulverised-fuel burner is situated at one end and the gases pass through both chambers, leave at the narrow end of the cone, and enter an inclined chute which also serves as a feed for metal. The melted metal overflows the constriction into the superheating chamber.

C. A. KING.

**Metal-melting pots.** H. T. THORP and W. GEDDES (B.P. 345,697, 7.1.30).—A furnace for melting metals of low m.p., e.g., lead, is divided into two intercommunicating compartments, the first, into which metal ingots are fed, being at rather a higher level so that molten metal flows into the second by gravity. The two pots are heated independently by means of oil or gas and a float on the molten metal in the delivery compartment controls the fuel supply to the ingots in relation to any predetermined level of molten metal. It is desirable to arrange the communicating passage and outlet in alinement to facilitate cleaning. C. A. KING.

**[Casting of] steel ingots.** A. G. EGLER (B.P. 345,983, 1.11.29. U.S., 9.11.28).—The mould is continuously jarred during the casting operation, thereby causing the slag and impurities to rise to the top and the depth of pipe to be considerably reduced.

A. R. POWELL.

**Casting of metals or alloys.** BRIT. & DOMINIONS FERROALLOY, LTD., and C. SYKES (B.P. 345,047, 14.2.30).—To prevent oxide inclusion in the metal, the casting of aluminium or of alloys containing iron and aluminium is carried out in an atmosphere composed chiefly of carbon and chlorine or of a chlorinated hydrocarbon (e.g., trichloroethylene or chlorinated pitch).

H. ROYAL-DAWSON.

**[Inhibitors for the] pickling of metals and like processes.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,162, 4.4.30).—Acetaldehyde derivatives of the general formula  $R'CH:CR\cdot CHO$ , in which R and R' are the same or different alkyl groups, are claimed, specifically  $\alpha$ -ethyl- $\beta$ -propylacetaldehyde. The addition of sulphite-cellulose waste and sodium hydrogen sulphate to the

pickling bath prevents the disengagement of objectionable spray.

A. R. POWELL.

**Treatment of ferromagnetic alloys to obtain enhanced magnetic properties.** VEREIN. STAHLWERKE A.-G. (B.P. 346,013, 23.9.29. Ger., 24.10.28).—Iron alloys containing a constituent the solubility of which is greater at high than at ordinary temperatures are quenched from a temperature above that at which the solid solubility commences to increase and below that at which a duplex structure appears, i.e., in the homogeneous  $\alpha$ -range, and subsequently reheated at temperatures below the quenching temperature. [Stat. ref.]

A. R. POWELL.

**Electrodeposition of platinum metals.** (A) C. W. KEITEL and (B) H. E. ZSCHIEGNER, ASSTS. to BAKER & CO., INC. (U.S.P. 1,779,436 and 1,779,457, 28.10.30. Appl., [A] 2.7.29, [B] 7.10.27).—(A) For the electrodeposition of platinum, rhodium, or palladium the bath consists of a 10% solution of ammonium sulphate or nitrate containing 5% of free ammonia and 2% of the corresponding diammino-platinum metal nitrite. The bath is operated at 95° with 2.2 volts and its composition is maintained by adding ammonia and the nitrite as required. (B) The bath is prepared by boiling the platinum-metal chloride with sodium nitrite until the solution is colourless or pale yellow and then adding an excess of ammonia.

A. R. POWELL.

**Improving copper or [silicon-iron]-copper alloys and hardener for use therein.** U. DE BERKER, P.M.G. METAL TRUST, LTD., W. MACHIN, and W. B. O'B. GOUDIELOCK (B.P. 347,098, 12.12.29).—See U.S.P. 1,777,192; B., 1931, 353.

**[Apparatus for] metallising by the melting and projection of metals.** SOC. NOUVELLE DE METALLISATION (B.P. 346,656, 9.12.29. Fr., 7.12.28).

**Processing of furnace dust. Heat removal by mercury. Installations for endothermic reactions.**—See I. **Refining furnaces. Welding.**—See XI.

#### XI.—ELECTROTECHNICS.

**Decomposition of iron oxide in vacuum tubes.** E. R. WAGNER (Trans. Amer. Electrochem. Soc., 1931, 59, 205—207).—In studying the possibility of using superficially oxidised iron as anode in a vacuum tube it was found that with a gradient of 300 volts/cm. between the plate and the filament the black surface layer was gradually reduced to metallic iron and gas accumulated in the tube. The amount of gas, however, was far less than that corresponding to the oxygen in the oxide layer. On increasing the voltage gradient a large amount of gas is suddenly released, as though it had accumulated during operation at the lower voltage, but remained occluded in the plate. It is concluded that the observed phenomena are due to electron bombardment rather than to electrolysis.

H. J. T. ELLINGHAM.

**Silicon carbide. Applications of rubber.**—See I. **Oxidation of ferrocyanide.**—See VII. **Electrochemistry and glass.**—See VIII. **Test for corrosion resistance. Resistance of metals. Aluminium. Chromium deposits on aluminium. Hardness**

of electrolytic metals. Nickel-plating. Cobalt-nickel alloys.—See X. Conductivity of molasses.—See XVII. Nitrogenous matter in milk.  $p_H$  of cheese whey.—See XIX.

See also A., May, 546, Glasses for ultra-violet rays. 555, System  $AlCl_3$ -NaCl. 569, Ternary alloys. 570, Dissolution and deposition of metals. 577, Sodium and lead arsenates. 578, Reduction of carboxylic acids. 593, Colorimeters etc.

## PATENTS.

**Electric [metal]-refining furnace.** T. F. BAILY (U.S.P. 1,778,578, 14.10.30. Appl., 28.1.28).—A resistance furnace comprises a vertical refining chamber of uniform cross-section, a top electrode movable throughout the height of the chamber, and a bottom electrode; the cross-sectional dimensions of the electrodes are about one half that of the molten slag resistor column extending between the electrodes and having a height at least three times its transverse dimension.

J. S. G. THOMAS.

**Electric induction furnaces [for refining metals].** D. F. CAMPBELL, and ELECTRIC FURNACE CO., LTD. (B.P. 345,635, 25.11.29).—Slag is heated from above by radiation from conducting material, *e.g.*, of carbon or metal, forming a secondary circuit for a helical primary coil extending to the level of the conducting body and surrounding the furnace lining.

J. S. G. THOMAS.

**Electric electrode furnaces.** E. BORNAND and H. A. SCHLAEPFER (B.P. 345,082, 11.3.30. Fr., 27.3.29).—A refractory plunger surrounding the electrodes and adapted to be lowered into, and to mix, molten metal contained in the furnace is claimed.

J. S. G. THOMAS.

**Manufacture of electric heating elements.** K. NOGUCHI, Assr. to MITSUBISHI TOSEN KABUSHIKI KAISHA (U.S.P. 1,779,822, 28.10.30. Appl., 25.6.28. Jap., 31.10.27).—The space between a heating element and a surrounding metallic sheath is packed with a powdered mixture of aluminium (2 pts.) and lime (1 pt.) to which water is added, whereby alumina is produced.

J. S. G. THOMAS.

**Electrode [for welding etc.].** T. S. FULLER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,778,668, 14.10.30. Appl., 30.6.27).—An electrode, containing 96% Cu, 0.8% Si, and 3.2% Ni, having hardness (Brinell) 125–175 and sp. resistance about 3–4 microhms/cm.<sup>2</sup>, is claimed.

J. S. G. THOMAS.

**Unilateral conductor for rectifying alternating currents.** H. G. ANDRÉ (U.S.P. 1,780,057, 28.10.30. Appl., 29.10.26).—A film-forming metallic cathode, *e.g.*, of nickel-silicon alloy (23% Si), wound with a helix, *e.g.*, of silver or other metal forming a conducting oxide, is immersed in sulphuric acid ( $d$  1.8), and alternating current passed between the cathode and helix. On removal from the acid the rectifier is dried or baked to remove excess acid.

J. S. G. THOMAS.

**Photoelectric cells.** WESTINGHOUSE LAMP CO., Assocs. of H. C. RENTSCHLER and D. E. HENRY (B.P. 345,043, 11.2.30. U.S., 28.2.29).—Metallic caesium is produced in the cell by reduction of caesium dichromate with silicon. Excess of alkali metal is fixed as a substantially inert compound by combination with a getter

forming stable solid compounds therewith. Thus, *e.g.*, part of the envelope of the cell is formed of lead glass, which, on heating, cleans up excess caesium.

J. S. G. THOMAS.

**[Filling for] photoelectric cells.** F. A. LINDEMANN (B.P. 345,242, 18.12.29).—In photoelectric cells comprising a sensitised cathode, *e.g.*, a unimolecular caesium cathode, the necessary operating voltage is reduced and the sensitivity increased by employing a filling consisting of krypton and/or xenon.

J. S. G. THOMAS.

**Photoelectric cells.** M. A. E. PRESSLER (B.P. 345,189, 29.7.30. Ger., 29.7.29).—Cells for television purposes are filled with xenon and/or krypton.

H. ROYAL-DAWSON.

**Manufacture of photoelectric cathodes.** GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 345,375, 6.1.30).—The supply of electropositive metallic vapour, *e.g.*, caesium, to a photoelectric cell heated considerably above 50°, *e.g.*, at 200°, is controlled by a constriction, and is stopped when the space current in the cell decreases rapidly.

J. S. G. THOMAS.

**[Electrodeless] electric-discharge devices [containing condensable vapour].** BRIT. THOMSON-HOUSTON CO., LTD., Assocs. of T. E. FOULKE (B.P. 344,906, 3.12.29. U.S., 3.12.28).—An electrodeless discharge is maintained in a vessel containing mercury vapour, part of which is continuously condensed in a condensing chamber, so that the vapour pressure in the discharge vessel is maintained below that at which a high-pressure discharge occurs.

J. S. G. THOMAS.

**Direct-reading [photochemical] photometer.** S. WEIN, Assr. to RADIOVISION CORP. (U.S.P. 1,779,574, 28.10.30. Appl., 8.11.29).—A portable photometer comprising a photochemical cell, *e.g.*, a cell consisting of two platinum electrodes immersed in an acidulated solution containing potassium iodide and ferric chloride, and an indicating device, *e.g.*, a micro-ammeter, connected with the electrodes, is claimed.

J. S. G. THOMAS.

**Chemical apparatus [for determining the hydrogen-ion concentration of solutions].** S. L. HANDFORTH, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,779,232, 21.10.30. Appl., 29.6.29).—Electrode potential apparatus, comprising a standard calomel cell and an electrode cell separated by a bridge cell containing a buffer solution, is claimed. Electrical connexion between solutions contained in the bridge cell and the other two cells, respectively, is effected by means of porous plugs fused into tubes sealed into partitions between the cells.

J. S. G. THOMAS.

**Polarising electrolyte.** E. BANNING (B.P. 344,776, 9.9.29).—A solution of a higher polysaccharide, *e.g.*, gum arabic, in glycerin or water is claimed.

J. S. G. THOMAS.

**Electric arc furnace [for melting ceramic materials, *e.g.*, mullite].** W. E. EVANS. From VITREFRAX CORP. (B.P. 345,236, 10.12.29).—See U.S.P. 1,747,756; B., 1930, 672.

**Generation of alternating electric currents for use in induction furnaces etc.** N. R. DAVIS, C. R. BURCH, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. B.P. 347,248, 20.1.30).

**Electric welding [of tubes of different degrees of hardness].** G. KALSCHNE, ASST. to GEN. ELECTRIC Co. (U.S.P. 1,779,278, 21.10.30. Appl., 15.9.27. Ger., 30.9.26).

**Sealing of glass to metal [for vacuum-tube apparatus].** BRIT. THOMSON-HOUSTON Co., LTD., and W. N. MISCHLER (B.P. 346,746, 13.12.29. U.S., 24.12.28).

**Gas producer. Viscosimeter. Sp. gr. of battery acid.**—See I. **Treatment of hydrocarbons. Insulating oil. Testing hydrocarbons.**—See II. **Antioxidant [for oils].**—See III. **Nitric acid. Decomposition of water.**—See VII. **Ferromagnetic alloys. Platinum deposits.**—See X. **Concentration of latex.**—See XIV.

## XII.—FATS; OILS; WAXES.

**Splitting of oils and fats nearly to completion.** G. PETROV and A. PICHUGINA (Masloboino Zhir. Delo, 1929, No. 9, 53—56).—Hydrolysis (97·87—99·6%) was effected by the use of naphthalenesulphonic acids (0·9—1% of the oil) in two stages.

### CHEMICAL ABSTRACTS.

**Lecithin retards hydrolysis of fats.** R. B. TRUSLER (Oil & Fat Ind., 1931, 8, 141—143, 157).—Examination of the Twitchell saponification indicates that the resistance to hydrolysis evinced by certain seed oils, cod-liver oil, etc. is attributable to the presence of lecithin or related substances; these impurities are readily inactivated by treatment with hydrochloric or sulphuric acid (20%) prior to saponification. E. LEWKOWITSCH.

**Some titer points of mixed fatty acids. I. Mixtures of commercial oils, fats, and fatty acids.** G. W. JENNINGS (Ind. Eng. Chem., 1931, 23, 413—415).—The titers of a number of binary mixtures of commercial fatty acids have been determined and the results plotted against the composition of the mixture. In the case of the mixed fatty acids of coconut oil and red oil the curve is practically identical with the straight line joining the values for the individual components; otherwise smooth curves a little above or below the theoretical lines are obtained. Red oil mixed with cottonseed oil fatty acids shows a marked irregularity at 25—30% content of the latter. E. LEWKOWITSCH.

**Preparation and properties of highly purified oleic acid.** J. H. SKELLON (J.S.C.I., 1931, 50, 131—134 r).—Existing methods of preparation of pure oleic acid have been investigated and two modified processes formulated, by which a highly purified oleic acid can be obtained from the crude acids of olive oil. One of these methods is based on Twitchell's lead salt-alcohol process and a modification of the method of Armstrong and Hilditch. The crude acids from olive oil are treated with sufficient lead acetate to combine with 23·5% of total fatty acids in a volume of 8 times their weight of boiling alcohol, which results in a quantitative separation of solid from liquid lead salts. The recovered liquid acids, dissolved in 5 times their weight of absolute alcohol, are neutralised with a boiling solution of lithium hydroxide containing 0·15 g. of base per gram of acid. The lithium salts are separated, dried, and recrystallised from a solution in 4 times their weight of alcohol. The recovered acid is further purified by fractional

distillation of the methyl ester in a vacuum. In the second method the liquid acids obtained by the above modified Twitchell process are converted into barium salts, in a solution of 12 times their weight of moist benzene and ethyl alcohol. The solution is quickly cooled, and the crystallised barium oleate is further purified from a mixture of 3 times its weight of moist benzene and alcohol. The properties of highly purified oleic acid have been investigated and criteria of purity established.

**Evaluation of stearine pitch.** G. SIEBERT and E. BLENNEMANN (Farben-Ztg., 1931, 36, 1090).—A general account is given of the properties of stearine pitch, the need for evaluation from technical as well as purely chemical aspects being stressed. Details are given of the classification of the pitch into hard, medium, and soft grades, according to flow characteristics. Both stearine pitch and wool-fat pitch give an acraldehyde test, but the former is completely soluble in hot alcoholic potash, whereas the latter gives a residue from which a fatty acid (m.p. 80—82°) may be isolated. Relatively high saponif. value of the pitch and acid value of distillation product therefrom distinguish stearine pitch from petroleum pitches. Tests for solubility in solvent naphtha, drying time, and general film properties are also described. S. S. WOOLF.

**Determination of the oil content of seeds in series.** A. LEBEDIANTZEV and N. DMITRIEV (Masloboino Zhir. Delo, 1929, No. 10, 3—10, No. 11, 3—9, No. 12, 9—17).—If the material is very finely powdered, extraction is complete in 6 hrs. at 30 siphonings per hr. with ether free from alcohol and dried over calcium chloride. Drying oils must be dried below 60° in nitrogen or carbon dioxide. The material should be dried or mixed with calcined calcium sulphate.

### CHEMICAL ABSTRACTS.

**Application of certain research methods of colloid chemistry to the drying of linseed oil.** H. FREUNDLICH and H. W. ALBU (Z. angew. Chem., 1931, 44, 56—60).—Linseed oil, with and without driers, and bodied oils prepared from them by blowing (up to 300 hrs. at ordinary temperature) or by heating in air or carbon dioxide (at 250°) were examined. Differences in the "depolarisation" of the various oils were very small and non-characteristic. The oils were tested in a Couette torsion viscosimeter, but no deviations from normal behaviour could be observed, even in the case of blown oils of high viscosity (e.g., absolute  $\eta = 82$ ). In confirmation of these results, ultramicroscopical examination (in conjunction with E. HAUSER), using a Spierer condenser (cf. B., 1930, 844), revealed no trace of colloidal structure. The changes produced in the oil by the various treatments appear to be entirely chemical in nature, but the possibility is not excluded that colloid processes may play a greater part at a more advanced stage of drying (or in wood-oil drying).

### E. LEWKOWITSCH.

**Seeds and oil of *Sinapis dissecta*, Lag.** N. BELIAIEV (Masloboino Zhir. Delo, 1929, No. 6, 25—26).—The seeds of this variety of *S. alba*, L., contain moisture 8·80, oil 28·95, fibre 15·06, ash 4·48, total nitrogen 3·83, crude protein 23·90, essential oil 0·09%. The oil

has  $d$  0.9150, f.p. 15°,  $n_D^{20}$  1.471, saponif. value 172.4, iodine value (Hübl) 100.52, acid value 1.64. The fatty acids have iodine value 99.38, m.p. 21°, f.p. 18°.

## CHEMICAL ABSTRACTS.

**Soya-bean oil.** M. SERGEEV (Masloboino Zhir. Delo, 1929, No. 3, 47—51).—The oil from Kuban beans (yield 19.6—25.3%) gives low values for  $n_D^{20}$ , acid value, and iodine value.

## CHEMICAL ABSTRACTS.

**Relationship between acidity and titer [solidification point] of oils.** I. STETZENKO and I. PANTELEYEV (Masloboino Zhir. Delo, 1929, No. 10, 20—21).—Although the f.p. of the fatty acids is higher than that of their glycerides, a high f.p. is not an indication of high acidity, since the neutral oil forms a eutectic mixture with the free fatty acids.

## CHEMICAL ABSTRACTS.

**Saponification values of highly coloured oils.** H. S. JOIS, B. L. MANJUNATH, and S. V. RAO (Mysore Univ. J., 1930, 4, 241—242).—The Albert method (cf. Coburn, B., 1930, 623) is of general applicability for the determination of saponification values of coloured or non-coloured oils.

H. BURTON.

**Cresol soap solutions.**—See XX.

See also A., May, 597, Oleastene. 602, Hydrogenation of linseed oil etc. Separation of glycerides. Rhodanometry of tung oil. 644, Colouring matter of milk. 658, Cod-liver oil.

## PATENTS.

**Spray-drying of soap and sprayed soap product.** E. P. STEVENSON and B. B. FOGLER, Assrs. to A. D. LITTLE, INC. (U.S.P. 1,779,516—7, 28.10.30. Appl., [A] 28.8.28, [B] 18.12.29).—(A) The soap solution is superheated and discharged horizontally by atomisation in a concurrent stream of heated air: the spray passes into a vertical vessel from which the hot air may be vented at the top (carrying with it any very fine over-puffed particles), whilst the soap granules separate and fall by gravity through a zone of relatively cold air. The moisture content of the powder may be controlled by admission of low-velocity conditioned air at the base of the cooling vessel. The design of the atomiser cup is detailed. Products bulking as high as 15—20 lb./cub. ft. and containing 15—20% of moisture are obtainable. (B) Sodium silicate incorporated in the soap so processed is retained in an easily soluble form in the non-dusty product.

E. LEWKOWITSCH.

**Recovery of rosin soap material from spent wood liquors.** V. DREWSEN, Assr. to WEST VIRGINIA PULP & PAPER CO. (U.S.P. 1,778,523, 14.10.30. Appl., 11.1.28).—The spent liquors are concentrated to  $d$  1.1—1.15, so that the sp. gr. of the liquor differs from that of the soap, and is centrifuged at 25—60° to separate the rosin soap.

E. LEWKOWITSCH.

**Separation of fatty or wax-like materials from their solutions.** J. K. PFAFF, K. BÖTTGER, and A. SIEWEKE, Assrs. to DEUTS. GASOLIN A.-G. (U.S.P. 1,779,287, 21.10.30. Appl., 2.7.29. Ger., 16.5.27).—An approximately equal weight of a soluble substance which crystallises well, but is chemically different, e.g., naphthalene, is added to the hot solution of the soft or badly-crystallising waxy material (such as paraffin wax, petroleum jelly, montan wax, etc.) in a petroleum

distillate or organic solvent: on cooling the solution to 0° the waxy materials are precipitated together with the added substance and may be readily separated by filtration. The naphthalene is removed from the precipitate and filtrate by steam-distillation etc.

E. LEWKOWITSCH.

**Manufacture of derivatives of sulphonated fatty acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,828, 2.9.29. Cf. B.P. 343,989; B., 1931, 501).—Unsaturated fatty oils are esterified with a monohydric alcohol (e.g., butyl alcohol) in the presence of a catalyst (e.g., hydrochloric acid) and sulphonated; the order of the processes may be reversed. [Stat. ref.]

E. LEWKOWITSCH.

**Extraction of [cashew] nut[shell] oil.** T. M. RECTOR, Assr. to FRANKLIN BAKER CO. (U.S.P. 1,777,808, 7.10.30. Appl., 8.11.28).—The whole nuts are submerged for about 1 min. in a bath of molten metal or alloy (m.p. not above 204°). The shell oil (about 12% on the raw nut) is expelled and floats to the surface, protecting the metal from oxidation; further, the shells are thereby conditioned for easy removal from the uninjured kernel.

E. LEWKOWITSCH.

**Manufacture of emulsions of waxes.** I. G. FARBENIND. A.-G. (B.P. 345,184, 21.7.30. Ger., 19.7.29).—A cellulose ether, ester, or ether-ester and a wax, e.g., beeswax, are dissolved by heating in a mutual solvent, in which, however, the wax is soluble only in the hot, and the solution is cooled rapidly while stirring. Pigments may be added to the emulsion, which is suitable for use as a protective coating.

E. LEWKOWITSCH.

**Milling of soap etc. Press for extracting liquids. Sp.-gr. balls.**—See I. Wetting etc. agents.—See III. Silicyl compounds.—See XX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Viscosity and "brushability" of paints.** H. WOLFF (Farben-Ztg., 1931, 36, 1088—1089, 1131—1132).—Results in concordance with those obtained by Beck, using a Couette torsion viscosimeter (B., 1929, 785) were obtained by means of the "Turbo viscosimeter." It is shown that the plasticity of a paint system may vary with the velocity of flow (the viscosity not being in linear relationship therewith) or be constant (the viscosity being in linear relationship with the velocity), zinc oxide in oil and nitrocellulose in butyl acetate illustrating these two types. Painting tests carried out by various observers indicate that the general speed of brushing paints is such that viscosity approaches its minimum value and its plasticity is negligible. Considerations of the change in viscosity of a paint owing to volatilisation of solvent etc. lead to the conclusion that viscosity is not a measure of brushability.

S. S. WOOLF.

**Red lead.** H. HEBBERLING (Farben-Ztg., 1931, 36, 1129—1130).—A commentary on the work of Wagner, Grohn, and others on the rust-preventative and general properties of red lead and the comparison of iron oxide pigments with it in this connexion. (Cf. B., 1930, 468, 622.)

S. S. WOOLF.

**Hydrogen-ion concentration and the colour of lead chromate pigments.** R. C. ERNST, E. PRAGOFF,

JUN., and E. E. LITKENHOUS (Ind. Eng. Chem. [Anal.], 1931, 3, 174—176).—Precipitates of lead chromate were made by mixing 0.5*N*-solutions of lead acetate and potassium dichromate and the acidity of the solutions was varied by the addition of acetic, hydrochloric, nitric, and sulphuric acids. The  $p_H$  was determined by means of the quinhydrone electrode. With decreasing hydrogen-ion concentration the colour changes from yellow at  $p_H$  3.5 to orange of a maximum brightness at  $p_H$  9, and thereafter the colours become dirty. With rise of temperature the colour changes from yellow at low temperatures to a maximum orange at 80° and becomes lighter again at higher temperature. Changes in concentration have no appreciable effect on the colour.

E. S. HEDGES.

**Colouring of paper.**—See V. Hop resin.—See XVIII. **Pigment of cereals.**—See XIX.

See also A., May, 564, **Ferric oxide colours.** 625, ***Cannabis indica* resins.** ***Euphorbium* resins.**

## PATENTS.

**Manufacture of paints.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,458, 6.12.29).—In organic or organic dyestuff pigments are suspended in an aqueous emulsion of a synthetic resin derived from a polyhydric alcohol, a polybasic acid, and a monobasic acid; a salt of ammonia or a volatile amine with an organic carboxylic acid of high mol. wt., e.g., a wax acid, is used as emulsifier.

S. S. WOOLF.

**Reducing the zinc oxide content of lithopone.** A. B. LAFTMAN, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,780,559, 4.11.30. Appl., 22.12.26).—Crude lithopone slurry is treated with a predetermined small amount of an acid, e.g., sulphuric acid, to render any acetic acid-soluble zinc compounds water-soluble, whence they are precipitated by adding the calculated amount of barium sulphide.

S. S. WOOLF.

**Production of titanium pigments.** R. H. MONK and L. FIRING (B.P. 345,668, 18.11.29).—Precipitated titanium hydroxide is treated with a peptising agent, e.g., nitric or hydrochloric acid or barium chloride, and the colloiddally dispersed mass resulting is mixed with a coagulating agent containing an ion of opposite charge to that of the dispersed hydroxide, e.g., barium carbonate, in amount chemically equivalent to the titanium dioxide present. The coagulated product is washed, dried, and calcined, e.g., at 840°, giving porous particles of high opacity and soft texture. Excess of barium chloride may be used in order to provide a catalyst for the reaction effected by calcination. (Cf. U.S.P. 1,760,513; B., 1930, 1151.)

S. S. WOOLF.

**Production of cellulose solutions [of high or known viscosity].** DEUTS. GOLD- U. SILBER-SCHNEIDEN-STALT VORM. ROESSLER (B.P. 345,613, 18.11.29. Ger., 26.11.28).—In the preparation of such solutions (particularly from cuprammonium and xanthate solutions of cellulose) both solvent and cellulose are completely freed from oxygen by means of, e.g., ammoniacal cuprous oxide or chloride, and the dissolution process is carried out in the absence of air and active light. If oxygen be quantitatively introduced into the mixture, e.g., as

persulphate, solutions of desired viscosity may be obtained.

S. S. WOOLF.

**Cellulose acetate [film-forming] compositions containing mixed volatile solvents.** KODAK, LTD. From EASTMAN KODAK CO. (B.P. 345,505, 29.3.30).—A mixture of 50 pts. by wt. of methyl acetate, 40—10 pts. of ethyl acetate, and 10—40 pts. of acetone forms a superior solvent for cellulose acetate to be used in the production of film.

E. LEWKOWITSCH.

**Compositions of matter [containing polymerised vinyl derivatives and cellulose derivatives] and articles or products obtainable therefrom.** BRIT. CELANESE, LTD. (B.P. 345,521, 10.4.30. U.S., 13.5.29. Cf. B.P. 308,658; B., 1930, 1146).—An increased proportion of a polymerised vinyl compound, e.g., vinyl acetate, can be tolerated in a cellulose ether or ester composition if a natural or synthetic resin, which is compatible with the cellulose derivative, is also incorporated. Solvents, plasticisers, pigments, etc. are added as required. The products are suitable as lacquers, for making photographic films, as adhesives for reinforced glass, etc.

E. LEWKOWITSCH.

**Coating composition.** W. E. LAWSON, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,652. 4.11.30. Appl., 18.5.28).—Compositions containing polymerised vinyl compounds, e.g., an ester or chloride, and tetrahydrofurfuryl alcohol are claimed.

S. S. WOOLF.

**Cellulosic [coating] composition.** E. E. REID and G. L. SCHWARTZ, ASSRS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,778,567, 14.10.30. Appl., 16.5.25).—Mixed alkyl cyclohexyl esters of phthalic acid, e.g., cyclohexyl butyl phthalate, are added to cellulose esters as softeners.

F. R. ENNOS.

**Synthetic resin varnishes.** IMPERIAL CHEM. INDUSTRIES, LTD., A. A. DRUMMOND, and H. H. MORGAN (B.P. 345,633, 19.9.29).—Small proportions of anhydrous metallic salts of a dehydrating character, e.g., ferric or calcium chloride, potassium hydrogen sulphate, dissolved, if desired, in an organic solvent, e.g., ethyl acetate, cyclohexanol, Cellosolve, are added to synthetic resin varnishes of the phenolic compound-formaldehyde-fatty acid ester type. Increased drying rate is claimed.

S. S. WOOLF.

**[Urea-formaldehyde] varnishes and lacquers.** A. V. KELLER and J. TAYLOR (B.P. 345,845, 17.4.30. Addn. to B.P. 331,428; B., 1930, 919).—A small proportion of an accelerating agent, e.g., sodium phosphate, is added to the urea paste before mixing. Formaldehyde and phenolic substances may replace part of the para-formaldehyde and urea derivative, respectively.

S. S. WOOLF.

**Resinous compositions and lacquers.** IMPERIAL CHEM. INDUSTRIES, LTD., R. HILL, and E. E. WALKER (B.P. 344,401, 30.8.29).—Resins of the polyhydric alcohol-polybasic acid type (including such resins containing drying or non-drying oils or fatty acids) are heated with formaldehyde-urea or -thiourea condensation products, or substances capable of yielding these, in an alcoholic solvent of b.p. above 100°, e.g., ethylenechlorohydrin, a glycol monoalkyl ether, butyl alcohol,



cyclohexanol. The resulting resins are rapid-hardening and, in the presence of an acid catalyst, *e.g.*, sulphuric, chlorosulphonic, hydrochloric, trichloroacetic, or tartaric acid, are capable of being hardened in the cold.

S. S. WOOLF.

**Substituted guanidine-aldehyde condensation product.** C. M. STINE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,636, 4.11.30. Appl., 8.10.25).—Infusible synthetic resins obtained by condensation of guanidine, its salts, or substitution products with aldehydes, *e.g.*, formaldehyde or benzaldehyde, are claimed.

S. S. WOOLF.

**Manufacture of [resinous] aldehyde condensation products.** H. KAPPELER (B.P. 345,891, 12.6.30. Switz., 12.6.29).—Furfuraldehyde or a derivative thereof, *e.g.*, furfuralamide, is condensed in one or more stages with another aldehyde or polymeride thereof, *e.g.*, paraformaldehyde, in the presence or absence of an indifferent solvent or diluent and in the presence of an acid condensing agent, *e.g.*, hydrochloric acid; dyes, fillers, or softening agents may be introduced at any stage.

S. S. WOOLF.

**Manufacture of white resin.** B. N. LOUGOVY (U.S.P. 1,779,551, 28.10.30. Appl., 10.12.23. Renewed 15.6.25).—Hexamethylenetetramine is incorporated with the resinous reaction product of phenol, formaldehyde, and urea, giving light-coloured, substantially light-fast resins.

S. S. WOOLF.

**Manufacture of synthetic resins.** BRIT. CELANESE, LTD. (B.P. 344,413, 23.11.29. U.S., 26.11.28).—A diphenylolcycloparaffin, *e.g.*, diphenylolcyclohexane, is condensed with formaldehyde in the presence of acid, neutral, or alkaline catalysts (preferably the first, *e.g.*, hydrochloric, phosphoric, or boric acid) and, if desired, other reactants capable of forming synthetic resins, *e.g.*, diphenylolpropane, urea, toluenesulphonamide. The product improves the adhesive properties of cellulose acetate lacquers etc.

S. S. WOOLF.

**Manufacture of artificial resins and lacquers and artificial masses therefrom.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 344,626, 10.2.30. Addn. to B.P. 327,673; B., 1930, 624).—The urea, thiourea, methylolurea or its anhydride of the prior patent is replaced by a mixture of such urea compounds.

S. S. WOOLF.

**Resinous compositions.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. V. ADAMS (B.P. 344,405, 28.10.29. U.S., 27.10.28).—A terpene compound, *e.g.*, terpene hydrate, camphoric anhydride, borneol, gutta-percha, is (a) added to a polyhydric alcohol and polybasic acid or its anhydride, with or without a monobasic acid radical, during the reaction period; (b) heated with either acid or alcohol component, the other component being added subsequently and the heating continued; or (c) caused to react with the condensation product of the alcohol and acid while the latter is still in the fusible, soluble condition. Improved resistance to water is claimed.

S. S. WOOLF.

**Moulding compositions and moulded articles made therefrom.** K. RIPPER (B.P. 344,872, 12.10.29. Austr., 17.10.28).—Formaldehyde (less than 4 mols.) is

heated with a mixture of urea and thiourea (2 mols. of the mixture) in a solution having  $p_H < 5$  (preferably at  $p_H 3$ ) until a hydrophobe mixed condensation product is formed, which is subsequently dried and polymerised. Alternatively, the urea constituents may be introduced individually. Fibrous materials are admixed before or after the condensation, and after the incorporation of fillers, if desired, the compositions are hot-pressed at 150–300 kg./cm.<sup>2</sup> in non-cooled moulds, and the pressed articles are given an additional air- or vacuum-drying.

S. S. WOOLF.

**Moulded compositions.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. E. BARRINGER (B.P. 345,390, 15.1.30. U.S., 28.1.29).—Strings or shavings of wood or metal, steel wool, rope waste, etc., of a sufficiently springy character, are coated with a solution of a binder, *e.g.*, an ammoniacal aqueous solution of a resin, blood-albumin, phenolic resin, and then drained and hot-moulded as required.

E. LEWKOWITSCH.

**Manufacture of plastic compositions.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. M. DEBELL (B.P. 345,489, 17.3.30. U.S., 15.3.29).—The filler is impregnated or associated with a slow-hardening binder, *e.g.*, drying oils, gilsonite, and hardened by polymerisation or oxidation; the product is comminuted and mixed with a small proportion, *e.g.*, 15%, of a fast-moulding phenolic resin and hot-moulded.

E. LEWKOWITSCH.

**Manufacture of artificial masses.** A. CAREMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,197, 7.11.29).—A powdered filling material, *e.g.*, zinc oxide or asbestos powder, is mixed with liquid or partly polymerised styrene, or a homologue thereof, and after polymerisation to the required degree the whole is moulded.

E. LEWKOWITSCH.

**Manufacture of hollow objects of synthetic resin materials.** E. E. NOVOTNY, Assr. to J. S. STOKES (U.S.P. 1,776,366, 23.9.30. Appl., 12.8.27).—A core of a solidified and cooling gaseous product, *e.g.*, carbon dioxide ice, is inserted in a predetermined position in a mass of mouldable, potentially reactive synthetic resin; heat and pressure are then applied to gasify the core, cause the resin to react, and give a hollow object of desired form.

S. S. WOOLF.

**Treating porous and non-porous [resinous] materials.** T. F. BRADLEY, Assr. to AMER. CYANAMID Co. (U.S.P. 1,780,375, 4.11.30. Appl., 25.11.29).—Such materials are treated with aqueous solutions of soluble salts, *e.g.*, ammonium salts, of polybasic acid-polyhydric alcohol resins, with or without the addition of casein solution, the resin being subsequently rendered water-insoluble, *e.g.*, by treatment with multivalent metal salts, acids, etc.

S. S. WOOLF.

**[Linoleum] floor covering.** ARMSTRONG CORK Co., Assees. of W. F. KAUFMAN and J. C. MCCARTHY (B.P. 347,527, 12.6.30. U.S., 15.7.29).

**Milling of paint etc. Sp.-gr. balls.**—See I. **Pigment dyes.** Azo dyes in varnishes.—See IV. **Impregnated sheets.**—See V. **Impregnated fabrics.**—See VI. **Antimony trioxide.**—See VII. **Rosin soap material.** Emulsions of waxes.—See XII. **Rubber composition.**—See XIV.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Graphical tensile-testing machine for rubber threads.** S. H. HAHN and E. O. DIETERICH (Ind. Eng. Chem. [Anal.], 1931, 3, 218—221).—An especially designed curve-drawing machine is described for making tensile tests on thin rubber thread such as is used in golf balls and elastic fabrics. D. F. TWISS.

**Application of rubber.**—See I. Manuring on rubber estates.—See XVI.

## PATENTS.

**Treatment of rubber latex.** F. H. UNTIEDT (U.S.P. 1,777,945, 7.10.30. Appl., 20.6.27. Renewed 12.8.30).—Rubber latex containing a foam-stabilising agent, *e.g.*, 1% of ordinary soap or 0.5% of saponin, is agitated, while air or other suitable gas is introduced, until a stiff dense foam is obtained which on drying yields a porous rubber. Compounding ingredients may be introduced prior to conversion into foam, and vulcanisation is best effected in the dried foam. D. F. TWISS.

**Concentration of rubber latexes and other aqueous dispersions of rubber.** SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 344,875, 8.11.29).—Such dispersions are concentrated by disposing therein a filtering medium, *e.g.*, an unglazed earthenware filter, and moving the medium and dispersion relative to one another at such a rate that the pores of the filter are maintained substantially free for the passage of the serum. D. F. TWISS.

**Concentration and purification of [rubber] latex.** NAUGATUCK CHEM. CO., Asses. of J. MCGAVACK (B.P. 344,647, 25.2.30. U.S., 16.3.29).—The creaming of latex, to which a hydrophilic colloid or ammonium alginate has been added, is accelerated by subjecting the latex to electric strain, *e.g.*, by applying an alternating current to two electrodes immersed in it. Using an alternating current of 60 cycles with electrodes of iron, steel, copper, or platinum, no deposition of rubber occurs at the electrodes. The amount of current passing may be restricted by thinly covering one of the electrodes with bakelite or collodion. D. F. TWISS.

**Treatment of rubber latex.** J. MCGAVACK, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,777,045, 30.9.30. Appl., 2.7.25. Renewed 8.5.29).—Latex is treated with an organic colloid, preferably pectin, in the presence of an acid, such as citric acid, in insufficient quantity to cause coagulation. The acid expedites the separation of a cream of concentrated latex. Separation may be further accelerated by centrifugal means. The separated concentrate may be purified by diluting with water and repeating the creaming operation. D. F. TWISS.

**Preservation of natural and synthetic varieties of rubber.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,491, 2.12.29).—The ageing of rubber is retarded by incorporating a substance of the formula  $RR'CX_Y$ , where R and R' are benzene or alkylbenzene nuclei, at least one of which is substituted by at least one hydroxyl or alkoxyl group, and X and Y are hydrogen atoms or XY is an alkylene residue. Examples are *p*-ethoxydiphenylmethane and di-*p*-hydroxyphenylcyclohexane. D. F. TWISS.

**Manufacture of coloured rubber.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 344,533, 31.10.29).—Unvulcanised or cold-vulcanised rubber in which fibrous material, *e.g.*, kapok, has been incorporated, preferably before coagulation of the rubber latex, is treated with a solution of a colouring matter which has no action on rubber, so that the colour is absorbed by the fibres. After washing for the removal of excess dye and drying, a translucent coloured rubber material is obtained. D. F. TWISS.

**Production of coloured synthetic rubber.** I. G. FARBENIND. A.-G., and J. Y. JOHNSON (B.P. 345,239, 16.12.29. Addn. to B.P. 241,214; B., 1927, 948).—By incorporating components of colouring materials into solutions or emulsions of intermediate products in the formation of synthetic rubber from diolefines, converting the components into practically insoluble colouring materials, and then completing the preparation of the solid synthetic rubber, improved coloured products are obtained. As examples of components of colouring materials may be instanced leuco-compounds which are subsequently oxidised or the phenolic and diazo reagents for the formation of an azo dye. D. F. TWISS.

**Manufacture of coloured articles from dispersions of rubber or the like.** DUNLOP RUBBER CO., LTD., R. G. JAMES, and D. F. TWISS (B.P. 344,537, 12.12.29).—The negative electric charge on the individual particles in an aqueous dispersion of an organic material such as rubber is converted into a positive one by adjustment of the  $p_H$  to a suitable value on the acid side of the isoelectric point; the dispersion is then mixed with a soluble organic dye, *e.g.*, methyl-violet, of which the coloured ions are cations. Latex so coloured can be used either for the manufacture of articles by spreading, spraying, etc., or to give a prior coating to formers which are subsequently immersed in latex the electric charge of which has not been inverted. D. F. TWISS.

**Compounding of rubber.** W. S. CALCOTT, W. A. DOUGLASS, and M. A. DAHLEN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,778,548, 14.10.30. Appl., 13.6.29).—Compounds of the formula  $HO \cdot R \cdot O \cdot R' \cdot Y$ , where R and R' represent aryl or alkyl-substituted aryl residues and Y represents hydrogen or hydroxyl, do not affect the rate of vulcanisation of a rubber mixture, but greatly increase the resistance of the vulcanised product to deterioration. Examples are *p*-hydroxy- and *pp'*-dihydroxy-diphenyl ether. D. F. TWISS.

**Manufacture of rubber compositions.** DUNLOP RUBBER CO., LTD., W. H. CHAPMAN, and D. W. POUNDER (B.P. 344,602, 20.1.30).—Scrap rubber-sponge trimmings and other fibrous or granular materials, such as comminuted vulcanised rubber or paper pulp, are mixed with an aqueous dispersion of rubber or similar material; the mixture is converted into a frothy condition, *e.g.*, by whipping, and is then caused or allowed to set to a permanent spongy or cellular structure. D. F. TWISS.

**Rubber[decarboxylated rosin] composition.** I. W. HUMPHREY, Assr. to HERCULES POWDER CO. (U.S.P. 1,776,857, 30.9.30. Appl., 7.10.27).—The residual product obtained by heating rosin with fuller's earth, *e.g.*, with 10% at 300—325°, is used as a softening

ingredient in rubber. The "decarboxylated rosin" is a viscous oil containing neutral rosin oil and a pitch which is different from ordinary rosin pitch; abietic acid may also be present. D. F. TWISS.

**Rubber composition and preservation of rubber.** C. W. BEDFORD, Assr. to B. F. GOODRICH CO. (U.S.P. 1,777,634, 7.10.30. Appl., 13.1.30).—The products obtained by interaction of an unsaturated aldehyde, e.g., crotonaldehyde, and an amine, such as aniline or  $\alpha$ -naphthylamine, in the presence of an acid, are substantially without accelerating influence on vulcanisation, but are effective in retarding the deterioration of rubber. D. F. TWISS.

**Preparation of an elastic-plastic body.** J. BAER (B.P. 345,175, 20.6.30. Ger., 24.6.29).—The product obtained from halogenated hydrocarbons of the  $C_nH_{2n+2}$  group and polysulphides in aqueous or alcoholic solution is dissolved in carbon disulphide and the solution mixed with rubber latex. H. ROYAL-DAWSON.

**Vulcanisation of rubber.** A. C. BURRAGE, JUN. (B.P. 344,406, 29.10.29).—In the vulcanisation of rubber with the aid of an ultra-accelerator, e.g., mercaptobenzthiazole or tetramethylthiuram disulphide, the action of the accelerator is controlled and scorching is prevented by the additional presence of a disubstituted nitrosoamine such as diphenyl-,  $\beta\beta'$ -dinaphthyl-, or phenyl-*p*-tolyl-nitrosoamine. D. F. TWISS.

**Vulcanisation processes.** J. E. POLLAK. From W. B. WIEGAND (B.P. 345,335, 19.12.29).—In the vulcanisation of rubber articles by "live" steam, in particular of automobile inner tubes, improved results such as absence of porosity are obtained by initially subjecting the articles to additional gas pressure, this pressure being progressively reduced during the course of the vulcanisation process. D. F. TWISS.

**Rubber vulcanisation process.** W. SCOTT and W. P. TER HORST, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,777,737, 7.10.30. Appl., 20.12.26).—Phenyl-substituted guanidines containing an ethoxy-group *para* to a phenyl nucleus, e.g., di-*p*-phenetidylguanidine, are used as accelerators. Di-*p*-phenetidylguanidine, m.p. 125.2—126.2°, is prepared by the action of cyanogen chloride on *p*-phenetidine or by desulphurisation and subsequent amidation of the reaction product of this base with carbon disulphide. Phenyl-*p*-phenetidylguanidine, m.p. 107.8—108.6°, *s*-tri-*p*-phenetidylguanidine, m.p. 186—188°, *o*-tolyl-*p*-phenetidylguanidine, m.p. 130.6—132.4°, and *p*-phenetidyldiguanide, m.p. 160.4—161.2°, are other examples. D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,777,738, 7.10.30. Appl., 24.2.28).—Accelerators of the thiol type, particularly mercaptobenzthiazole, are applied to the vulcanisation process in the form of their reaction products with a diguanide, especially monophenyldiguanide. D. F. TWISS.

**Antioxidant [for rubber].**—See III. Coated fabrics. Aircraft covering. Balloon fabric. Mine-ventilating tubing.—See VI. Treatment of concrete. Floor coverings.—See IX.

## XV.—LEATHER; GLUE.

**Method for testing agar and gelatin jellies.** H. C. LOCKWOOD and R. S. HAYES (J.S.C.I., 1931, 50, 145—151  $\tau$ ).—The principle of the method depends on the rigidity of the jelly when the supporting vessel is removed. The jelly is made of standard volume and shape, and the height measured, first when supported and then when inverted and the container removed. The decrease in height is dependent on quality, and an apparatus, essentially a depth micrometer, called the Ridgelimeter, is used to obtain the necessary readings. The strength of the standard agar jelly is 0.5%, which gives a Ridgelimeter decrease of 10.8% at 18°, whilst 0.3% and 1.0% jellies give decreases of 24.8% and 0.8% respectively. A 5% standard gelatin jelly shows a similar decrease of 10.8% at 9°, whilst the results for 3% and 10% are 29.6% and 2.4% respectively. The low temperature was more satisfactory for gelatin owing to the great susceptibility of gelatin jellies to temperature changes, especially above 12°. From tables compiled from experimental data, the Ridgelimeter decrease in height can be converted into "relative strength," arbitrary standards having been adopted for agar and gelatin jellies. Duplicate determinations of "relative strength" agreeing to well within 2% can readily be obtained after short experience and, as gelatins having "relative strengths" ranging from 0.66 to 1.79 are considered, this accuracy is sufficient. Tables and graphs show the effect of temperature on the rigidity of jellies, and such factors as the effect of tartaric acid and the time of heating are also discussed. For example, when a 0.5% agar solution is boiled for 1 min. with 0.05% of tartaric acid, the jelly strength is reduced by 50%, whilst gelatin jellies boiled for 10 min. with 0.1% of tartaric acid suffer a reduction of 6.0—8.0% in jelly strength.

**Fur dyes.**—See VI.

See also A., May, 627, Tea tannin.

### PATENTS.

**Preparation of hides and skins for vegetable tanning.** G. PEACE (B.P. 344,417, 27.11.29).—Limed hides or skins are treated with a solution adjusted to  $p_H$  3.5—5 by means of mineral acid and a salt of a weak acid, or a solution of sodium acetate and acetic acid, or an exhausted tan liquor with added acid, and are then tanned with vegetable tanning liquors maintained at a similar  $p_H$  till the hides are struck through. D. WOODROFFE.

**Waterproofing of leather.** C. J. M. M. LE PETIT (B.P. 344,346, 22.11.29. Fr., 28.11.28).—The latex of certain species of *Euphorbia* and cactaceous plants, e.g., *E. candelabra* or *resinifera*, before or after polymerisation or hydrogenation by means of catalysts or radiations, is extracted with a solvent, e.g., tetrachloroethane, which is afterwards evaporated off *in vacuo*, and the product, to which may be added sodium sulphoricinate and/or aluminium stearate, is used for treatment of leather. D. WOODROFFE.

**Manufacture of inextensible leather suitable for transmission belts or belting etc.** R. CHAVAND (B.P. 345,382, 10.1.30. Fr., 12.2.29).—A narrow band

of skin is stretched in a suitable frame (which is described) and tanned thereon. D. WOODROFFE.

**Protecting leathers, skins, fabrics, etc. during their manipulation or making up into manufactured goods.** ETABL. R. SCHNEIDER, and A. POELMAN (B.P. 344,453, 6.12.29. Fr., 8.12.28).—Materials used in the manufacture of fancy leather goods are protected from soiling during making up, by applying a film prepared with a cellulose derivative such as cellulose acetate, nitrocellulose, benzylcellulose, or rubber; the film is stripped off after making up. A. J. HALL.

**Spangled etc. leather.**—See VI.

## XVI.—AGRICULTURE.

**Laws of soil colloidal behaviour. V. Ion adsorption and exchange.** S. MATTSON (Soil Sci., 1931, 31, 311—331; cf. B., 1931, 556).—The base-exchanging properties of isoelectrically-precipitated "silicates," "phosphates," and "humates" described previously are examined. At  $p_H$  7.0 the exchange capacities increased with the proportion of acidoid (silicate, phosphate, etc.) to ampholytoid (sesquioxide) present in all materials examined, ferric complexes showing higher capacities than the corresponding aluminium complexes. Acidoid and ampholytoid constituents are assumed to exist in partial combination with each other, the products exhibiting both acidic and basic properties. Free acidoid valences are the active agents in cation exchange. The exchange capacity can be altered by a rearrangement of the linkings. Treatment of electronegative complexes with neutral salts produces exchange acidity, and electropositive complexes similarly cause exchange alkalinity. Exchange "neutrality" does not coincide with the isoelectric point, but varies with the nature of the ions of the salt. The cataphoretic and the Donnan isoelectric points do not appear at the same  $p_H$  value. A. G. POLLARD.

**Absorptive capacity of soils.** J. F. RADU and T. A. RUSSEV (Bul. Acad. Inalte Studii Agron. Cluj, 1930, No. 1; Proc. Internat. Soc. Soil Sci., 1931, 6, 11—12).—In Knops' method for determining the ammonia-absorptive power of soils the period of absorption may be reduced to 2 hrs. The absorptive power of soil is related to its maximum water capacity. Absorption of calcium is mainly influenced by the type of soil and the period of contact allowed. Absorption of phosphate is affected chiefly by the period of contact. Maximum absorption of potassium is reached after 2 hrs. A. G. POLLARD.

**Soil building and improvement.** V. FALCKE (Oesterr. Chem.-Ztg., 1931, 34, 64—65).—Soil-like composts are described in which sand is mixed with organic matter (manure, vegetable refuse, etc.), mineral nutrients, and water-glass. Use of the last-named leads to an improved physical condition in the fermented compost. It may be used as an ameliorant for difficult soils. A. G. POLLARD.

**Soil-forming processes in the Hawaiian Islands from the chemical and mineralogical points of view.** H. S. PALMER (Soil Sci., 1931, 31, 253—265).—Analyses of original and weathered minerals are utilised in a discussion of the changes involved in soil formation. A. G. POLLARD.

**Conception of flow-plasticity as applied to soils.** G. W. S. BLAIR (Soil Sci., 1931, 31, 291—298).—Experimental data indicate that variations in the flow-plasticity of soil pastes are produced by the addition of chalk or lime, by the addition of sand to heavy soils, and by the normal leaching process. Flow-plasticity measurements should be of value in characterising the physical properties of soils. A. G. POLLARD.

**Soil groups and their relationship to the failure of plants, a new soil relationship.** S. GOY (Pflanzenbau, 1930, 6, 351; Bied. Zentr., 1931, 60, 97—99).—Soils are classified into six groups according to their chalk and exchangeable calcium contents and their total and exchange acidities. The acid status, adsorptive capacity, and buffer power of soils are interrelated and changes in any one factor quantitatively influence the others. The bearing of these on liming practice is discussed. A. G. POLLARD.

**Sampling market-gardening soils for nitrates.** J. E. BLANEY and J. B. SMITH (Soil Sci., 1931, 31, 281—290).—Nitrate determinations, by the phenol-disulphonic acid method, in soils variously cropped and fertilised are recorded. The distribution of nitrates in soil was much less uniform than is usually supposed. For areas of  $\frac{1}{10}$  acre at least 50 samples were necessary to reduce the probable error for nitrate content to 5% of the mean value. A. G. POLLARD.

**Soil organic matter-temperature relationship in the eastern United States.** H. JENNY (Soil Sci., 1931, 31, 247—252).—In soils from the numerous areas examined the organic contents decreased exponentially with increasing mean temperature (cf. B., 1929, 407). A. G. POLLARD.

***Azotobacter* reaction in Moravian regional soil types.** F. KOSTIUK (Mitt. Czechoslov. Akad. Landw., 1930, 480; Proc. Internat. Soc. Soil Sci., 1931, 6, 21—22).—Podzols, brown earth, black earth, and rendzina (humus-carbonate) soils are examined for the presence of *Azotobacter*. The ability of the organism to survive in these soils in the natural condition and after chalking is tested by inoculation experiments. A. G. POLLARD.

**Relationship between the development of *Azotobacter* and the buffer power of soils.** H. JOHL (Diss., Göttingen, 1930; Bied. Zentr., 1931, 60A, 27).—*Azotobacter* can tolerate temporary acid conditions ( $p_H$  4.5) without injury. Growth of *Azotobacter* is a satisfactory indicator of the buffer power of soils. For determining buffer capacity the soil is shaken with a buffer solution and, after filtration, the solution is titrated. The method is not suited to humus soils. A. G. POLLARD.

**Soil acidity, with special reference to the electrolyte content of soil suspensions.** A. TERÄSVUORI (Valtion Maatalouskoetöiminnan Julkaisu, 1930, No. 29; Bied. Zentr., 1931, 60A, 19—20).—Air-drying of soils prior to analysis does not, in most cases, affect the  $p_H$  or electrolyte contents to an appreciable extent. Mineral soils dried at 100—110° have an increased electrolyte content and a lower  $p_H$ . Peat soils are less affected. Storage of water-saturated soils causes a decrease in electrolyte content and an increased  $p_H$ .

Reverse effects are observed in soils stored at half saturation and less. Changes in  $p_H$  resulting from additions of acid or salt solutions are most marked in soils the electrolyte content of which is small.

A. G. POLLARD.

**Acidity and degree of saturation of moorland soils.** E. KASAKOV (Arb. Melior. Vers.-Stat. Leningrad, 1930, [5]; Bied. Zentr., 1931, 60A, 21).—Types of acidity exhibited by moorland soils are examined. The degree of saturation with bases varied considerably with the nature of the soil, averaging 12–18% in high-moor soils ( $p_H$  3.6 approx.) and up to 91% in low-moor soil ( $p_H$  6.8–7.0).

A. G. POLLARD.

**Transient changes in acidity and the humus content of forest soil.** D. FEHÉR (Pflanzenbau, 1930, 4, 74–87; Chem. Zentr., 1931, i, 993).—The reaction of forest soils shows regular periodic changes. The highest values (summer) correspond with the highest humus content.

A. A. ELDRIDGE.

**Acidity and plant growth.** E. MORGENROTH (Pflanzenbau, 1929, 1, 434–470; Chem. Zentr., 1931, i, 993).—The  $p_H$ , hydrolytic acidity, exchange acidity, and buffering power of loam, sandy loam, and humous sand were studied in relation to their plant-physiological utilisability.

A. A. ELDRIDGE.

**Sulphur oxidation and reaction effects in Alberta soils.** O. R. YOUNGE (Sci. Agric., 1931, 11, 535–541).—The sulphur-oxidising power of a number of soils is examined. Use of fertilisers did not affect the rate of sulphur oxidation, except in one soil treated with superphosphate. Organic matter (dried clover meal) stabilised the oxidation process, the amounts of sulphate produced appearing in steady increments. In soils of different buffer capacity no relationship existed between the change in  $p_H$  and the amount of sulphur oxidised.

A. G. POLLARD.

**Nitrification [in soils].** A. HARDER (Bot. Arch., 1931, 31, 312–348).—Nitrification in soil is largely dependent on accessibility of oxygen and may be depressed where other oxygen-consuming processes predominate. Intermediate products have an inhibitory effect. In some instances nitrification proceeds in soils of  $p_H$  3.0. During the activity of nitrifying organisms a peroxide is produced.

A. G. POLLARD.

**Base exchange in soils rich in organic matter.** L. SMOLÍK (Bull. Czechoslov. Acad. Agric., 1930, 6, 912; Proc. Internat. Soc. Soil Sci., 1931, 6, 6).—The base-exchange capacity ( $Ca^{++} + Mg^{++} + Na^{+} + K^{+} + H^{+}$ ) of highly organic soils was much greater than that of mineral soils. No relationships exist between the nitrogen content and the total exchange capacity. Removal of organic matter from soils by hydrogen peroxide reduced their exchange capacity.

A. G. POLLARD.

**Electrodialysable bases in soils.** L. SMOLÍK (Bull. Czechoslov. Acad. Agric., 1930, 645; Proc. Internat. Soc. Soil Sci., 1931, 6, 9).—The amounts of exchangeable bases removed from soils by electrodialysis corresponded with those obtained by leaching with ammonium chloride solution. Repeated electrodialysis yielded the same values. Bases removed during electrodialysis may be determined by titration.

A. G. POLLARD.

**Relationships between the degree of saturation with bases and the  $p_H$  of certain soil types.** S. ODÉN and LÖDDESÖL (Medd. 368, Centralanst. försöksv. jordbruks. Avdel. lantbrukskemi, No. 40; Bied. Zentr., 1930, 60A, 20).—Changes in the  $p_H$  of soils accompanying the removal of bases by electrodialysis are examined. The rate of removal of bases by dialysis decreases with falling  $p_H$  to a minimum at  $p_H$  5.2 and subsequently increases as the process continues. The first stage of dialysis (to  $p_H$  5.2) corresponds with the removal of  $K^{+}$ ,  $Na^{+}$ , and  $Ca^{++}$  ions. In moorland soils the  $Ca^{++}$  ions are more firmly held than in mineral soils. In the second stage much  $Fe^{+++}$  and  $Al^{+++}$  are removed. The relationship between the degree of saturation with bases ( $\beta$ ) and the  $p_H$  may be thus expressed:  $\beta = (p_H - C_1)C_2$ , where  $C_1$  and  $C_2$  are characteristic constants of the soil examined.

A. G. POLLARD.

**Production of carbon dioxide in arable soil and its diffusion into the atmosphere.** H. MAGERS (Pflanzenbau, 1929, 2, 472–544; Chem. Zentr., 1931, i, 994).—An apparatus for sampling soil-air is described. The course of respiration and the effect of crops, rain, and tilling were studied.

A. A. ELDRIDGE.

**Relative solubility of the phosphoric acid of surface and sub-soils of different types.** O. ENGELS (Z. Pflanz. Düng., 1931, 10B, 181–187).—In soils poor in phosphate, subsoils contain less phosphorus soluble in 10% hydrochloric acid, and less root-assimilable phosphorus (Neubauer) than do the corresponding surface soils. Also the subsoil phosphate has a low relative solubility. The differences in the nature and proportion of phosphate in soil and subsoil cannot be correlated with the reaction of the soil or its calcium and magnesium contents (cf. Hasenbäumer, B., 1930, 579).

A. G. POLLARD.

**Determination of the matrix and manner of weathering of soil from the composition of the kaolinic silicate.** R. GANSEN, K. UTESCHER, H. PFEIFFER, A. LAAGE, H. HALLER, and M. TRÉNEL (Mitt. Lab. Preuss. Geol. Landesanst., 1930, No. 10, 1–24; Chem. Zentr., 1931, i, 672).—The ratios of base to alumina for the zeolitic and kaolinic portions of soil are compared and related to the weathering process.

A. A. ELDRIDGE.

**Determination of organic material in soil with chromic and sulphuric acids.** G. LEONCINI (Ann. Sperim. Agrar., 1930, 1, 241–246; Chem. Zentr., 1931, i, 673).—The dry soil (2–5 g., 1 mm.), coarse sand (20 g.), and dry, finely powdered potassium dichromate (25 g.) are treated (at first in the cold) with sulphuric acid (40 c.c.), the carbon dioxide being passed through barium chloride solution into 0.5N-potassium hydroxide. After air free from carbon dioxide has been drawn through the apparatus the carbonate is determined titrimetrically. Soils containing much carbonate must first be treated with phosphoric acid.

A. A. ELDRIDGE.

**Determination of plant-nutrient contents in soils.** P. LOHMANN (Bot. Arch., 1931, 31, 489–518).—Existing methods are compared. Analyses of soil extracts indicate much lower nutrient contents than does the *Aspergillus* method, differences being much more marked in the case of phosphates than of

potassium. The amount of soil phosphate utilised by the fungus far exceeds that indicated as being available by the customary chemical extraction methods. Values obtained by this method are 4–5 times the citric-soluble phosphate content, and approximate to the amounts extracted by boiling the soil with 50% acid. Neubauer values for assimilable phosphate were relatively small. The *Aspergillus* method yields values for assimilable potassium which are of the same order as those shown by the Neubauer and Wiesmann tests.

A. G. POLLARD.

**Determination of the phosphorus requirement of soil.** A. L. MASLOVA and O. M. DOBROTVORSKAYA (Udob. Urozhai, 1930, 2, 276–283).—Soils (10 g.) were treated with 0.1N-hydrochloric acid (2–12 c.c.) and the mixture was diluted with water to 100 c.c.; after 24 hrs. the phosphate content and  $p_H$  value were determined. The  $p_H$  values at which the same quantity of phosphate is dissolved differ (chernozem 3.36–4.45, dark grey loams 2.97). The  $p_H$  of soil is correlated with the response to phosphorus fertilisation.

CHEMICAL ABSTRACTS.

**Chemical method for determining fertiliser requirements [of soils] and the action of phosphatic fertilisers.** L. DWORAK (Z. Pflanz. Düng., 1931, 10B, 201–203).—Némec's remarks (B., 1930, 1000) are criticised. The method is purely a relative one, the limiting values assigned varying with type of soil and crop.

A. G. POLLARD.

**Microbiological analysis of soil fertility. IV. Nitrification and soil fertility.** J. ZIEMIECKA (Wydział Zapomogi Akad. Nauk Technicz., 1930; Proc. Internat. Soc. Soil Sci., 1931, 6, 36).—The rate of nitrification of ammonium salts by soil organisms was examined both in silica-gel cultures and in soils. The oxidation of nitrite was consistently slower than the first stage of oxidation. The most rapid nitrification occurred in soils treated with ammonium sulphate. In unfertilised soils nitrification was slow. Nitrification is not markedly affected by phosphate deficiency in soils. In garden soils nitrification was rapid, less in field soils of  $p_H$  7.0, and least in soils of  $p_H$  < 6.4. The growth of *Azotobacter* is a more delicate test of soil reaction than is that of nitrifying organisms. (Cf. B., 1930, 160.)

A. G. POLLARD.

**Theory of the hydrochloric acid extract in comparison with other methods for determining soil fertility.** R. G. PENSELER (Kühnarchiv [Sonderb. 5], 1930, 26, 413–443; Bied. Zentr., 1931, 60, 99–102).—Methods for determining soil fertility, other than the Ganssen hydrochloric acid method, are limited by petrographic, genetic, and climatic factors. The molecular ratios as calculated by Ganssen cannot be correlated closely with acidity values in chalk soils for which too great an alkalinity is indicated. Modification of Ganssen's method, by allowing a more intensive action of acid on the soil, leads to calculated molecular ratios more closely in agreement with acidity figures. Moreover, lime-requirement values so obtained accord well with those indicated by Gehring's method. By prolonged acid extraction a decreased silica : base ratio as compared with the molecular ratio of the clay is obtained; such action of the acid is, however, too severe

for the examination of zeolites or exchangeable soil permutits. For the determination of silica in the residues from acid extraction of soils, the use of 5% sodium carbonate solution is unsatisfactory. The methods of Utescher (2.5% sodium hydroxide solution) and of van Bemmelen (4% potassium hydroxide solution) give consistent results. High proportions of chalk in soils reduce the concentration of the extracting acid sufficiently to vitiate results. The exchangeable or plant-assimilable potassium, calcium, and phosphate in soils constitute only a fraction of that removed by hydrochloric acid. The exchangeable potassium determined by sodium chloride leaching agrees closely with the assimilable potassium content indicated by Neubauer tests. These values show a general parallelism with the K : Ca saturation values of Gehring. A. G. POLLARD.

**Determination of the lime requirement of soils.** G. ROHDE (Pflanzenbau, 1929, 1, 226–272; Chem. Zentr., 1931, i, 995).—Comber's qualitative method is accurate only for sandy soils. The result of the *Azotobacter* test depends not only on the lime content, but also on the microbiological condition of the soil, and is qualitative only. The  $p_H$  determination is qualitative; for sandy soil the limit is  $p_H$  6, and for loam and clay soils  $p_H$  7. The hydrolytic acidity of the soil depends on the salt used, the volume of the liquid, and the temperature. The exchange-acidity method gives good results with sandy soils, but low results on clayey and loamy soils. Hissink's method gives high limits for lime requirement; Gehring's method is inapplicable to soils high in lime. An improvement is suggested. A. A. ELDRIDGE.

**Method of determining total exchangeable bases in soils.** A. N. PURI (Soil Sci., 1931, 31, 275–279).—The total exchangeable base content of soils is calculated as the difference between the carbonate content determined by the author's method (cf. B., 1930, 1000), and the total base content ascertained by treatment with dilute hydrochloric acid.

A. G. POLLARD.

**Simplified Kappen method for determining absorbed bases [in soil].** D. V. DRUZHININ and Z. I. STROGANOVA (Udob. Urozhai, 1930, 2, 194–199).—The Kappen and Bobko-Askinasi methods give equally good results. For approximate determination, 1 pt. of soil is treated with 2.5 pts. of 0.1N-hydrochloric acid, the mixture is shaken for 1 min., kept for 5 min., filtered, and an aliquot part is titrated, using methyl-orange. All the methods are satisfactory when the soil is free from lime and calcium phosphate and is low in absorbed bases.

CHEMICAL ABSTRACTS.

**Effect of admixture of magnesium with lime applications.** O. K. ZIKHMAN-KEDROV (Udob. Urozhai, 1930, 2, 186–189).—Application to soil of magnesium carbonate had no injurious effect; it stimulated the effectiveness of calcium carbonate.

CHEMICAL ABSTRACTS.

**Absorption of ammonium- and nitrate-nitrogen by various plants at different stages of growth.** J. A. NAPEL (J. Amer. Soc. Agron., 1931, 23, 142–158).—Absorption by cotton, wheat, and maize takes place at all stages of growth; at first the ammonium-nitrogen is the more rapidly absorbed, but later the reverse holds. The absorption of the former is increased, and of the

latter only slightly affected, when the acidity of the culture solution is decreased. The highest total nitrogen absorption took place with both forms present at  $p_H$  6.0.

CHEMICAL ABSTRACTS.

**Comparison of [crop] yields on weakly acid and weakly alkaline soils.** JORET (Compt. rend. Acad. Agric., 1930, 16, 374; Proc. Internat. Soc. Soil Sci., 1931, 6, 32—33).—In the absence of nitrogenous fertilisers wheat yields from slightly acid soils ( $p_H$  6.5—7.0) were definitely lower than those from similar but slightly alkaline soils, especially in a dry season. Differences were small after treatment of the soils with ammonium sulphate and disappeared entirely when sodium nitrate was used. In general, on slightly alkaline soils, ammonium sulphate proved superior to nitrates. The reverse was the case on slightly acid soils. On the soils examined, the optimum  $p_H$  for oats in a dry season was 7.0 and at  $p_H$  6.0—6.5 (the usually accepted optimum) yields were depressed. A. G. POLLARD.

**Comparison of various forms of nitrogen fertilisers.** E. I. RATNER (Udob. Urozhai, 1930, 2, 291—300).—Using oats, the effects of sodium nitrate, ammonium sulphate, urea, calcium cyanamide, and ammonium hydrogen carbonate on podsolised sandy loam, loam, degraded chernozem, dark chestnut-brown, Turkestan loess, and red soils were compared. For the unsaturated soils calcium cyanamide was preferable; on heavy loams ammonium sulphate was slightly superior. On limed soils ammonium sulphate was as good as were the other forms. If acid phosphate was used as the source of phosphorus cyanamide was superior, even in presence of lime. With precipitated phosphate calcium cyanamide was inferior.

CHEMICAL ABSTRACTS.

**Urea fertilisers.** J. BORDAS and G. MATHIEU (Compt. rend. Acad. Agric., 1930, 16, 904; Proc. Internat. Soc. Soil Sci., 1931, 6, 33—34).—The transformation of urea into ammonium carbonate in soils is more rapid where much organic matter is present. Application to soil of cultures of ammonifying bacteria or of urease accelerated the process, notably in soils normally less active. The formation of ammonium carbonate from urea is accompanied by the dissolution of soil organic matter and a partial mobilisation of reserve nitrogen. In soils in which the urea transformation is slow a portion of the urea-nitrogen is converted into organic compounds of other types. The co-existence in such soils of urea-, ammonia-, and nitrate-nitrogen leads to considerable nitrogen losses. Such losses do not occur when urease or ammonifying organisms (in small amounts of fresh manure) is added to the soil. The latter effect is confirmed by field trials.

A. G. POLLARD.

**Composition and action of basic slag.** A. WILHEMY (Tagung Ges. Naturf. Ärzte Königsberg, 1930; Bied. Zentr., 1931, 60A, 45).—The solubility of the phosphate of slag in water varies with the soluble silica content. By shaking slag with water and a soil poor in phosphate the soluble-phosphate content of the mixture decreases due to fixation of phosphate by the soil. With soils rich in phosphate an increased phosphate solubility is observed. The plant-assimilable phosphate in slag (Neubauer test) is paralleled by its

soluble-silica content. The soluble phosphate of slag is in the form of pure calcium tetrphosphate and not as a double compound with calcium silicate.

A. G. POLLARD.

**Influence of antiseptics on the supply of nutrients in the soil.** G. A. CHIGAREV and N. B. MYAKIVA (Udob. Urozhai, 1930, 2, 312—317).—The supply of nitrate is largely inhibited by carbon disulphide, but is stimulated by chlorobenzenes. The phosphorus régime is scarcely affected; the ammonia is stimulated.

CHEMICAL ABSTRACTS.

**Toxic action of aluminium in connexion with plant growth.** B. E. GILBERT and F. R. PEMBER (Soil Sci., 1931, 31, 267—273).—In water-culture experiments the yield of lettuce was not appreciably affected by changes in  $p_H$  in the nutrient from 3.2 to 7.5, but was greatly influenced by the addition of small amounts of aluminium salts. Yields of barley in a variety of soils under different climatic conditions were more closely correlated with the amounts of "active" aluminium (Burgess, B., 1923, 617 A) in the soils than with their  $p_H$  values.

A. G. POLLARD.

**[Toxicity to seeds of] nickel and cyanogen compounds.** A. NIETHAMMER (Pflanzenbau, 1930, 4, 607—634; Chem. Zentr., 1931, i, 994).—The toxicity falls in the order: potassium nickel cyanide, nickel nitrate, carbonate, chloride, sulphate; the penetrability of the seed is of importance.

A. A. ELDRIDGE.

**Utilisation of agricultural wastes.** H. GILMAN, R. E. BROWN, J. B. DICKEY, A. P. HEWLETT, and G. F. WRIGHT (Proc. Iowa Acad. Sci., 1929, 36, 265—266).—Experiments relating to the preparation and application of various furan derivatives are summarised.

CHEMICAL ABSTRACTS.

**Effect on sugar-cane culture of fertilisation with molasses.** G. DORFMÜLLER (Deut. Zuckerind., 1930, 55, 1230—1231; Chem. Zentr., 1931, i, 1026—1027).—The effects on the biological processes in the soil are discussed; the nitrate-nitrogen may completely disappear by conversion into organic nitrogen compounds. Use of molasses with ammonium sulphate is advantageous.

A. A. ELDRIDGE.

**Utilisation of nutrients by sugar beet.** BOTTRICH (Ernähr. Pflanze, 1931, 27, 103—105).—Field trials with sugar beet are recorded in which heavier dressings of potash fertilisers gave proportionally greater relative increases in yield than lighter dressings. It is suggested that beet are able better to utilise potash supplies in soil when the latter is saturated with potash.

A. G. POLLARD.

**Time for applying sodium nitrate to sugar beet.** E. T. SYKES (J. Min. Agric., 1931, 38, 162—170).—Applications of sodium nitrate, up to 3 cwt. per acre, may be made with the seed of sugar beet without loss of sugar yield.

A. G. POLLARD.

**Ensilage of beet leaves of poor feeding value.** G. RUSCHMANN and G. GRÄF (Bied. Zentr., 1931, 60A, 17—18).—The ill-effects of feeding beet leaves to milch cows are examined. In certain districts beet leaves had a higher oxalate content than normal and were alkaline. The oxalate content was considerably reduced by ensiling the leaves, both hot- and cold-fermentation



processes giving similar results. The cold-fermented material still produced injurious effects, which are ascribed to the high nitrate content of the leaves. More suitable manuring of the growing beet with reduced dressing of sodium nitrate is recommended for the production of leaves of lower oxalate and nitrate contents.

A. G. POLLARD.

**Fodder experiments with soured and with dried beet slices with added lactic acid.** K. POSPIŠIL and V. SÁZAVSKÝ (Z. Zuckerind. Czechoslov., 1931, 55, 390—394).—Cows fed with the dried beet slices to which lactic acid had been added failed to produce the same favourable effect on milk production which results from the customary use of soured slices. On changing over from soured slices to dried slices, besides a decrease of milk the live weight also diminished. On drying the slices the fodder value of the product is diminished by about 20%.

J. P. OGILVIE.

**Fertiliser trials with potatoes.** K. VIEWEG (Ernähr. Pflanze, 1931, 27, 135—137).—The use of 40% potash salts (potassium and magnesium sulphates) in potato trials produced heavier crops than potassium chloride of equivalent potassium content, and, moreover, increased the resistance of the plants to drought conditions.

A. G. POLLARD.

**Manuring of early potatoes.** T. REMY (Ernähr. Pflanze, 1931, 27, 145—148).—A discussion of the fertiliser requirements of early and late potatoes with reference to the time of application and relative availability of fertilisers used.

A. G. POLLARD.

**"Leaf diagnosis" of Lagatu applied to potatoes, with special reference to the root-soluble nutrient content of soils [Neubauer].** W. SANDROCK (Z. Pflanz. Düng., 1931, 10B, 188—201).—The phosphorus, potassium, and nitrogen contents of potato leaves varied with the nutrient condition of the soil and with the nature and quantity of fertilisers applied. Relationships exist between differences in the composition of the leaves and the effect of fertilisers on crop yield. Variations in leaf composition cannot be correlated with Neubauer values for soil nutrients, and it is suggested that during the growth period plants utilise nutrients other than those registered by Neubauer tests at one particular growth period. In preparing leaves for analysis, drying whole leaves at 105° for 3 hrs. is satisfactory.

A. G. POLLARD.

**Manuring of acid soils.** H. KAPPEN (Ergeb. Agrik. Chem., 1930, 2, 77—87; Bied. Zentr., 1931, 60A, 42).—Increased soil acidity following applications of ammonium sulphate is the direct result of the nitrification process rather than the outcome of a specific physiological acidity of the ammonium sulphate itself. Use of rock phosphate on acid soils gives unsatisfactory results. Applications of potash salts to strongly acid mineral soils sometimes leads to a form of plant injury which may be partly remedied by treatment with magnesium salts.

A. G. POLLARD.

**Peat manuring.** A. A. KUPRIANOV and N. S. ROSANOV (Arb. Centr. Torfstat., Moscow, 1930, 2, 9—40; Bied. Zentr., 1931, 60A, 31).—The water-soluble phosphate in composts of sphagnum peat and phosphorite

was 20—30 times that of similar composts made with wood sedge, and represented 32% of the total phosphate content of the phosphorite. Manure made with peat litter produced heavier crops and was more rapidly nitrified in soil than that made with straw.

A. G. POLLARD.

**Manuring experiments on the Government rubber estate "Serpong."** W. C. VAN HEUSDEN and J. S. VOLLEMA (Arch. Rubbercultuur, 1931, 15, 125—146).—Experiments on a backward estate with poor soil which had been under cultivation for centuries show that green manures or nitrogen fertilisers have little beneficial effect. Definite benefit, however, accrues from green manuring in conjunction with phosphate-potash fertiliser.

D. F. TWISS.

**Manurial action of the anions and secondary constituents of potash salts.** O. ECKSTEIN (Ergeb. Agrik. Chem., 1930, 2, 125—142; Bied. Zentr., 1931, 60A, 43).—In soils of low adsorptive capacity the growth of oats was depressed by the Na<sup>+</sup> ion, but on strongly adsorptive soils of low sodium content treatment with sodium salts had a favourable effect. Treatment with magnesium salts produced stronger plants of increased magnesium content. Growth was improved by applications of sulphate only when used simultaneously with magnesium salts. No beneficial effects of the chloride ion was observed.

A. G. POLLARD.

**Behaviour of and profit from different varieties [of crops] with increased nitrogen manuring.** II. NILSSON-EHLE (Z. Pflanz. Düng., 1931, 10B, 169—181).—Different varieties of an individual crop responded very differently in yield and to some extent in quality to nitrogenous fertilisers. Varietal differences in this respect were more apparent with the heavier dressings of fertiliser.

A. G. POLLARD.

**"Finger-and-toe" and manuring.** J. KREUZ-POINTNER (Ernähr. Pflanze, 1931, 27, 172—173).—The adoption of appropriate fertiliser schemes in the prevention of finger-and-toe disease is only reliable if applications of farmyard manure are omitted.

A. G. POLLARD.

**Fruit tree carbolineums.** E. PROFFT and G. GOETZE (Zentr. Bakt. Par., 1931, II, 83, 127—164).—Chemical examination of numerous carbolineum preparations is recorded, and the physiological effects of the constituents on insects and plants are examined. Chemical characteristics of suitable preparations of this class are enumerated.

A. G. POLLARD.

**A rust of Clingstone peaches in California.** M. C. GOLDSWORTHY and R. E. SMITH (Phytopath., 1931, 21, 134—167).—Urediniospores of the fungus were more susceptible to sulphur and sulphur compounds than to copper salts, whilst mineral oil stimulated their germination.

A. G. POLLARD.

**Fingerling's process for the ensilage of green plants.** M. GERLACH (Tierernährung, 1930, 1, 179—193; Bied. Zentr., 1931, 60, 109—112).—The process (addition of hydrochloric acid to the fodder in the silo to produce pH 2) is discussed. Better results are obtained by increasing the amount of acid-liquor acid and the pressure applied at the surface. The proportion of acid necessary varies with the nature of the fodder and its

moisture content. The covering layer of oil, originally used, is unnecessary if the acid is added in portions as the silo is filled. The organic acid content of silage so produced is small. A. G. POLLARD.

**Stimulation of *Lemna major* by organic matter under sterile and non-sterile conditions.** N. A. CLARK and E. M. ROLLER (Soil Sci., 1931, 31, 299—309; cf. B., 1924, 484).—*Lemna major* grew for several years in purely inorganic but non-sterile media. Addition of small amounts of extracts of soil, manure, vegetable matter, etc. under these conditions stimulated reproduction, there being a simultaneous increase in the micro-organisms present in the media. Under sterile conditions the organic materials were without effect on or reduced reproduction, but the original stimulative effects were produced by reinoculation. Sterile plants grew more rapidly than non-sterile. Pure cultures of certain bacteria in the presence of organic matter increased reproduction in sterile media, but others were ineffective. None of the pure organic substances examined caused stimulation under sterile or non-sterile conditions. A method of sterilising the plants by the use of potassium mercuric iodide and bleaching powder is described. A. G. POLLARD.

**Ability of dry fungicides to adhere to seeds.** A. SINSKI (Udob. Urozhai, 1930, 2, 206—212).—Experiments with wheat, oats, and barley, employing calcium arsenate, Paris green, copper carbonate, and copper sulphate, are described. Copper carbonate and sulphate adhere best to wheat, and calcium arsenate adheres best to oats. CHEMICAL ABSTRACTS.

**Superphosphate from apatite. Solubility of Bordeaux mixture.**—See VII. **Butyric fermentation in silage.**—See XVIII. **Turnip tops.**—See XIX.

See also A., May, 596, Soil erosion. 660, Effect of nitrogen on plant yield. Nodule bacteria and leguminous plants.

#### PATENTS.

**Manufacture of phospho-nitrogenous manures, ammonium phosphate, and sodium bicarbonate.** Soc. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTR. (B.P. 344,962, 21.12.29. Holl., 27.12.28).—A mixture of carbon dioxide with phosphorus oxides is used instead of carbon dioxide alone in the ammonia-soda process. After removal of the precipitated sodium bicarbonate, the liquor is concentrated to decompose ammonium carbonate and to crystallise part of the excess sodium chloride; the mother-liquor on cooling deposits a mixture of ammonium chloride and phosphate suitable for use as a fertiliser. Alternatively, the liquor remaining after removal of the sodium bicarbonate is enriched in ammonium phosphate by further treatment with ammonia and the mixed gases. L. A. COLES.

[Fertilisers from] sewage.—See XXIII.

### XVII.—SUGARS; STARCHES; GUMS.

**Importance of a preliminary purification of (beet) diffusion juice.** I. B. MINTZ and B. E. KRASILSHCHIKOV (Nauk. Zapiski Tzuk. Prom., 1930, 9, 515—537).—Diffusion juice should be filtered through asbestos or other fibrous material. The total nitrogen content of

carbonatation juices, obtained from diffusion juices previously boiled or treated with 0.5—1% of kieselguhr, is 9—12% lower than that of ordinary treated juice. Good clarification is obtained by sulphitation to  $p_H$  4.0 at 20—40°, followed by neutralisation to alkalinity of 0.1 with lime and rise of temperature to 80°, and then by further filtration and carbonatation to alkalinity of 0.01—0.02. CHEMICAL ABSTRACTS.

**Bleaching effect of washing the massecuite of the second skip.** V. V. TSCHESHCHCHEV (Zhur. Sakh. Prom., 1930, 4, 305—308).—When massecuite was washed with greens ( $d$  1.26) the yield of yellow sugar decreased by 9.5%, with an increase in purity of 0.9%, the colour decreased by 47.9% and the centrifuging time by 19.2%. Values for washing with hot final molasses were 1.5, 0.9, 23.1, 10%, and for washing with 3.4—5.0% of its weight of molasses at 90—95% 9.2, 3.7, 83.7, and 25%. CHEMICAL ABSTRACTS.

**Conditions for increasing the velocity of crystallisation in "sugar loaves."** M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauk. Zapiski Tzuk. Prom., 1930, 9, 450—492).—The best cooling-room temperature is 5—10°. The quality of the sugar is unaffected by cooling to the lower temperature. CHEMICAL ABSTRACTS.

**Cold aqueous digestion [method of determining sugar in the beet].** J. VONDRÁK and B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1931, 55, 395—399).—Using pulp prepared by the Herles press, this method gives distinctly lower results than does hot aqueous digestion, the difference depending on the mesh of the sieve used in the press. It is recommended that the hot aqueous method only be employed for fresh slices for factory control purposes, though the cold method be retained for exhausted slices. J. P. OGILVIE.

**Electrical conductivity of molasses for after-product control.** J. PELLER (Z. Zuckerind. Czechoslov., 1931, 55, 399—403).—A study of the relationship between the saccharisation (Balling degree) and the electrical resistance of beet molasses showed that besides the water content its composition exerts a great influence on the conductivity of the undiluted product. In the presence of grain the resistance of the product is increased. J. P. OGILVIE.

**Iodometric determination of sugar.** M. S. FILOSOF (Nauk. Zapiski Tzuk. Prom., 1930, 9, 586—589).—Diluted molasses is inverted with hydrochloric acid, cleared with lead acetate, sodium carbonate, and alumina, and treated with Fehling's solution; the excess of copper is determined by addition of potassium thiocyanate and iodide, followed by titration with thio-sulphate. CHEMICAL ABSTRACTS.

**Measurement of turbidity with a spectrophotometer, with especial reference to sugar-house products.** R. T. BALCH (Ind. Eng. Chem. [Anal.], 1931, 3, 124—127).—Existing methods of determining the turbidity of sugar solutions are subject to large errors through lack of colour compensation. A method is described, in which light of a definite wave-length is used and the colour is compensated by using as a standard a portion of the same sugar solution from which suspended solids have been removed. Filtration through

paper, with the aid of a slow-filtering grade of commercial kieselguhr, is recommended in place of asbestos or sand for the preparation of the standard. The turbidities are expressed in  $-\log t$  values, using the equation  $-\log t = (-\log T)/cb$ , where  $t$  is the transmittancy, reduced to unit conditions of concentration and depth of solution,  $c$  is the concentration in g. per c.c.,  $b$  the thickness of the solution in cm., and  $T$  the transmitted fraction of incident light. This is recommended as being the most convenient method, in spite of the fact that values of  $-\log T$  are not strictly proportional to the concentration or depth of the suspension. Cube sugar showed distinctly lower turbidity than standard granulated sugars. The turbidity of raw cane sugars can be correlated roughly with the ease of filtration.

E. S. HEDGES.

**Sugar-cane culture. Sugar beet. Beet slices as fodder.**—See XVI. **Evaluation of honey.**—See XIX.

### XVIII.—FERMENTATION INDUSTRIES.

**Bakers' yeast. II. Quick-acting yeast.** E. ROSENBAUM (Z. Unters. Lebensm., 1931, 61, 80—84; cf. B., 1931, 217).—Ten samples of quick-acting bakers' yeast (variously known as record-, double-, noble-, and strong-yeast) were examined, and the high rates obtained for fermentation (especially initially), carbon dioxide evolution from 10% sucrose, and increase in dough volume (at 30°) are considered of especial value to bakers. Analyses gave: water 72.1—74.4%, acidity 0.7—2.2 c.c. of 0.1N-sodium hydroxide per 10 g., N 50.92—59.03% (dry solids),  $P_2O_5$  2.36—4.38% (dry solids), total fermentation period 147—186 min. (191—266 after 14 days at 20°).

J. GRANT.

**Dissolution and precipitation of the  $\alpha$ -resin of hops (humulone) during wort boiling.** W. WINDISCH, P. KOLBACH, and C. VOGL (Woch. Brau., 1931, 48, 139—144, 149—152).—To 150 c.c. of deoxygenated wort 5 c.c. of an alcoholic solution of humulone were added. The wort was boiled under reflux for various periods and cooled in an atmosphere of nitrogen. The coagulum was collected and washed on a filter-paper and the flask finally rinsed with alcohol into the filtrate. From this the resin was extracted by acidifying and shaking with 30 c.c. of chloroform, from which, after washing and centrifuging, 15 c.c. were pipetted. This was evaporated *in vacuo* and the residue cooled in a current of carbon dioxide and weighed. The coagulum was treated with 30 c.c. of alcohol and 175 c.c. of water before extraction with chloroform. Corrections were determined and applied for the solubility of the chloroform in the aqueous liquid, also for material extracted from the wort. Duplicate determinations agreed to about 1% on the portion recovered from the wort, or to about 3% on the total recovery. With increasing hydrogen-ion concentration the amount of resin in the wort was found to decrease, as was expected, but the amount recoverable from the coagulum was insufficient to balance this, hence the total recovery decreased with increasing acidity. This is the reverse of the result obtained by boiling humulone in pure buffer solutions (B., 1928, 31), and is attributed to the occlusion of a portion of the resin in the colloid coagulum, in a state

resistant to extraction by chloroform. This view is supported by the facts that when wort of low protein content was used, the apparent loss was less than with normal worts; that longer boiling, hence greater protein precipitation, increased the loss, though the amount of resin recoverable from the coagulum varied irregularly; and that the loss was less when part of the protein was precipitated by boiling before addition of the humulone. The actual apparent losses at  $p_H$  6.5, 6.0, 5.5, and 5.0 were with normal worts 5.2, 7.2, 8.9, and 10.6%, and with low-protein worts 3.2, 4.6, 5.5, and 6.1%, respectively. At the lowest acidity practically all the humulone added (165—187 g./litre) would be in solution as a salt, whilst with increasing acidity larger amounts would exist as the almost insoluble free acid, and would be much more liable to occlusion. The smaller losses observed under the more alkaline conditions do not greatly exceed those actually due to decomposition (1—3%) and previously noted on boiling humulone with buffer solutions.

F. E. DAY.

**Decarbonation of brewing waters.** J. H. COLLETT (J. Inst. Brew., 1931, 37, 274—277).—Carbonates are precipitated from the water by the addition of calcium hydroxide. The small amounts of calcium hydroxide and calcium carbonate which still remain in solution are decreased by the addition of calcium chloride, and, after the clear liquid is run into a second tank, the residual amount in solution is exactly neutralised by the addition of acid, or, preferably, by potassium or sodium bisulphate. The sedimentation of the calcium carbonate is completed in 3 hrs. if the addition of the constituents to the water takes place at 65°.

C. RANKEN.

**Decarbonation of mashing liquor [in brewing].** R. L. SIAU (J. Inst. Brew., 1931, 37, 272—274).—The raw water is pumped through an exchanger in which it is heated to 43° by the outgoing boiling water. It then enters the condenser in which it is heated to 99° by the steam from the boiling tanks, and thereafter passes through a steam-jacketed pipe before entering the tanks which are provided with steam coils and steam vents. By using this method of regenerative heating, the carbonate content of the water can be reduced to 2.5 pts. per 100,000 without using much more steam than is required to raise the temperature of the water to 65°.

C. RANKEN.

**Plastered wine.** A. BORNTAEGER (Z. Unters. Lebensm., 1931, 61, 1—38).—Little potassium hydrogen sulphate results from the reaction of tartar with calcium sulphate in grape must treated with gypsum. The variation in acidity of wines from highly plastered musts may be due to reaction, in the presence of alcohol and potassium hydrogen sulphate, of free tartaric acid with the potassium salts of organic acids, and the lowering of the acidity by precipitation of tartar as an acid salt. Plastering therefore raises the "acid energy" (i.e., power to produce acidity) without necessarily raising the actual acid value. The solubility of calcium sulphate is lowered by potassium sulphate and by alcohol, and raised to slight extents by tartaric, malic, lactic, succinic, and acetic acids (in decreasing order), but not by tannic acid. In alcohol at 96°, equivalent mixtures of potassium sulphate and tartaric acid are converted into

tartar and potassium hydrogen sulphate, the latter then being decomposed into potassium sulphate and sulphuric acid, only the sulphuric acid and residual tartaric acid being soluble. In winter new wine deposits more potassium bitartrate than does old wine, especially if it has been highly plastered. Other points investigated are the formation of potassium hydrogen sulphate in highly plastered wines, the effects of the constituents of wine on the reaction between tartar and gypsum, the separation of calcium sulphate during fermentation, the solubility of tartar in aqueous solutions of the organic acids of wines and of their potassium salts, and the relation of the alkalinity of the ash to the gypsum content.

J. GRANT.

**Detection of fruit wine in grape wine by the dibenzylidenesorbitol method.** M. KLOSTERMANN and W. FACHMANN (Z. Unters. Lebensm., 1931, 61, 100—103).—The wine is decolorised with animal charcoal, the filtrate evaporated under reduced pressure, and the residue extracted with absolute methyl alcohol to eliminate pectins and dextrin. Amino- and organic acids etc. in the extract are precipitated by lead acetate, the excess of lead in the filtrate is removed with hydrogen sulphide, and the extract is evaporated under reduced pressure and acetylated with 3 c.c. of pyridine and acetic anhydride on the water-bath for 15 min. After neutralisation, the resulting oil is extracted in ether and isolated by evaporation and recrystallisation from a solution in hot water (m.p. 98—99°). The method is sensitive to additions of 2.5% of fruit wine (or less if Jahr's microscopical method is used; cf. B., 1930, 787), and is more rapid and reliable than the production of dibenzylidenesorbitol (Werder, B., 1929, 619; also von der Heide and Hennig, B., 1929, 574, 735).

J. GRANT.

**Use of the quartz lamp in the examination of raisin and currant wine.** P. BERG and E. KRÖGER (Wein u. Rebe, 1930, 12, 327—331; Chem. Zentr., 1931, i, 699).—Substances which inhibit luminescence are removed by treatment with carbon; a blue coloration then indicates the presence of raisin wine. The luminescence is given also by fermentation products of other dried fruits.

A. A. ELDRIDGE.

**Methyl alcohol in alcoholic drinks. I. Methods of testing and its determination.** N. A. ESPINOSA (Anal. Asoc. Quím. Argentina, 1930, 18, 57—73).—Two methods of determination have been used, after slight modification, for large numbers of analyses. In the Denigès-Villavecchia method the colour is matched with that given by standard solutions after keeping in the cold for 15 hrs. It is sensitive to 0.0002 c.c. of methyl alcohol. The Chapin method, using the Schiff, and not the Schiff-Elvove, reagent, is sensitive to 0.00008 c.c. Glycerol, which is separated by distillation, and formaldehyde, which is allowed for by a blank determination before oxidation, are the only substances liable to interfere with the determination. Rothera's reaction can be used for the determination of acetone.

R. K. CALLOW.

**Butyric fermentation in silage.** G. RUSCHMANN and L. HARDER (Bied. Zentr., 1931, 60A, 18—19).—New methods for determining the number of *Amylobacter* in silage are described. Results are closely paral-

leled by chemical determinations of the butyric acid present. In silage to which sugar was added the number of *Amylobacter* was small.

A. G. POLLARD.

**Sterilisation of enzymes.**—See XIX.

See also A., May, 653, **Diastase from wheat.** 655, **Protein crystals possessing tryptic activity.** Cellulose-fermenting organism. 656, **Acetic fermentation.** Propionic group of bacteria.

## PATENTS.

**Manufacture of a product containing vitamin [B].** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 345,669, 22.11.29).—A yeast extract obtained by liquefying pressed yeast is added to an aqueous solution of a fermentable type of sugar and the whole fermented by a distillery yeast or, preferably, a wine yeast.

C. RANKEN.

## XIX.—FOODS.

**Sterilisation of flours and pulverised enzymes.**

A. J. J. VANDE VELDE (Bull. Acad. roy. Belg., 1930, [v], 16, 1396—1403).—The specific activities of malt amylase, powdered pepsinase, takadiastase, trypsinase, pancreatin, and rennin are undiminished, and in some cases slightly increased, by one or two treatments with carbon disulphide. They are, however, completely sterilised.

C. W. GIBBY.

**Predetermining flour soundness.** D. A. COLEMAN (Amer. Miller, 1930, 58, 434).—Maximum normal fat-acidity values of wheat are: hard red winter 8.5, hard red spring 5.5, soft red winter 8.0, durum 4.0, white 5.5. Damaged wheat gave values between 8.6 and 44.

CHEMICAL ABSTRACTS.

**Pigment of cereals.** O. SCHAMBERG (Z. Ges. Mühlenwesen, 1930, 7, 105—106; Chem. Zentr., 1931, i, 866).—The location and artificial variation of the colour are discussed, and means for the detection of sulphur, organic dyes, dichromate, and polishes are described.

A. A. ELDRIDGE.

**Determination of mixtures of rye and wheat products in flour and bread. Recognition of patent flours.** K. SEIDEL (Z. Ges. Getreidew., 1930, 17, 206—212; Chem. Zentr., 1931, i, 1034).—Sedimentation tests on dried, sieved crumb in solutions of formaldehyde, chloral hydrate, sodium hydroxide, potassium thiocyanate, sodium monohydrogen phosphate, and calcium nitrate are described. Rye bread gives the greater volume of deposit and exhibits the longer separation time. Conditions affecting the test and the viscosity of alkaline extracts are mentioned. Aqueous extracts cannot be differentiated by Millon's test; the biuret reaction gives a blue colour which with rye becomes yellow at 80°. Colours obtained with alkaline and alcoholic extracts in the biuret, iodine, and other colour reactions are described. Tillmans' trifuorosan determination affords a qualitative distinction. With Molisch's reaction rye bread gives a dark wine-red, and wheat bread a faint pink solution, mixtures giving intermediate tints. Extracts of patent wheat flour give deep, and of rye faint, colorations with violet-black.

A. A. ELDRIDGE.

**Coagulation of milk.** M. CHIÒ and S. REPETTO (Boll. Soc. Ital. Biol. Sper., 1929, 4, 3 pp.; Chem.

Zentr., 1931, i, 1033—1034).—Sodium oleate and stearate coagulate milk with different velocities. Calcium oleate retards, and calcium stearate accelerates, coagulation, whilst the effect of the calcium salts of milk-fatty acids is intermediate. A. A. ELDRIDGE.

**Determination of nitrogenous matter in milk by means of the decrease in conductivity.** R. STROHECKER (Z. Unters. Lebensm., 1931, 61, 69—80).—The constituent sugars, fats, and salts exercise only a small constant influence on the conductivity of milk, marked differences being produced by changes in protein content. The conductivities ( $l$ ) of 25 c.c. of milk diluted with (1) 25 c.c. of conductivity water, and (2) 25 c.c. of 0.1*N*-hydrochloric acid, and (3) a mixture of 12.5 c.c. of 0.2*N*-acid and 37.5 c.c. of water, were determined under similar conditions, and the value  $l_F = (l_1 + l_3 - l_2)$  plotted for a number of milks. The resulting straight line ( $l_F$  115 for 22% N and 145 for 3.4% N) enables the nitrogen content of any milk to be determined from  $l_F$ . The method is rapid and gives close agreement with the Kjeldahl method for normal, watered, and condensed (unsweetened) milks, and with milks from diseased animals. It is unaffected by acidity or by small amounts of preservatives, but sodium carbonate or sucrose (e.g., in sweetened-condensed or ordinary milk) increases  $l$ , a correction for the latter being made by deducting from the nitrogen content the same percentage of its value (usually 10%) as the percentage of sucrose present. J. GRANT.

**Refractive index of milk.** A. GRONOVER and F. TÜRK (Z. Unters. Lebensm., 1931, 61, 85—94).—The sp. gr., acidity,  $n$ , and dry non-fatty solids are tabulated for milks as supplied (145), collected (104), and drawn from a single cow (39). In each case the last two values are approximately linearly related, but the graphs are too irregular to allow the method to be used even for sorting purposes (cf. Mundinger, B., 1929, 795). J. GRANT.

**Determination of  $p_H$  of cheese whey.** G. WODE (Lait, 1930, 10, 1083—1087; Chem. Zentr., 1931, i, 706).—An electrometric apparatus is described.

A. A. ELDRIDGE.

**Viscosity of egg-albumin, and changes in fresh and preserved eggs.** D. CORTESE (Ann. Sperim. Agrar., 1930, 3, 125—132; Chem. Zentr., 1931, i, 865).—The viscosity of the albumin of eggs stored at the ordinary temperature decreases in spite of evaporation; in cold storage the decrease is slower, but in both cases rectilinear. The decrease is very rapid in bad eggs owing to the action of liquefying bacteria. A. A. ELDRIDGE.

**Determination of caffeine in coffee, tea, and maté.** J. GROSSFELD and G. STEINHOFF (Z. Unters. Lebensm., 1931, 61, 38—56).—The following procedure is based on the Lendrich-Nottbohm (B., 1909, 379) and Fendler-Stüber (cf. Pritzker and Jungkunz, B., 1926, 605) methods, but is quicker, and avoids errors due to adsorption of caffeine, to incomplete removal of impurities, to loss of caffeine by oxidation, and to oxidation of fats to water-soluble fatty acids. A mixture of the ground, ammoniacal sample and sand is extracted under reflux with carbon tetrachloride, and the residue after evaporation re-extracted with hot water,

and (except with tea or raw coffee) the solution oxidised in the cold with 5% potassium permanganate solution (15 min.), the excess of which is removed with 10% sodium thiosulphate after addition of copper sulphate. The mixture is precipitated with *N*-sodium hydroxide, filtered, the filtrate extracted with chloroform, and the extract weighed after evaporation. A Kjeldahl nitrogen determination serves as a check. The results are satisfactory, though Hag coffee yields a small nitrogen-free impurity (0.02%), and coffee substitutes give high nitrogen values (0.007—0.015%). The results for tea are higher than those obtained by the Lendrich-Nottbohm method. J. GRANT.

**Evaluation of honey.** I. L. LABAND, W. BARTELS, and A. FAUTH (Z. Unters. Lebensm., 1931, 61, 56—69).—Gothe's diastase test (cf. Fiehe and Kordatzki, B., 1928, 543, 911) has been tested on 200 German and foreign honeys, and satisfactory results were obtained. A limiting value of 8.3, corresponding with a fermenting power of 17% (i.e., diastase value  $50 \equiv 100\%$  fermentative power), is suggested for honey marketed under the German (March, 1930) regulations, which state that honey should not be heated so as to destroy or appreciably to weaken the diastatic power. The appearance of a stable cherry-red colour in Fiehe's test (*loc. cit.*) is not always evidence of the presence of artificial invert sugar, since it is also produced by strongly heated unadulterated honeys. The colour of the Fiehe test increases with duration and temperature of heating, and there is evidence of a relationship between the intensity and the diastatic power. J. GRANT.

**Juice of sour raspberries.** R. COHN (Deut. Destillateur-Ztg., 1930, 51, 522; Chem. Zentr., 1930, ii, 2453).—The volatile acidity is high, and the non-volatile low (0.36—0.67% instead of 1.0—1.4% as citric acid). A. A. ELDRIDGE.

**Effect of washing of ensiled turnip tops on the loss of crude and digestible nutrients.** F. HONCAMP and W. SCHRAMM (Bied. Zentr., 1931, 60A, 17).—Washing of turnip tops either before or after ensilage does not involve appreciable losses in nutrient value. A. G. POLLARD.

**Examination for benzoic acid in food products.** U. HORDH (Anal. Asoc. Quím. Argentina, 1930, 18, 31—36).—In order to isolate the benzoic acid free from disturbing impurities the material is distilled in steam, the distillate extracted with ether, the extract washed with a little water, and the residue obtained by evaporation is dissolved in the minimal amount of water and tested by Jonescu's reaction (oxidation by hydrogen peroxide in presence of ferric chloride). This is best carried out in the cold, when a stable and intense violet coloration develops after 24 hrs. R. K. CALLOW.

**Use of buffers in the determination of colour [in foods] by means of titanium trichloride.** II. O. L. EVENSON and R. H. NAGEL (Ind. Eng. Chem. [Anal.], 1931, 3, 167—169; cf. Evenson and McCutchen, B., 1928, 704).—Dyes in foods may be determined by reduction with standard titanium trichloride solution in the presence of buffer solutions which also act as catalysts. It is essential to use the correct buffer solution with each individual dye and suitable buffers

are listed for all the water-soluble coal-tar dyes permitted in foods. In all cases it is found possible to use either sodium citrate or sodium hydrogen tartrate.

E. S. HEDGES.

**Bacteria and moulds of milk curd. Rôle of symbiosis in the ripening of Camembert cheese.**

F. SANSONETTI (Lait, 1930, 10, 627—640, 782—793, 858—874, 989—1001, 1009—1023; Chem. Zentr., 1931, i, 703—704).

**Tinplate containers and food products.**—See X. **Beet leaves. Beet slices as fodder.**—See XVI. **Bakers' yeast. Silage.**—See XVIII.

See also A., May, 627, **Tea tannin.** 644, **Colouring matter of milk.** 658, **Vitamin-A in maize.** 659, **Vitamin-B in peanut.**

#### PATENTS.

**Conditioning of grain.** J. A. HALL and W. F. C. GEORGE (B.P. 344,538, 12.12.29).—Grain passing through a tower is submitted to the action of gaseous or liquid chemicals in a totally enclosed recirculating plant. The process may be used for sterilising the grain, or for adjusting the  $p_H$  or the enzymic activity.

E. B. HUGHES.

**Conservation of heat in milk dryers.** C. C. KERSHAW and H. L. SOLIE (U.S.P. 1,776,030, 16.9.30. Appl., 10.5.29).—The wet exhaust from the drum dryers passes through traps and into an expansion apparatus, and its temperature is thus lowered from about 150° to 100° without serious loss of heat. This heat is then used to preheat the milk before drying.

E. B. HUGHES.

**Preservation of animal and vegetable substances.** R. WILLSTÄTTER (B.P. 344,394, 4.12.29).—Meat, fruit, etc. is preserved for several weeks by storage in a closed vessel in an atmosphere containing 0.004—0.01 vol.-% of hydrogen cyanide. Free water, iron, and alkalis must be excluded. The residual acid has no toxic effect, but may be removed by aeration if desired.

E. B. HUGHES.

**Production of jellifying substances.** H. G. LOESCH, Assr. to GEN. FOODS CORP. (U.S.P. 1,776,362, 23.9.30. Appl., 29.12.25).—To the cool aqueous extract of citrus pomace about 0.09% of starch (in solution) is added to remove the turbidity due to hesperidin. The almost clear supernatant pectin solution is then drawn off, filtered, concentrated *in vacuo*, precipitated with alcohol, pressed, and dried.

E. B. HUGHES.

**Smoke-flavour and aroma-imparting binder for food products etc.** J. ALSBERG, Assr. to B. HELLER & Co. (U.S.P. 1,777,026, 30.9.30. Appl., 31.10.25).—Maple, hickory, beech, or birch wood, etc., preferably the first, in small pieces is destructively distilled in the presence of a limited supply of air. The distillate, which contains the flavouring constituents, is evaporated to half its weight by exposure in shallow pans or on a rotating drum to an air blast, to remove deleterious constituents. The desired flavour is imparted to the binder, such as flour, *e.g.*, for sausages, by mixing in about 10% by wt. of this concentrate.

E. B. HUGHES.

**Milling of chocolate etc. Dough mixer. Press for extracting liquids. Centrifugal separator for milk.**—See I. **Product containing vitamin-B.**—See XVIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Cresol soap solutions.** J. LÉVOLT (Magyar Gyóg. Társ. Ert., 1930, 6, 474—480; Chem. Zentr., 1931, i, 657).—For the determination of cresol in liquor cresoli sap. the soap is preferably precipitated with barium chloride, the cresol being extracted from the acidified liquid with light petroleum and determined gravimetrically. In presence of resin and naphtha acids the cresol soap solution is treated with concentrated calcium chloride solution; the cresol is distilled with steam and extracted with alkali from the distillate before acidification and extraction.

A. A. ELDRIDGE.

**Tincture of iodine: variations and adulteration. Tincture prepared from denatured alcohol.** G. PANOPOULOS and J. MEGALOIKONOMOS (Ext. Prakt. Acad. Athènes, 1927, 2, 15 pp; Chem. Zentr., 1930, ii, 2405).—The formation of ethyl iodide and hydrogen iodide occurs independently of the action of light, even in 85% alcohol, but it is prevented by the addition of potassium iodide. The tincture prepared from alcohol denatured with 5% of wood spirit, 25% of acetone, 0.5% of petroleum, and traces of dyes quickly forms hydrogen iodide and organic iodine compounds, chiefly iodoacetone.

L. S. THEOBALD.

**Refractometric examination of tinctures.** F. WRATSCIKO and J. KOWARZ (Pharm. Presse, 1930, 97—99, 115—122, 131—135, 146—152; Chem. Zentr., 1931, i, 655).—The alcohol and extract contents are obtained graphically from determinations of  $d$  and  $n_D$ . The application of the method is discussed.

A. A. ELDRIDGE.

**Coffeinum natrium benzoicum.** A. HERAY (Magyar Gyóg. Társ. Ert., 1930, 6, 468—473; Chem. Zentr., 1931, i, 656).—A double salt is formed by 2 mols. of caffeine and 3 mols. of sodium benzoate. The method of analysis is described.

A. A. ELDRIDGE.

**Titration of Solutio arseniatis natri.** E. V. CHRISTENSEN (Arch. Pharm. og Chem., 1930, 37, 350—358; Chem. Zentr., 1931, i, 822).—When the sodium arsenate content is up to 25% greater or less than is prescribed, the method of the Danish Pharmacopœia (1907) gives good results if a 0.2—0.5 c.c. excess of silver nitrate is used.

A. A. ELDRIDGE.

**Seasonal variations in alkaloid content of Indian *Ephedra* species.** T. P. GHOSH and S. KRISHNA (Arch. Pharm., 1930, 268, 636—643).—The alkaloids from *Ephedra nebrodiensis* and *E. Gerardiana* contain 60—70% of ephedrine; those from *E. intermedia* usually about 10% (50% in samples from Baluchistan). *E. intermedia* from the Chini Range, Bashahr Division, Punjab, is a convenient source of  $\psi$ -ephedrine (1.8% of the drug). The altitude of the source does not affect the alkaloid content. A mixture of chloroform (1 vol.) and ether (3 vols.) may be used to extract ephedrine without risk of converting it into the hydrochloride. The seasonal variations are dependent on the times of rain- and snow-fall, both of which cause a decrease in the content of ephedrine and of total alkaloid. In general, both these values decrease from May until August and then rise to their maximum in October or November. The drug should be gathered immediately

before the onset of frost. When kept dry it retains its alkaloid content unaltered indefinitely.

H. E. F. NOTTON.

**Influence of source and time of collection on *Rhizoma filicis*.** L. KOFLER and E. MÜLLER (Arch. Pharm., 1930, 268, 644—652).—Contrary to general opinion, no regular differences in the content of extractive matter and of crude filicin, or in toxicity towards fish, can be detected between samples of this drug gathered at widely-differing altitudes, or between spring- and autumn-gathered samples. Among 22 autumn- and 6 spring-gathered samples the greatest variations in biological activity, using filmarone as standard, are less than 50% of the mean. An indication that the active principle of the drug gradually undergoes a change which cannot be detected by the usual chemical assays is given by the examination of 6 samples, 4—20 years old, which were indistinguishable in extractive and crude filicin content from the fresh drug, but were from 2 to 10 times less active biologically. Of the 22 autumn-gathered samples only 3 complied analytically with the requirements of the D.A.B. VI, which are regarded as too stringent.

H. E. F. NOTTON.

**Histo-chemical detection of cetraric acid in *Lichen islandicus*.** M. PRONER (Pharm. Zentr., 1931, 72, 227).—Cetraric acid is detected by the appearance of an intense brownish-red coloration in the central (gonidial) portion of the fibre when it is treated for 2—3 min. with 25% ammonia solution and then with 5% ferric chloride in 50% alcohol.

H. E. F. NOTTON.

**Micro-m.p. determinations with drugs.** L. KOFLER and W. DERNBACH (Arch. Pharm., 1931, 269, 104—114).—The m.p. under the microscope, using the apparatus of Kofler and Hilbck (cf. A., 1931, 189), is recorded for each substance for which the m.p. is given in the D.A.B. VI. The values are, except for substances containing loosely combined water, in good agreement with those determined by the usual method. Most of the substances examined sublime in characteristic forms, the sublimation temperatures being all higher than those recorded by Kempf (A., 1923, ii, 216).

H. E. F. NOTTON.

**Colorimetric determination of strychnine in pharmacopœial preparations.** A. FRANÇOIS (Bull. Soc. Pharm. Bordeaux, 1930, 68, 158—163; Chem. Zentr., 1931, i, 823).—The procedure is based on comparison with a standard of the colour produced by boiling with zinc amalgam and hydrochloric acid, cooling, and adding sodium nitrite solution. Suitable dilutions for pharmacopœial preparations of nux vomica are mentioned.

A. A. ELDRIDGE.

**[Quantitative] reaction for capsaicin.** K. VON FODOR (Z. Unters. Lebensm., 1931, 61, 94—100).—A clear extract (5 c.c.  $\equiv$  1 g. of sample) of the powdered sample in dry acetone is gently shaken with 9 drops of hydrochloric acid and 0.1 g. of ammonia vanadate, when a colour results varying from blue (for 0.08%) through green to green-brown (for 0.01% of capsaicin, the limit of concentration). The brown tint is derived from oxidation of carotene etc. from the sample. Addition of 20 drops of a 1% solution of vanadium oxychloride in carbon tetrachloride to 10 c.c. of an extract

in dry ether gives a more sensitive reaction; the reagent is stable. By means of a range of colours produced from known amounts of capsaicin the reaction is made quantitative. Capsaicin was absent from Spanish pepper, 0.01% or less being found in refined Hungarian paprikas. The blue compound was shown to be *vanadylcapsaicin*,  $C_{18}H_{26}NO_3 \cdot VOCl_2$ .

J. GRANT.

**Determination of santonin in "*Trochisci santonini*."** P. J. CLAUS (Pharm. Weekblad, 1931, 68, 414—424).—The sample is dried at 105° and extracted with ether for 4—5 hrs.; the residue after distillation of the ether is mixed with 0.5 g. of solid paraffin and refluxed with 50 g., and then with 15 g., of ethyl alcohol for 30 min., and the cooled solutions are filtered. The filtrate is shaken with about 15 g. of light petroleum (b.p. 40—70°), and the alcoholic layer, after separation, is heated to boiling to remove dissolved petroleum and is neutralised to phenolphthalein with 0.1N-alkali. The solution is then refluxed with 10 c.c. of 0.1N-alkali for 20 min. and, on cooling, the excess of alkali is titrated with 0.1N-hydrochloric acid. After compensation, from a blank determination, for alkali derived from the apparatus, the error does not exceed 2%. The method of the D.A.B. VI is criticised.

H. F. GILLBE.

**Distillation of essential oils.** V. I. VARENTZEV (J. Chem. Ind., Moscow, 1929, 6, 1602—1604).—The extraction of essential oils from seeds by steam-distillation is expressed by  $S = a/(1-r)$  and  $l = ar^{n-1}$ , where  $a$  is the amount of oil extracted during the first hour and  $r$  is the ratio for two consecutive hours.

CHEMICAL ABSTRACTS.

**Oleonaphtha as absorber of ethereal oils from distillation water.** V. I. VARENTZEV (J. Chem. Ind., Russia, 1929, 6, 966—967).—The condensed water containing droplets of essential oils is passed through a layer of oleonaphtha, whereby a saving of 1.77% of the product is effected. With terpeneol the increase in yield was 3.33%.

CHEMICAL ABSTRACTS.

**Essential oil of fennel.** N. SOBYANIN and S. SAAKOV (Masloboino Zhir. Delo, 1929, No. 6, 34—36).—The following variations were observed with oil cultivated in the Krasnodar district from various kinds of fennel: yield 1.21—5.3%,  $\alpha$  +5° to +20.3°,  $n$  1.528—1.5405, f.p. 3—12°.

CHEMICAL ABSTRACTS.

**Rosenthaler's reaction for peppermint oil.** I. M. KORENMAN (Pharm. Zentr., 1931, 72, 273—276).—The intensity of the colour formed by the addition of Rosenthaler's reagent (1% vanillin solution in concentrated hydrochloric acid) to peppermint oil is in inverse ratio to the content of menthol, which alone gives no colour. The colour is due to the presence of hydrocarbons. Both menthol and peppermint oil give colour reactions with a 1% vanillin solution in sulphuric acid ( $d$  1.8), which is probably due to menthene and dimenthene formed by the dehydrating action of the acid on menthol. Various colour reactions and their sensitivities are described.

E. H. SHARPLES.

**Medicinal carbon.**—See II. **Calcium sulfuratum solutum.**—See VII. **Oil of *Sinapis dissecta*.**—See XII. **Caffeine in coffee etc.**—See XIX. **Alkyl *p*-hydroxybenzoates for sterilisation etc.**—See XXIII.



See also A., May, 561, Colloidal silver and gold. 588, Detection of silver. Lead in tartar emetic. 612, Polycyclic aromatic hydrocarbons. 627, Fluoran derivatives. 629, Local anæsthetics. 636, Aconitine. Derivatives of *p*-arsanilic acid. 637, Water-soluble organomercury compounds. 639, Determinations of nicotine and pyramidone. Thalleioquinine reaction. Detection of alkaloids in plants. 651, *Digitalis* leaves. 656, Antipneumococcic and antimeningococcic horse sera. Diphtheria toxin. 658, Pure male hormone. 661, Analysis of tobacco smoke.

## PATENTS.

Manufacture of easily soluble salts of benzylmorphine [peronine]. CHEM. FABR. VORM. SANDOZ (B.P. 343,397, 7.5.30. Ger., 24.5.29).—The lower alkylsulphonates (*e.g.*, methane- and ethane-sulphonates) of benzylmorphine are crystalline salts, giving stable aqueous solutions suitable for injection. C. HOLLINS.

Preparation of [therapeutically valuable] silicyl compounds. H. P. KAUFMANN (B.P. 343,165, 15.11.29. Ger., 16.11.28).—Hydroxycarboxylic acids, esters, or salts are treated with a silicon halide (tetrachloride), especially in presence of pyridine or other base; or the salts are used in a solvent (pyridine or ether); or the esters are heated with alkyl silicates. The silicyl derivatives of hydroxylated higher fatty acids (ricinoleic or dihydroxystearic acid) are soluble in fats and in ammonia. Silicyl derivatives of ethyl ricinoleate, ricinoleic acid (calcium salt and dibromide), dihydroxystearic acid, lactic acid, and methyl and amyl silicylates are described. C. HOLLINS.

Manufacture of benziminazolonestibinic acids. I. G. FARBENIND. A.-G. (B.P. 343,072, 7.11.29. Ger., 7.11.28).—*o*-Phenylenediaminestibinic acids are phosgenated, or a diazotised aminobenziminazalone is treated with antimonious acid. Examples of starting materials are: 3-amino-4-methylaminobenzenestibinic acid (from 1-chloro-2:4-dinitrobenzene condensed with methylamine, partly reduced with sodium sulphide, acetylated, reduced, diazotised, and treated with antimonious acid in glycerin and aqueous sodium hydroxide, and finally hydrolysed); *o*-phenylenediamine-4-stibinic acid (from diacetylated 4-nitro-*o*-phenylenediamine); 6-chloro-*o*-phenylenediamine-4-stibinic acid (from 6-chloro-1:2:4-trinitrobenzene); 5-aminobenziminazalone and its 1-methyl, 1-ethyl, and 7-chloro-derivatives. C. HOLLINS.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., May, 541, Sensitivity of photographic plates. 579, Developed films. Ripening. Silver bromide. 592, Filters to reproduce sunlight and daylight. 593, Spectrophotometers.

## PATENTS.

Manufacture of photographic surfaces for the bleaching-out process. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 343,212, 2.12.29).—Basic nitrogen-free oxonium dyes other than anthocyanins are used in place of flavindulines for yellows in the bleaching-out process. Examples are 3:7-dihydroxy-

10-methylxanthoxonium chloride, 3:7-dimethoxy-10-phenylxanthoxonium bromide. The latter is used, *e.g.*, with 5-phenylamino-9-diethylamino-2:1-naphthaphenoxazine chloride and 3-methylamino-7-dimethylamino-5-phenyl-2:8-dimethylphenazine chloride, the coloured print being fixed with a 3:1 mixture of benzine and anisole. C. HOLLINS.

Photomechanical printing surfaces. H. WADE. From A. R. GOLRICK (B.P. 346,672, 14.1.30).

[Half-tones on] paper etc.—See V. Cellulose acetate films. Compositions containing cellulose derivatives etc.—See XIII.

## XXII.—EXPLOSIVES; MATCHES.

See A., May, 574, Colloidal powders. 619, Detonation of benzoyl peroxide.

## PATENT.

Fireworks or pyrotechnic devices. J. PAIN & SONS, LTD., and A. W. MILHOLLAND (B.P. 347,003, 25.4.30).

## XXIII.—SANITATION; WATER PURIFICATION.

Alkyl *p*-hydroxybenzoates for preservation, sterilisation, and disinfection. T. SABALITSCHKA (Arch. Pharm., 1930, 268, 653–673).—The phenol coefficients of the esters towards *Staphylococcus aureus* increase with the size of the alkyl group from 2.6 for the methyl ester to 83 for the benzyl ester. All have a very low toxicity to animals. The organisms which are invariably present in distilled water and pharmaceutical solutions of inorganic substances and alkaloids when these are preserved under ordinary conditions are killed within a few days by the addition of 0.15–0.2% of the methyl ester, or of 0.1% of a 1:1 mixture of the methyl and propyl esters. The propyl ester and its sodium salt kill *S. aureus*, *B. coli*, and *B. paratyphosus* in 1 day at concentrations of 0.06% and 0.07%, respectively, and are applicable to the sterilisation of injection and ophthalmic solutions. Powders are also sterilised when the ester is thoroughly mixed with them by gentle heating ("Tyndallisation") and the products act as powerful antiseptics when brought into contact with bacterial cultures or wound surfaces.

H. E. F. NOTTON.

Brewing water.—See XVIII.

See also A., May, 589, Determination of mercury in air. 622, Antiseptic derivatives of indan-1:3-dione. 643, Determination of alkali metals in seawater. 656, *B. coli* in water. 657, Sodium hydroxide, carbonate, and phosphate as germicides.

## PATENTS.

Drying of sewage sludge. W. W. HARRITT, Ass. to L. L. KEENE (U.S.P. 1,779,810, 28.10.30. Appl., 14.11.27).—Partly-dried sludge, carried by an endless belt conveyor or on trays stacked on trucks, is passed through a heat-insulated tunnel which is traversed in the opposite direction by a current of hot air. The waste products are discharged through a stack and the dry sludge is crushed and delivered into a storage bin by compressed air for use as fertiliser. C. JEPSON.

Filter bed.—See I; Gas from garbage.—See II.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 3 and 10, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Cheaper power for the chemical industry.** W. S. JOHNSTON (Ind. Eng. Chem., 1931, 23, 474—478).—The reorganisation of the power plant of a fertiliser works is described. It included increase of boiler pressure, the use of bleeder turbines for current generation, and of exhaust steam for heating services. The design of a modern steam boiler for solid fuel with mechanical stoker and preheated air under forced draught is described. This is also used in connexion with a back-pressure turbo-generator. In both plants zeolite-softened water, de-aerated by preheating, is used.

C. IRWIN.

**Evaporation of water by hot, dry air.** R. H. NEWTON and T. C. LLOYD (Ind. Eng. Chem., 1931, 23, 530—532).—Air, preheated electrically, was passed up a tower in countercurrent to a water spray, the water collecting at the base being circulated by a pump. The influence of the rates of air flow and of water circulation, of the form of water spray, and of the temp. of the hot air on the humidity of the air leaving the top of the tower was studied. The rate of water flow was found to be of greater importance than were the other variables. Curves are given.

H. INGLESON.

**Continuous control of the salt content of boiler feed-water by visual conductivity measurement.** K. SCHIERJOTT. Also E. ROTHER and G. JANDER (Z. angew. Chem., 1931, 44, 312—314).—SCHIERJOTT maintains that Rother and Jander's apparatus (A., 1930, 1548) is unsuited for the measurement of the low salt contents required for modern very high boiler pressures, that the necessity for accurate temp. control is a great difficulty, and that the use of a Wheatstone bridge makes results dependent on a uniform e.m.f. Further, that other and better instruments for the purpose have been available for years. ROTHER and JANDER reply that their intention was to produce a lower-priced instrument than those at present obtainable; they contradict Schierjott's criticisms generally and point out that automatic e.m.f. control is provided.

C. IRWIN.

**Metallic ions as catalysts for removal of sulphur dioxide from boiler-furnace gases.** H. F. JOHNSTONE (Ind. Eng. Chem., 1931, 23, 559—561).—Owing to the low concentration of  $\text{SO}_2$  and  $\text{SO}_3$  in the flue gases even from the burning of a coal rich in S, the washing of the gases requires very large amounts of  $\text{H}_2\text{O}$ . Experiments were made with a suspension of  $\text{CaCO}_3$  as well as with solutions containing  $\text{Fe}^{+++}$  and  $\text{Mn}^{+++}$ . The catalytic effect of  $\text{Fe}^{+++}$  in promoting the oxidation of  $\text{SO}_2$  to  $\text{H}_2\text{SO}_4$  by dissolved  $\text{O}$  is somewhat less than that of  $\text{Mn}^{+++}$ , but the capacity of the solution for dis-

solving  $\text{SO}_2$  is greatly increased by circulating it over scrap Fe. The activity of  $\text{Mn}^{+++}$  is completely inhibited by the presence of a trace of Cu.

H. INGLESON.

**Effect of boiler scale on heat transmission and utilisation.** A. POLLITT (J. Inst. Fuel, 1931, 4, 285—286).—The coeff. of heat conductivity of boiler scale is 1.3—2.0 B.Th.U./sq. ft./hr./ft. $^{\circ}$ F. Scale  $\frac{1}{8}$  in. thick reduces boiler efficiency by approx. 2%, or even less if the scale is dense, but the decreased heat conductivity may cause the temp. of the boiler metal to rise so high that it creeps and failure may ensue.

D. K. MOORE.

**Control of moisture content of air and wood in fresh-air chambers.** I. HATFIELD (J. Agric. Res., 1931, 42, 301—305).—An apparatus is described in which the relative humidity (R.H.) is maintained at the required percentage by means of various salts in contact with their saturated solutions. Salts capable of giving a range of R.H. between 2 and 98% are described.

E. HOLMES.

**Methyl alcohol antifreeze and methyl alcohol poisoning.** W. P. YANT, H. H. SCHRENK, and R. R. SAYERS (Ind. Eng. Chem., 1931, 23, 551—555).—An interim report of an investigation by the U.S. Bureau of Mines into the possible dangers to the public employing MeOH as an antifreeze in motor-car radiators and to the workers engaged in its manufacture. It is estimated that  $4 \times 10^6$  gals. were used during the past winter for this purpose in the U.S.A. Experiments conducted on animals seem to show that there is no hazard to health attending reasonable use of MeOH for an antifreeze either by absorption through the skin or by inhalation of the vapour; 1—2 oz. of MeOH taken into the stomach may cause blindness and death.

H. INGLESON.

**Causticisation and filtration.**  $\text{Na}_3\text{PO}_4$ . Solid  $\text{CO}_2$ .—See VII. Acid-proof tanks and towers.—See IX. Cracking of boiler plate. Corrosion problems.—See X. Heating elements.—See XI. Malt-extract evaporators.—See XVIII. Utilisation of refuse.—See XXIII.

### PATENTS.

**Rotary-hearth furnace.** A. D. DAUCH, Assr. to G. J. HAGAN Co. (U.S.P. 1,782,956, 25.11.30. Appl., 20.3.26).—A rotary, annular-hearth furnace has the operative surface of the hearth composed of a number of tilting trays. Means are provided for tilting the trays in succession when they are opposite the discharge opening, such means being independent of the rotation of the hearth and, in fact, preferably operated when the hearth is momentarily stopped.

B. M. VENABLES.

\* The remainder of this set of Abstracts will appear in next week's issue.

**Furnaces fired with pulverised fuel.** INTERNAT. COMBUSTION ENG. CORP., Assees. of M. FRISCH and E. LUNDGREN (B.P. 345,962 and Addn. B.P. 346,809, [A] 29.11.29, [B] 19.12.29. U.S., [A] 3.1.29, [B] 5.2.29).—In (A) the bottom of the combustion chamber is formed of steam-evaporating tubes which are sufficiently close together to retain a bed of slag the upper part of which remains molten, owing to the impingement of flames downwardly directed during primary combustion. An overflow for molten slag is provided. The upper part of the combustion zone is defined by converging tubes spaced sufficiently far apart for the entry of fuel and air, a limited amount of air being admitted with the fuel to maintain the slag in a reducing atmosphere; the secondary air is admitted higher up, preferably above the restriction formed by the converging tubes. In (B) a method of preventing the overflowing slag from freezing in the spout is described. B. M. VENABLES.

**Tube-still furnace.** H. THOMAS, Assr. to SUN OIL Co. (U.S.P. 1,782,885, 25.11.30. Appl., 6.7.27).—In a still comprising a number of long tubes heated by combustion gases, the combustion is effected in a Dutch oven not immediately beneath the tubes, and the latter are supported at intermediate points of their length by a system of posts, main girders, and smaller beams, being slung by links from the girders and from each other.

B. M. VENABLES.

**Roasting apparatus.** R. H. TALBUTT and F. A. ALLNER (U.S.P. 1,782,946, 25.11.30. Appl., 18.4.29).—The material is placed in a rotating drum provided with inclined stirring blades and is heated by radiation from electrical resistors within the drum; the latter are protected by a sloping roof which affords additional heating surface for material that is lifted and slides down it.

B. M. VENABLES.

**Drying of minerals and other substances [e.g., coal].** C. M. SMITH (B.P. 346,783, 17.1.30).—The material is dried while being lifted in an elevator having swinging buckets. Arrangements are made to tip the contents of a bucket into the next below at a number of desired points, and to permit this only every other bucket is charged at the bottom. The return run of the elevator is in the same casing and subjected to the same current of hot gases, so that the buckets are pre-heated.

B. M. VENABLES.

**Combined pneumatic drying and subsequent pneumatic heating of materials.** P. ROSIN (B.P. 346,541, 7.3.30. Ger., 9.3.29).—Divided material is introduced into a later portion of a conduit conveying a stream of hot gases, and the mixture passes through a preliminary separator, whence coarse material drops out into a disintegrator. The gas and finer material continue to a cyclone separator whence the gas is discharged in a cool saturated condition, whilst the finer material from the cyclone is passed to the earliest part of the hot gas conduit and, after passing through a zig-zag or other device giving sufficient time to effect the heating of the mixture, is separated in a second cyclone, the gas passing on to the point where the material is fed.

B. M. VENABLES.

**Combined spray dryer and burner.** I. HECHENBLEIKNER, Assr. to CHEM. CONSTRUCTION CORP. (U.S.P.

1,782,822, 25.11.30. Appl., 18.5.27).—The hot gases for a spray dryer are produced in a combustion chamber formed in a false roof of the spray chamber, the spraying apparatus being situated in an axial passage through the roof, which also permits cold air to enter.

B. M. VENABLES.

**Treatment of grainy material. [Drying of cereal grains in silos.]** H. EDHOLM (B.P. 346,386, 31.12.29. Swed., 2.1.29).—Grain or other material in a silo or other container through which it moves more or less slowly is dried by air forced into it underneath A-shaped cross-girders which are arranged in a number of tiers so that the intermediate inoperative zones are several times longer than the zones subjected to the drying. Either cool or hot air may be used according to its humidity, or preferably automatic hygrometric control may be provided to start the drying when the weather is dry enough for unheated air to be effective.

B. M. VENABLES.

**Catalytic apparatus.** I. HECHENBLEIKNER, Assr. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,782,824, 25.11.30. Appl., 18.5.28).—A catalyser for exothermic reactions is provided with a main catalyst mass, which is automatically cooled by the entering gases, and with one or more auxiliary masses. The temp. of the gas while passing from one catalyst to another is regulated by external heating or cooling means which are controlled by thermostats buried in the respective preceding catalyst mass.

B. M. VENABLES.

**Manufacture of heat-insulating material [e.g., "85% magnesia"].** H. W. GREIDER, Assr. to PHILIP CAREY MANUFG. Co. (U.S.P. 1,782,383, 18.11.30. Appl., 19.2.26).—A semi-fluid aq. paste containing MgO (alone or together with kieselguhr, CaCO<sub>3</sub>, asbestos, etc.) and a "film-forming and stabilising agent" (e.g., sulphonation products, alkali soaps, etc. of saturated or unsaturated fatty acids above C<sub>15</sub>, or saponin, alkali resins, etc.), is blown with air and moulded at 25–50 lb./in.<sup>2</sup> to expel most of the H<sub>2</sub>O; after removal from the moulds the articles are dried. L. A. COLES.

**Hammer mill.** C. A. JAMISON, Assr. to PENNSYLVANIA CRUSHER Co. (U.S.P. 1,781,904, 18.11.30. Appl., 20.6.28).—A mill is described in which preliminary crushing is effected by the projection of hammers through grid bars which form the bottom of the feed hopper.

B. M. VENABLES.

**Hammer crusher.** G. W. BORTON, Assr. to PENNSYLVANIA CRUSHER Co. (U.S.P. 1,781,891, 18.11.30. Appl., 6.7.28).—The impact grid of a disintegrator is wound up into its operative position by means of a chain and hand-worked windlass, and is held by a pawl which has a frangible safety pin. B. M. VENABLES.

**Pulverising mills.** INTERNAT. COMBUSTION ENG. CORP., Assees. of C. E. NEEDHAM (B.P. 346,428, 6.1.30. U.S., 5.1.29).—A form of bearing for and method of lubrication of the rollers of a mill of the Raymond type is described.

B. M. VENABLES.

**Roller pulveriser.** R. E. DUNHAM, Assr. to DUNHAM Co. (U.S.P. 1,782,201, 18.11.30. Appl., 23.2.28).—Rollers suitable for agricultural and other purposes are assembled from a number of spoked discs of which the edges are V- or cusp-shaped and are wavy when viewed

edgeways, but not when viewed from an axial direction. Each roller has two sets of spokes staggered in relation to each other, and the rollers are freely mounted on their axles. B. M. VENABLES.

**Grinding or crushing mills.** F. E. MARCY (B.P. 346,665, 7.1.30).—A mill of the ball, pebble, or rod type is provided with a manhole in the circumference of the shell for emptying purposes; the manhole is not provided with a small section of lining attached to it, but is covered by a section of the main lining. Access to the interior of the mill is obtained through another manhole in one of the end walls. The discharge grating is supplied with additional water by means of a pipe through the outlet trunnion, and the supply is kept as local as possible to avoid diluting the main bulk of pulp. B. M. VENABLES.

**Machines, such as ball mills, for the reduction of materials.** J. E. KENNEDY (B.P. 346,375, 3.10.29).—A ball mill is rendered silent by supporting an inner shell from an outer shell by means of more than one layer of porous or cellular, sound-insulating, and, if desired, heat-insulating, material, each layer having different porosity, the outer layer being preferably less porous than the inner. B. M. VENABLES.

**Grinder for pigments etc.** E. DE ROZIERES (U.S.P. 1,781,808, 18.11.30. Appl., 23.5.28).—The rotor of a centrifugal grinder is surrounded by grinding rollers and guides to deflect the material into the nips between the rollers and co-acting grinding blocks. B. M. VENABLES.

**Grading and separation of granular materials.** J. LORD (B.P. 346,481, 22.1.30).—Grain or granular material preliminary to grading by a pneumatic process is passed between fixed and rotating abrasive and/or brushing surfaces; preferably a rotating brush runs against, first, a concave block of emery or similar material and, second, a concave brush. B. M. VENABLES.

**Separation of dry materials.** C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 346,266, 7.11.29).—A pneumatic shaking table has a surface free from riffles, one straight edge (unless the table is double), substantially parallel to the motion, over which all the products are delivered, and one (or two) inclined banking bars. B. M. VENABLES.

**Rotary separating or screening apparatus.** N. H. R. GRIFFIN (B.P. 346,330, 8.10.29).—The apparatus comprises a cage of annular bars which are alternately fixed by longitudinal bolts and loosely mounted on rollers thereon. B. M. VENABLES.

**Preparing wet [damp] mixtures.** J. C. MAC-ILDOWIE, ASSR. to ASBESTOS WOOD & SHINGLE CO. (U.S.P. 1,781,728, 18.11.30. Appl., 29.10.27).—Material which is neither wet nor dry, *e.g.*, asbestos-cement paste, is thoroughly mixed and disintegrated by subjecting it to the shearing action of perforated plates reciprocating in opposite directions and spaced somewhat apart from each other. The material is preferably fed by gravity and its general flow is perpendicular to the plates. The divided particles are in convenient form for transfer by conveyors and for tamping in moulds. B. M. VENABLES.

**Apparatus for preparing viscous mixtures.** W. L. SMITH, ASSR. to VISCOSE CO. (U.S.P. 1,782,370, 18.11.30. Appl., 17.8.29).—At least two mixing tanks having jackets for temp. control, a pump, and a grinding mill are connected by conduits and multi-way cocks so that the material may be passed in various circuits. B. M. VENABLES.

**Filter apparatus.** R. S. BUTLER, ASSR. to SOUTHWESTERN ENG. CORP. (U.S.P. 1,780,905, 11.11.30. Appl., 12.12.28).—A number of filter leaves are attached to a chain conveyor and surrounded by frame-like supports which are joined together by bellows-like flexible members so that when the filters are on the straight upper run of the conveyor the frames and bellows form tanks for prefilter or wash liquid. The filter leaves are connected by nipples to a flexible belt which runs over a fixed flexible member, forming seals on the principle of a vacuum cup between one or more vacuum chambers and the belt. The motion of the apparatus is preferably intermittent, but filtration is continuous. B. M. VENABLES.

**Filter presses for treatment of potters' clay etc.** E. W. MALKIN, G. E. LEESE, and MANOR ENG. CO., LTD. (B.P. 346,964, 27.3.30).—A method of closing a press by means of a worm and screw is described. B. M. VENABLES.

**Filter device.** A. SCHREMPF (U.S.P. 1,783,143, 25.11.30. Appl., 4.1.27. Ger., 9.1.26).—The filter medium is composed of superposed sheets punched into holes similar to a nutmeg grater, the jagged teeth acting both as filters and as spacers for the sheets. The holes are evenly distributed over the sheets and are staggered in adjacent sheets. B. M. VENABLES.

**Filter.** G. F. THOMAS and O. J. HERB, ASSRS. to ALEMITE CORP. (U.S.P. 1,781,964, 18.11.30. Appl., 30.11.25).—An easily dismembered strainer suitable for insertion in a petrol pipe is described. B. M. VENABLES.

**Centrifugal extractor.** H. KRANTZ (U.S.P. 1,781,984, 18.11.30. Appl., 18.8.28. Ger., 24.2.28).—A timing device with interlocks for the power, brake, and cover is described. B. M. VENABLES.

**[Laboratory] centrifugal extractor.** F. RODLER (U.S.P. 1,782,179, 18.11.30. Appl., 2.11.27).—Apparatus suitable for extracting road materials etc. is described. The sample and a solvent are placed in the bowl and the solution is centrifugally filtered through paper on the underside of a flat cover. B. M. VENABLES.

**Distilling apparatus.** W. S. ELLIOTT (U.S.P. 1,782,959, 25.11.30. Appl., 27.3.23).—The apparatus comprises a water heater and one or several evaporating chambers through which the water passes in series at progressively decreasing temp., separate condensers being provided for the vapours so that the first stage may be used mainly for de-aeration. Unevaporated water is returned to the heater. B. M. VENABLES.

**Distillation of liquids.** J. DELATTRE-SEGUY, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,782,810, 25.11.30. Appl., 28.6.26).—The liquid is allowed to flow in a thin layer down a trough and is heated from above by radiation only, from a conduit containing a

stream of molten metal; the latter effects further heating of the vapours evolved by direct contact.

B. M. VENABLES.

**Evaporator.** H. W. HOW, Assr. to BUFFALO FOUNDRY & MACHINE CO. (U.S.P. 1,782,143, 18.11.30. Appl., 12.1.27).—A method of forced circulation by an internal rotor is described.

B. M. VENABLES.

**Production of foam by mechanical means.** J. C. HANSEN-ELLEHAMMER (B.P. 347,048, 12.6.30. Denm., 2.11.29).—A foam-producing material,  $H_2O$ , and air are admitted together to a rotary pump, whence they are expelled through several layers of wire gauze or similar material tangentially into a cylindrical vessel in which the whirling action causes the finer foam to work to the axis, at which point the outlet is situated.

B. M. VENABLES.

**Nebulising of liquids.** W. E. SHORE (B.P. 346,747, 3.1.30).—A mixture of a gas and liquid, *e.g.*, air and petroleum fuel for an internal-combustion engine, obtained by known spraying means is subjected to a reduction of pressure followed by sudden restoration of normal pressure in a machine resembling a rotary compressor. [Stat. ref.]

B. M. VENABLES.

**Fluid-treating apparatus.** J. A. MACKENZIE (U.S.P. 1,782,735, 25.11.30. Appl., 21.3.27).—A form of tray for a bubbling tower is described. A pair of parallel upstanding baffles are provided, extending the full length of chords, and the downflow is effected in the outer zones from either side alternately, the central zone being given up to bubbling; there are no caps, but the perforations for gas flow have upturned tongues.

B. M. VENABLES.

**Liquid of low f.p. for cooling [of automobile radiators] and for sealing vessels [gasometers] and the like.** O. JORDAN, Assr. to I. G. FARBERNIND. A.-G. (U.S.P. 1,780,927, 11.11.30. Appl., 9.3.27. Ger., 5.3.26).—The use is claimed of a mixture of  $H_2O$ , a glycol monoalkyl ether, and, if desired, a glycol.

L. A. COLES.

**De-aeration of boiler feed-water.** W. F. CAREY, G. H. C. CORNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,523, 22.2.30).—The water is allowed to trickle down a tower (with filling) while subjected to a vacuum which is produced by a steam ejector drawing from the top of the tower.

B. M. VENABLES.

**Liquid and gas contact apparatus.** F. H. WAGNER, Assr. to BARTLETT HAYWARD CO. (U.S.P. 1,782,862, 25.11.30. Appl., 14.3.28).—In a tower providing countercurrent flow of liquid and gas and divided into stages, the flow of gas at each stage is upwards through a large central aperture in an impermeate plate, and downwards under a hood to below a perforated plate through which it passes to the next stage. An additional diaphragm of wire mesh or the like may be placed above the perforated plate. Scrapers operated by a vertical shaft are provided to keep the perforated plates free from solid matter.

B. M. VENABLES.

**Filtering and dust-collecting apparatus.** I. HECHENBLEIKNER, Assr. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,782,823, 25.11.30. Appl., 17.4.28).—The apertures at the ends of a vertical casing are used only for charging and discharging a granular filter medium

which is gradually and continuously renewed. The gases to be filtered are admitted and exhausted by means of hollow grid-like members at points intermediate the length of the casing.

B. M. VENABLES.

**Washing apparatus for effecting dust removal from gases.** G. H. C. CORNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,750, 9.1.30).—In an apparatus as described in B.P. 321,268 (B., 1930, 41) the irrigated surfaces are coated with vitreous enamel.

B. M. VENABLES.

**Apparatus for gas analysis.** H. H. DOW, Assr. to DOW CHEM. CO. (U.S.P. 1,782,032, 18.11.30. Appl., 28.7.26).—A difference in sp. gr. between two gases is measured by their inertia. The sample and standard are brought to the same temp. and delivered in alternate equal puffs by the opposite sides of the piston of a pump. They impinge from opposite directions on to a vaned wheel which operates a pointer through gearing; to prevent the pointer winding continuously in one direction, a cam is attached which deflects the jet of one gas from or towards the tangent to the impact wheel. Backlash is left in the gearing to prevent continual small oscillations of the pointer.

B. M. VENABLES.

**Agitation and aeration of liquids.** W. N. JONES (B.P. 346,815, 13.1.30).—See U.S.P. 1,765,338; B., 1931, 140.

**Oil-fired heating apparatus for use with melting pans and the like.** A. J. TIGHT and F. H. EBNER (B.P. 347,859, 14.4.30).

**Non-corrosive alcoholic solution.**—See III. Filter paper and fabrics.—See V. Silica compounds etc.—See VII. Lumber kiln.—See IX. Heating furnace. Roller-mill guide.—See X. Precipitating particles from gases.—See XI. Pigments for painting etc.—See XIII. Rubber goods etc.—See XIV. Treatment of massecuites. Sugar dryer.—See XVII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Experimental determination of bound oxygen in organic compounds [*e.g.*, coal].** M. DOLCH and H. WILL (Brennstoff-Chem., 1931, 12, 141–146, 166–169).—Values of the O content of organic compounds determined by Ter Meulen's method were low, due principally to the incomplete hydrogenation of the CO formed. A method of determining the O content of coal or coke, based on the determination of the total vol. and  $CO_2$  content of the combustion gases when a known wt. of the substance is burned completely in air or O, failed because the unavoidable error in the  $CO_2$  determination led to considerably larger errors in the calc. O content. Much better results were obtained by burning the substance completely in a known vol. of O and determining the amount of O used up and the amounts of  $CO_2$  and  $H_2O$  formed. The apparatus, which formed a closed circuit, comprised a  $SiO_2$  combustion tube, which could be heated electrically, absorption vessels for  $CO_2$  and  $H_2O$ , storage and measuring vessels for the O, two manometers, and a gas-circulating pump. The substance, contained in a Pt boat, was introduced into one end of the combustion tube, the remainder of which was packed with broken

SiO<sub>2</sub>. After complete combustion of the sample, the apparatus was cooled to the original temp. and the vol. of residual O was measured. Determinations were made also of the H<sub>2</sub>O content of the substance, preferably by the "cryohydrate" method (B., 1931, 97), and, where necessary, of the combustible S. A number of analyses of coals, cokes, and tars, and the O distribution in the carbonisation products of a coal and a lignite, are tabulated.

A. B. MANNING.

**Preparation of coal-dust sections and their analysis by means of the projected micro-image.**

E. STACH (Brennstoff-Chem., 1931, 12, 147—150; cf. Stach and Kühlwein, B., 1929, 382).—Further experimental details are given of the author's methods of embedding and relief-polishing samples of fine coal. The specimens are examined preferably by projecting a magnified image of the section on to a white screen.

A. B. MANNING.

**Determination of the caking properties of coal in conjunction with the determination of volatile matter.**

D. J. W. KREULEN (Chem. Weekblad, 1931, 28, 318—319).—The method described in "Brandstof-chemie," 1929, is criticised: the use of a Ni crucible is not advisable, the quantity of sand employed causes excessive retardation of cooling, and the sand is too coarse to permit the formation of a homogeneous mixture. A modification of the method, giving satisfactory results for caking ratios up to 10, is described; a Pt crucible is employed and the procedure closely resembles that used for the determination of volatile constituents.

H. F. GILLBE.

**Gasification of bituminous coal in the water-gas producer.** I. J. GWOSDZ (Brennstoff-Chem., 1931, 12, 150—153, 169—171).—Some recent developments in the design of plant for the complete gasification of bituminous coal are briefly described.

A. B. MANNING.

**Formation of high- and low-temperature cokes.**

J. ROBERTS (J. Inst. Fuel, 1931, 4, 255—269).—When coal is carbonised in beehive ovens coking proceeds downwards. After the maximum expansion, in which a 24-in. charge increases to 32 in. in height, shrinkage occurs, particularly in a lateral direction, causing the formation of pillars. These show four structures: stalactitic, stalagmitic, hair-carbon, and fish-rope. The C deposited on the coke by the cracking of the hydrocarbons makes the coke robust and less liable to attack by CO<sub>2</sub>. In the production of by-product coke the charge is heated from the sides, the gases pass outwards and become partly decomposed, and the deposited C gives the coke a graphitic lustre. Cauliflower, columnar, onion-like, and honeycomb structures may be seen. The quality of the coke is influenced by the type and size of coal, temp. of treatment, and width of oven. Gasworks' coke made under static conditions is denser than that made by continuous processes. High-temp. free-burning coke can be made by blending coking and non-coking coal, or, when the latter is not available, part of the former can be rendered non-coking by oxidising at 100—350°. Several examples of low-temp. cokes are described.

D. K. MOORE.

**Gas-producing steel-works' practice.** W. R. ROSSINGTON (J. Inst. Fuel, 1931, 4, 289—291).—Fine

coal should be avoided, but it is economical to use good quality fuel. The blast should be worked with a saturation temp. of 54—60°, or, with a badly clinkering coal, of 60—64°. The use of a regulator is indicated.

D. K. MOORE.

**"Glycerin" gas-drying process.**

A. F. H. KNOWLES (Gas J., 1931, 194, 347—349).—Results obtained with a four-bay washer-scrubber, using glycerin as the drying medium and operating at the gasholder outlet, are described. It was found possible to work down to 75% of glycerin at the inlet and 55% at the outlet before concentration of the liquid was necessary. Further particulars are given for a second plant of the same type.

R. H. GRIFFITH.

**Organic basis for gas purification.**

R. R. BOTTOMS (Ind. Eng. Chem., 1931, 23, 501—504).—Di- and tri-ethanolamines absorb CO<sub>2</sub> and H<sub>2</sub>S from gas at room temp., yielding unstable compounds which decompose at above 50°. A 50% aq. solution of the mixed bases is suitable, and the vol. to be circulated is much less than if Na<sub>2</sub>CO<sub>3</sub> solution be used. A bubble tower is recommended for absorption, provided the gas pressure allows of its use and the absorbent is stripped with steam in a second similar tower with reflux condenser. The reagent forms stable salts with the stronger acids and with SO<sub>2</sub>, for the removal of which it is unsuitable. It is intended particularly for the purification of natural gas for the production of He.

C. IRWIN.

**Detection of very small traces of acetylene.**

E. PIETSCH and A. KOROWSKI (Z. angew. Chem., 1931, 44, 309—312).—As little as  $3.7 \times 10^{-4}$  vol.-% of C<sub>2</sub>H<sub>2</sub> can be detected by precipitation as Cu acetylide if the test is carried out in the following manner:—1 g. of cryst. CuSO<sub>4</sub> is dissolved in a little H<sub>2</sub>O, 4 c.c. of 20% NH<sub>3</sub> solution are added, and then 3 g. of NH<sub>2</sub>OH.HCl are slowly added with shaking and the mixture is diluted to 50 c.c. The reagent will keep for 3 days. The authors' trials were made with C<sub>2</sub>H<sub>2</sub> mixed with H or CO<sub>2</sub>. The reaction is, of course, inhibited by atm. O, or by H<sub>2</sub>S. The characteristic cherry-red coloration of Cu acetylide requires for its good development the relative proportions of NH<sub>3</sub> and NH<sub>2</sub>OH salt indicated above. In great dilution it is best seen by absorption from the reagent on a filter paper. It was found that if small traces of C<sub>2</sub>H<sub>2</sub> were allowed to remain in the evacuated apparatus without diluent gas the apparent concentration was gradually reduced by adsorption on the glass.

C. IRWIN.

**Solubility of asphalt bitumen and asphaltenes in certain mixtures.**

F. J. NELLENSTEYN (Chem. Weekblad, 1931, 28, 313—314).—The author's surface tension theory (cf. B., 1927, 132, 739) of the preparation of a solvent for asphaltenes by mixing two liquids in both of which the asphaltene is insoluble is applied to the preparation of such solvents by mixing Et<sub>2</sub>O and NH<sub>2</sub>Ph, and CH<sub>2</sub>Ph.OAc and C<sub>6</sub>H<sub>6</sub>.

**Shales.** B. HOLMBERG, S. KALLENBERG, and S. PYK (Ingen. Vetensk. Akad. Handl., 1930, No. 101, 5—78; Chem. Zentr., 1931, i, 1206).—Analyses of Swedish shales are recorded. In those affording at least 4% of oil, 27% of the C and 28% of the H are found in the oil,

and 10% and 25–30%, respectively, in the gases; 5–10% of the II forms water. Of the S, 20–30% is found in the gas and 1–2% in the oil. For the crude oil H:C = 1.26–1.48; S 2, O 1–4, PhOH 0.1–1.9%.

A. A. ELDRIDGE.

**Vapour-phase treatment of cracked gasolines in the Vickers cracking unit.** B. P. FRADKIN and D. A. STROM (Nef. Choz., 1930, 18, 445–448).—The gasoline vapour is passed through fuller's earth filters at 230–235°. The yield of final gasoline was 91.1%; it had  $d$  0.737, gum 0.3%, and gave satisfactory corrosion and doctor tests. CHEMICAL ABSTRACTS.

**Gases from crude oil cracked in the vapour phase.** M. B. MARKOVICH and V. V. PIGULEVSKI (Nef. Choz., 1930, 18, 425–444).—The permanent gas, "blau gas," and light gasoline contained, respectively, H 2.4, 0.2, 0; saturated hydrocarbons 49.0, 29.6, 3.0;  $C_2H_4$  25.2, 17.1, 0;  $C_3H_6$  12, 24.3, 4.0; butylene 2.9, 9.0, 35.0; divinyl 2.0, 6.6, 18.0; higher unsaturated hydrocarbons 0, 2.5, 0; N,  $CO_2$ , O, etc. 3.0, 0.4, 0 wt.-%.

CHEMICAL ABSTRACTS.

**Gasoline dopes.** H. C. DICKINSON (Ind. Eng. Chem., 1931, 23, 517–519).—The addition of dopes cannot appreciably affect the volatility at low temp. or the distillation curve of the mixture. Claims for easier starting or improved total volatility cannot therefore be substantiated. Routine tests of the Bureau of Standards on about 150 dopes submitted have shown the latter to be valueless in improving engine performance. Claims for improved power development, apart from suppressing detonation, have no scientific basis. The value of dopes in preventing C deposits is difficult to prove, as C formation is dependent on such factors as carburettor setting and lubrication.

T. A. SMITH.

**Photographic flame studies in the gasoline engine.** L. WITHROW and T. A. BOYD (Ind. Eng. Chem., 1931, 23, 539–547).—A special head, fitted with a quartz glass window and carrying a camera, was fitted to the cylinder of an engine. Flame and pressure records were obtained for knocking and non-knocking fuels. Knocking is shown to be due to a manifold increase in the rate of inflammation within the latter portion of the charge. The results are similar to those obtained by Wheeler and co-workers in experiments with bombs. The conditions in the cylinder as deduced from the pressure curves are in agreement with the photographic evidence. The knock is apparently due to auto-ignition of the residual mixture and may result from the high temp. of that portion of the charge, owing to adiabatic compression. The violence of the knock is determined by the amount of unburnt mixture when these conditions occur. The effect of  $PbEt_4$  is to prevent the extremely rapid inflammation of the latter portion of the charge; it has no effect on the velocity or character of the flame prior to the time at which knock would occur in its absence.

T. A. SMITH.

**Dielectric constants of petroleum and its products.** V. ZHUZE (Azerbaid. Nef. Choz., 1930, No. 12, 88–98).—The dielectric const. of petroleum and paraffins increase with increase in  $d$ , b.p., and mol. wt., and are approx. equal to  $n_D^2$ . For petroleum the temp.

coeff. is negative. The dielectric const. of naphthenic acids increase with increase in  $d$ , b.p., surface tension,  $n_D$ , and mol. wt.

CHEMICAL ABSTRACTS.

**[Electrical] conductivity of petroleum emulsions.** J. Wolk (Przemysl Chem., 1931, 15, 172–180).—A Boryslaw emulsion contained 51%  $H_2O$  (dispersed), 10.7166% Cl, 5.944% Na, 0.7645% Ca, 0.0661% Mg, 0.0194% K, 0.7445% ( $Al_2O_3 + Fe_2O_3$ ), 0.3811%  $SiO_2$ , 0.0159%  $SO_4$ , and 0.0130%  $CO_2$ . The viscosity of emulsions is increased by homogenisation. Conductivity in both neutral and homogenised emulsions is a function of the p.d., the tension of the current passing being approx. proportional to the number of watts. Certain observed deviations from Ohm's law confirm the view that conductivity varies in the vicinity of the electrodes. Conductivity increases with temp. as a result of diminished viscosity.

R. TRUSZKOWSKI.

**Alignment chart for estimating viscosity-gravity constant of petroleum lubricating oils.** W. F. HOUGHTON and J. A. ROBB (Ind. Eng. Chem. [Anal.], 1931, 3, 144–145).—The viscosity-gravity const. as determined by Hill and Coats (cf. B., 1928, 592) has been found useful not only for defining and classifying oils, but also for following refining processes. A chart has been devised so that the const. may be read directly when the sp. gr. and viscosity have been determined.

T. McLACHLAN.

**Analysis of petroleum and its distillates for reducible substances and adsorbable matter.** B. GOSMAN and J. HEYROVSKÝ (Trans. Amer. Electrochem. Soc., 1931, 59, 41–63).—Waves on polarographically recorded current-voltage curves, obtained by electrolysis with a dropping mercury cathode, of  $NH_4Cl$  solutions which have been shaken with a petroleum fraction and freed from dissolved O by means of  $Na_2SO_3$ , serve for the detection and approx. determination of reducible substances. Anomalous results with certain American oils, especially when extracted with NaOH solutions, are attributed to substances (probably naphthenic acids) which are adsorbed on the cathode and inhibit the reaction between O and sulphite. The presence and acidic character of these substances was also shown by their capacity for suppressing peaks on the current-voltage curves. Determination of the quantity of an oil required to halve the height of the peak due to Cu deposition from a dil.  $CuSO_4-H_2SO_4$  solution in MeOH affords a measure of the adsorbable substances in the oil. Comparative data for various fractions of oils from different sources are quoted.

H. J. T. ELLINGHAM.

**Acetic acid.**—See II. "Phenolates."—See III. **Gypsum.**—See VII. **Fuel control in steel industry.**—See X. **Oxidation of oils.**—See XII. **Refuse fuels.**—See XXIII.

#### PATENTS.

**Coking of hydrocarbon material, liquid or liquefiable by heat.** BARRETT Co., Assees. of H. H. BAILEY (B.P. 347,030, 20.5.30. U.S., 22.5.29).—Tar or pitch is distilled in an externally heated retort until a semi-coke of volatile content 7–10% is produced. The external heating is then discontinued and air introduced into the retort in such a manner as to subject the semi-coke to a type of "beehive" carbonisation,



preferably for about 36 hr. A dense coke of low-S, ash, and volatile content is produced.

A. B. MANNING.

**Manufacture of products from carbonaceous materials by treatment with hydrogen or other reducing gases.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 346,689, 6.12.29, 14.12.29, and 30.1.30. Cf. B.P. 332,944 and 338,192; B., 1930, 1057; 1931, 286).—Coals, tars, mineral oils, etc. are treated with reducing gases (H etc.) at raised temperatures and pressures in the presence of a catalyst prepared by absorbing a compound or compounds of Cr, W, U, Mn, Co, Ni, or Fe [e.g.,  $\text{Fe}(\text{OH})_3$  or Prussian blue], in colloidal condition, on activated C or finely-divided brown coal.

A. B. MANNING.

**Production of carbon black.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,680, 12.10.29).—Gaseous or vaporous unsaturated hydrocarbons are passed over catalysts, e.g., metals, or oxides of metals, of the Fe group, at a temp. below that at which complete decomposition of the hydrocarbon occurs, and preferably such that 85–90% of the C therein is converted into C black.

A. B. MANNING.

**Production of carbon and hydrogen chloride.** J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,858, 23.1.30).—Heavy hydrocarbons are cracked in the vapour phase in the presence of HCl, and the products are caused to react with Cl to form C black and further quantities of HCl, as described in B.P. 317,165 (B., 1929, 803).

A. B. MANNING.

**Removal of tar particles prior to recovery of clean oils from coal-distillation gases.** BARRETT CO., ASSEES. OF S. P. MILLER (B.P. 346,805, 18.12.29. U.S., 18.12.28. Cf. B.P. 315,374; B., 1929, 970).—The gases collected from the ovens or retorts in the collector main are led while still hot to a de-tarring chamber wherein they are passed through fine sprays of tar or pitch.

A. B. MANNING.

**Distillation of tar.** BARRETT CO., ASSEES. OF S. P. MILLER (B.P. 346,753, 10.1.30. U.S., 10.1.29).—Two separate supplies of tar are distilled simultaneously, the first by direct contact with a stream of highly heated gases, e.g., hot coke-oven gases, in a non-externally heated still, and the second by indirect contact with the gases leaving the first still. The supplies of tar and gases are so regulated that in the first distillation a pitch of high m.p. (200° or above) is produced, and in the second a pitch of low m.p. and a light oil distillate are the products.

A. B. MANNING.

**Aqueous emulsions of tar.** N. BENDIXEN and J. G. Y. D. MORGAN (B.P. 346,978, 3. and 24.4.30).—Emulsions suitable for the surface-dressing of roads are prepared by using as emulsifier a saccharate formed from a mono- or di-saccharide, in particular Ca or K saccharate, in an amount not exceeding 10 wt.-% of the tar.

A. B. MANNING.

**Preparation of tarry and bituminous solutions [for road-making].** R. ARNOT (B.P. 346,681, 15.10. and 15.11.29).—To tar, bitumen, etc., which naturally may contain unsaturated compounds, a flux or diluent is added in the form of an oil, e.g., topped shale oil, also

containing unsaturated hydrocarbons, which will resinify, polymerise, or condense with the unsaturated compounds in the original material. Portland cement, CaO, or other basic material may be added to the mixture to accelerate drying.

A. B. MANNING.

**Cracking of petroleum oil.** R. T. POLLOCK, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,781,128, 11.11.30. Appl., 9.3.27).—Oil of high b.p. is subjected to a primary cracking treatment and the products are separated into at least three components, e.g., a gasoline and an intermediate oil fraction, and a residuum. The gasoline and residuum are removed from the cracking zone, and the cracking of the intermediate fraction is continued in successive stages, removing at each stage the gasoline fraction and residual products and treating in each succeeding stage only the intermediate fraction of the preceding stage. The cracking conditions at each stage are controlled so that a high yield of low-boiling distillate is obtained.

H. S. GARLICK.

**Refining of hydrocarbons [cracked gasoline].** E. C. HERTHEL, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,781,388, 11.11.30. Appl., 29.2.28).—Cracked gasoline, which will give more than 3% of polymerides on treatment with, e.g., fuller's earth, is passed downwards, in the vapour phase, through a bed of refining agent and then fractionated in a column. The high-boiling polymerides are returned to the cracking plant, where they are used to scrub the cracked vapours and to remove tar.

T. A. SMITH.

**Dehydration of [hydrocarbon] oil.** W. L. PALMER, ASSR. to NAT. DEHYDRATOR CORP. (U.S.P. 1,781,076, 11.11.30. Appl., 4.4.27).—An emulsion is separated into its constituents by passing it over plates which are rapidly oscillated in a direction at right angles to the flow of the emulsion. Spaced plates are mounted on a horizontal axis and rapidly oscillated, the emulsion being distributed over the upper edges of the plates and flowing down between them. It is unnecessary to heat the emulsion.

T. A. SMITH.

**Manufacture of lubricants.** F. S. DENGLE, ASSR. to TEXAS Co. (U.S.P. 1,781,444, 11.11.30. Appl., 15.10.26).—Paraffin or semi-paraffin base crude is distilled until the residue has a flash point of 190–245°, and this residue is then oxidised by means of air at 205–270° until the product melts at 95–150°.

T. A. SMITH.

**Production of chatterless [lubricating] oil.** T. W. DOELL and E. N. KLEMGARD, ASSR. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,781,167, 11.11.30. Appl., 12.10.26).—About 3% of the complex Pb soap prepared by co-precipitation from a mixed solution of the alkali salts of unsaturated fatty acids (e.g., oleic, ricinoleic acids) and naphthenic acids (from petroleum oils) is added to a mineral lubricating oil.

E. LEWKOWITSCH.

**Fractionally extracting petroleum hydrocarbons with alcohol.** T. A. WERKENTHIN, ASSR. to SOLAR REFINING Co. (U.S.P. 1,781,420—1, 11.11.30. Appl., 30.12.27).—The oil is caused to spread in a thin film over trays or perforated plates and comes into contact with alcohol vapours from an extract alcohol still. The undissolved oil and dissolved oil extract pass to a

settling tank, where the bulk of the undissolved oil separates. Final separation is accomplished centrifugally. The extract is passed to a still, the alcohol is distilled off, and the extracted oil passed to storage. The undissolved oil separated in the settling tank and centrifuge is returned for re-treatment. The entire process is operated under a pressure approx. 6–8 lb./in.<sup>2</sup> above atm.

H. S. GARLICK.

**Treatment of liquid partial-oxidation products [of oils].** J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,753,516—7, 8.4.30. Appl., [A] 7.3.21, [B] 2.7.25).—(A) Oxidised oil, *e.g.*, oxidised mineral oil, is fractionally distilled, and higher fractions (above 200°) are sulphonated, the sulphonic acids being extracted with brine and boiled with NaOH to give soaps. (B) The aldehydo-fatty acids present in oxidised oils are removed by conversion into soaps, *e.g.*, by boiling with alkali, and the residual oil is further oxidised.

C. HOLLINS.

**Tube-still furnace. Drying of coal. Filter. Nebulising liquids.**—See I. **Coating compositions.**—See II. **Conversion of acetylene. Removing acids from crude oils. Chlorinated hydrocarbons. Dispersing agents etc.**—See III. **Waterproofing of sheets.**—See VI. **Zeolites.**—See VII. **Mastic asphalt.**—See IX. **Carbon electrodes.**—See XI. **Insecticide.**—See XVI.

### III.—ORGANIC INTERMEDIATES.

**Opium alkaloids as reagents for formaldehyde.** C. C. FULTON (Ind. Eng. Chem. [Anal.], 1931, 3, 200—201).—The opium tests for the detection of  $\text{CH}_2\text{O}$  react best at an acid concentration of 75–85% and not, as usually stated, in conc.  $\text{H}_2\text{SO}_4$ . The delicacy of the test is much enhanced by the addition of traces of  $\text{Fe}_2(\text{SO}_4)_3$  or  $\text{HNO}_3$  as an oxidant; with such addition, apomorphine, codeine, pseudomorphine, and papaverine give the best results, being sensitive to 2–1 p.p.m. in water. The test is not specific as it is given by other aldehydes, although a comparison of colours with several alkaloids makes identification fairly certain.

T. McLACHLAN.

**[Salkovski's] reaction for formaldehyde and its application to the investigation of foodstuffs.** A. VAN DRUTEN (Chem. Weekblad, 1931, 28, 283–288).—The conditions of Salkovski's reaction, and the influence of the concentration of the peptone,  $\text{FeCl}_3$ , and  $\text{HCl}$  has been investigated. The following method of carrying out the test is recommended: 0.1 g. of Witte's peptone is dissolved in 5 c.c. of the sample by shaking and heating just to the b.p.; 0.3 c.c. of 1%  $\text{FeCl}_3$  solution and 5 c.c. of 8N-HCl are added, and the solution is again heated just to the b.p. After 5 min. 10 c.c. of water are added and the colour is observed; a stable blue or violet colour indicates the presence of  $\text{CH}_2\text{O}$ , which, however, is not necessarily present in the original solution. Certain aromatic aldehydes gives a positive reaction.

H. F. GILBE.

**Acetic acid and cellulose acetate in the United States.** E. P. PARTRIDGE (Ind. Eng. Chem., 1931, 23, 482–497).—A considerable amount of AcOH is still manufactured by fermentation of EtOH, but the maximum concentration possible is 13–14%. The crude

product is distilled and used for the manufacture of AcOEt. Wood-distillation processes in which AcOH is produced directly from pyroligneous liquor include the Brewster process, which employs extraction with  $\text{Et}_2\text{O}$ , or more recently with isopropyl ether. The latter reagent yields practically anhyd. AcOH. The Suida process employs an extracting agent with b.p. above that of AcOH, *e.g.*, wood oil. The most modern form of plant used in this process with increased rectification capacity gives 90–95% acid. The MeOH instead of being previously removed as in other extraction processes is carried through the columns as vapour and afterwards condensed. The Shawinigan process using  $\text{CaC}_2$  as starting material is described briefly. Little further development of either synthetic or wood AcOH manufacture is likely in America for some time owing to economic conditions, and both processes are threatened by newer synthetic processes working from EtOH. The consumption of AcOH in various industries is surveyed in detail. The properties and uses of cellulose acetate are described. The Brewster process has been applied to the recovery of AcOH from its dil. solution in the end-liquor. The  $\text{Ac}_2\text{O}$  required is also made from the end-liquors after concentration and conversion into AcONa, using  $\text{SO}_2\text{Cl}_2$ . In Germany a process is operating in which glacial AcOH is vaporised and passed over  $\text{NaPO}_3$  as catalyst at 400–800°. The interaction of AcCl and glacial AcOH may be economical in the near future. Liquid  $\text{SO}_2$  as a solvent in place of glacial AcOH has been recently applied commercially.

C. IRWIN.

**"Phenolates."** K. LUTS (Tehnika Ajakiri, 1930, 9, 170–173; Chem. Zentr., 1931, i, 1210–1211).—A method for determining neutral oil in "phenolates" depends on extraction with xylene in presence of NaOH in a two-bulbed flask with a graduated neck. Material from shale oil has the formula  $\text{C}_{21}\text{H}_{28}\text{O}_{1.7}$ ; owing to hydrolysis it appears to continue to yield neutral oil to  $\text{Et}_2\text{O}$  or  $\text{CS}_2$ . Continued exposure to air affords a solid product.

A. A. ELDRIDGE.

**MeOH antifreeze.**—See I. **Acenaphthene for dyes.**—See IV. **Ursolic acid etc. [for lacquers].**—See XIII. **Lactic acid.**—See XVIII. **Ethylene oxide as fumigant. Detection etc. of butyric acid.**—See XIX. **Trinitrotoluene. Amines.**—See XXII.

#### PATENTS.

**Conversion of acetylene into higher-boiling hydrocarbon products.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 344,470, 28.10.29. Holl., 13.11.28).—In the condensation of  $\text{C}_2\text{H}_2$  under pressure explosion is avoided by effecting reaction in presence of a liquid medium, *e.g.*, decalin, paraffin oil, transformer oil, etc. The  $\text{C}_2\text{H}_2$  may be generated from  $\text{CaC}_2$  and  $\text{H}_2\text{O}$  in the autoclave. Olefine gases may be added.

C. HOLLINS.

**Production of ketones [from acetylene and alcohols, aldehydes, acids, etc.].** HOLZVERKOHLS-UNGS-IND. A.-G. (B.P. 344,449, 3.12.29. Ger., 25.1.29).—The exothermic conversion of  $\text{C}_2\text{H}_2$  into  $\text{COMe}_2$  is combined with a non-exothermic ketonisation, *e.g.*, of EtOH, AcOEt, MeCHO, or AcOH. The catalysts of B.P. 302,759 (B., 1929, 163) are used. A mixture of

EtOH vapour,  $C_2H_2$ , and steam, passed over rusty Fe sponge containing 3% of Mn oxide at  $470^\circ$  gives  $COMe_2$  in 89% yield. C. HOLLINS.

**Manufacture of ketones [acetone from aliphatic primary alcohols].** H. DREYFUS (B.P. 344,479, 28.11.29).—An alcohol ( $C_2$  or over) and steam are passed at  $475$ – $485^\circ$  over a Zn catalyst, *e.g.*, the acetate, or ZnO and Ca silicate; preferably O is added to the vapours. C. HOLLINS.

**Transformation of organic acids into ketones. [Removal of acids from crude oils.]** J. W. BECKMAN (U.S.P. 1,753,642, 8.4.30. Appl., 23.2.28).—Hydrocarbon oil containing carboxylic acids is passed as vapour through CaO at  $200^\circ$ , the Ca salts being subsequently heated at  $400^\circ$  to give ketones. The vapours pass, *e.g.*, through two lime-packed chambers at  $200^\circ$  and  $400^\circ$  respectively; the temp. and direction of the vapours are then reversed; naphthenic ketones distil alternately from the two chambers. C. HOLLINS.

**Polymerisation of ketone-formaldehyde condensation products.** C. E. BURKE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,755,099, 15.4.30. Appl., 6.6.28).—The product from  $COMe_2$  and  $CH_2O$  is polymerised in presence of carbamide or an amine at about  $100^\circ$ . If nitrocellulose or cellulose acetate etc. is first dissolved in the above product polymerisation gives an infusible resin. C. HOLLINS.

**Chlorination of unsaturated hydrocarbons.** C. J. STROSACKER, Assr. to DOW CHEM. Co. (U.S.P. 1,754,656, 15.4.30. Appl., 16.5.28).— $C_2H_4$  or other olefine (*e.g.*, cracked gases) is led into, or passed through, a spray of  $SbCl_5$  dissolved in ethylene dichloride (or reaction product from a previous batch). Only dichloride is formed, no substitution taking place. C. HOLLINS.

**Production of 1 : 1 : 2 [ $\alpha\alpha\beta$ ]-trichloroethane.** I. G. FARBENIND. A.-G. (B.P. 344,592, 10.1.30. Ger., 31.1.29).—Trichloroethylene is made in one operation from  $C_2H_2$ , Cl, and HCl, explosion being avoided by effecting reaction in a constricted space or in presence of excess of  $C_2H_2$ , HCl, or inert gas. Contact materials, such as coke, quartz, pumice, may be employed, impregnated, if desired, with metal salts. C. HOLLINS.

**Production of formaldehyde from methane.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 343,461, 15.11.29. Ger., 15.11.28).— $CH_4$  and air in equal vols., or coke-oven gas, are passed through an electric field in which luminous discharges of high frequency are passing, *e.g.*, between electrodes 15–25 cm. apart at 80,000 volts and 400,000 frequency. Preferably a catalyst (CuO) is used. The yield is 350–480 g. per cu. m.  $CH_4$ . C. HOLLINS.

**[Manufacture of] synthetic methanol [methyl alcohol].** L. A. STENGEL, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,754,371, 15.4.30. Appl., 17.11.26).—The mixture of carbon oxides and H is scrubbed with MeOH before reaction in order to remove noxious impurities. C. HOLLINS.

**Catalytic production of acetaldehyde [from alcohol].** G. B. ELLIS. From C. F. BOEHRINGER & SÖHNE, G.M.B.H. (B.P. 344,554, 16.12.29).—EtOH vapour is passed at  $250$ – $300^\circ$  over reduced Cu containing about 0.2% of  $CeO_2$ . C. HOLLINS.

**Production of acetaldehyde and acetic acid from acetylene.** C. C. TANNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 344,638, 20.2.30).—A Bi compound, particularly a phosphate or molybdate, is used as catalyst. Preferably 2–10 vols. of steam are mixed with the  $C_2H_2$ . C. HOLLINS.

**Treatment [stabilisation] of aldehyde-amines.** NAUGATUCK CHEM. Co., Asses. of S. I. STRICKHOUSER (B.P. 344,690, 1.4.30. U.S., 17.4.29).—An aromatic di- or poly-hydroxy-compound (quinol) is added to aldehyde-amine condensation products (accelerators, *e.g.*, heptaldehyde-aniline) to prevent deterioration, especially in a master rubber batch. C. HOLLINS.

**Manufacture of acetic anhydride.** E. BERT (B.P. 344,482, 28.11.29. Austr., 30.11.28).—AcOH vapour is passed at  $700^\circ$  over a Cu or Ag catalyst on tungstic acid, pumice, or asbestos, preferably with addition of a little air (or the catalyst is treated with air at intervals). C. HOLLINS.

**Preparation of anhydrides and chlorides of aliphatic acids from aliphatic salts.** SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (B.P. 344,116, 27.12.29. Fr., 8.1.29).—In the manufacture of aliphatic acid anhydrides or chlorides from aliphatic salts and  $SO_2Cl_2$  or  $SO_2$  and Cl, with or without catalyst and/or solvent, alkaline-earth salts may be used provided that they are diluted with sand or other inert powder. Preferably an acid-absorber, *e.g.*, CaO, is added. C. HOLLINS.

**Non-corrosive alcoholic solution.** W. S. CALCOTT and H. W. WALKER, Asses. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,754,457, 15.4.30. Appl., 20.9.28).—Addition of an aromatic diamine (*m*-tolylenediamine), a higher fatty acid (stearic), and a diarylamine (phenyl- $\alpha$ -naphthylamine) to glycol or other polyhydric alcohol prevents attack of Fe by the diluted mixture, which is thus suitable for use in coolers. C. HOLLINS.

**Prevention of polymerisation of vinyl compounds and of reaction of the latter with aldehydes.** K. G. BLAICKIE, Assr. to CANADIAN ELECTRIC PRODUCTS Co., LTD. (U.S.P. 1,768,434, 24.6.30. Appl., 15.8.27).—Addition of 0.1% of S to vinyl compounds prevents polymerisation during distillation or storage and also prevents reaction with aldehydes. C. HOLLINS.

**[Catalysts for] synthesis of organic compounds containing oxygen [from carbon monoxide and steam].** SOC. CHIM. DE LA GRANDE-PAROISSE AZOTE & PROD. CHIM. (B.P. 343,807, 10.4.30. Fr., 7.2.30).—Artificial zeolites, prepared by fusion or by precipitation from alkaline solution and reduced if desired, are used as catalysts. Examples are:  $KNaMnO_4$  with borax and an AcOH solution of  $Ce_2(CO_3)_3$ ,  $Al(OAc)_3$ , and malachite, precipitated at  $p_H$  7, and reduced in CO at  $200$ – $350^\circ$ ; borax and  $Na_2Al_2O_4$  with a formic acid solution of  $Li_2CO_3$ ,  $K_2CO_3$ , thorium formate and  $Al(OAc)_3$ , precipitated at  $p_H$  7.5, and dried. [Stat. ref.] C. HOLLINS.

**Manufacture of pentaerythritol ethers.** C. E. BURKE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,774,500, 2.9.30. Appl., 27.1.28).—Pentaerythritol is etherified, *e.g.*, with EtCl and NaOH in petrol, to give mono-, di-, and tri-ethyl ethers. Distillation

gives fractions, b.p. 150—165°/44 mm. (80% tri-ether), 165—200°/50 mm. (80% di-ether), and 200—260°/50 mm. (mono-ether), which are plasticisers for cellulose esters and ethers. C. HOLLINS.

**Manufacture of hexamethylenetetramine.** G. E. LANDT and W. H. ADAMS, JUN., Assrs. to CONTINENTAL DIAMOND FIBRE Co. (U.S.P. 1,774,929, 2.9.30. Appl., 4.1.27).—CH<sub>2</sub>O and NH<sub>3</sub> are caused to react in 95% EtOH. C. HOLLINS.

**Preparation of derivatives of 3:4-dihydroisoquinoline.** BOOTS PURE DRUG Co., LTD., R. CHILD, and F. L. PYMAN (B.P. 344,166, 31.1.30).—The Pictet-Decker method is applied to the synthesis of 1-cyanoalkyl or 1-chloroalkyl derivatives of 6-methoxy-, 6:7-dimethoxy-, and 6:7-methylenedioxy-3:4-dihydroisoquinolines. 1-Chloromethyl-6:7-dimethoxy-3:4-dihydroisoquinoline (hydrochloride, m.p. 217°) is prepared from *N*-chloroacetyl-β-veratrylethylamine and POCl<sub>3</sub> in toluene; the 1-ω-chlorobutyl (picrate, m.p. 156—157°), 1-bromomethyl (picrate, m.p. 185°), and 1-cyanomethyl (m.p. 171—172°) compounds, and 1-chloromethyl-6-methoxy- (m.p. 169—170°) and -6:7-methylenedioxy- (picrate, m.p. 179—180°) -3:4-dihydroisoquinolines are also described. C. HOLLINS.

**Manufacture of wetting, cleansing, and dispersing agents [hard-water soaps].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 343,524, 343,872, 343,899, and 343,900, 7.8.29).—(A) A saturated aliphatic or alicyclic acid of C<sub>6</sub>—C<sub>18</sub> or an unsaturated aliphatic acid above C<sub>6</sub> is condensed with a primary arylamine (which may be sulphonated), or (c) any higher aliphatic or alicyclic acid is condensed with NH<sub>3</sub> or a non-hydroxylated alkyl-, cycloalkyl- or aralkyl-amine, in all cases in absence of ZnCl<sub>2</sub> or other polymerising condensing agent. The acid amides so obtained are sulphated below 40° or sulphonated above 40° (if sufficient sulphuric or sulphonic groups are not already present). (B) Non-carboxylated olefines above C<sub>8</sub>, or corresponding monohydric, non-primary alcohols, are sulphated below 40° until soluble, but still hydrolysable by hot 10% HCl. (D) Hydroxyalkyl ethers of alcohols or phenols above C<sub>8</sub>, free from CO<sub>2</sub>H and NH<sub>2</sub> groups, are sulphated. (A—D) The products are wetting agents and soaps not pptd. by acids, alkalis, or hard water. They may be used alone or with soaps, high-mol. sulphonic acids, glue, vegetable gums, alkali carbonates, sodium silicate, NaHSO<sub>4</sub>, organic solvents, bleaching agents, etc. They disperse insoluble soaps (curds), dyes, fats, waxes. Examples are: (A) oleic anilide; anilides of acids C<sub>12</sub>—C<sub>15</sub> from oxidation of paraffin wax; (B) penta- triacontanol from reduction of stearone; cetene from distilled spermaceti wax; olefines from dehydration of liquid oxidation products of naphthenes; (C) oleic amide or ethylamide; fatty acid chlorides from coconut oil, amidated with sodium taurine (product needs no sulphonation); ricinoleic amide; hydroxystearic amide; methyl ricinoleate, sulphated and amidated; (D) *o*-tolyl β-hydroxyethyl ether, sulphated. (B) Examples of applications include degreasing of wool, emulsification of paraffin for oiling fibres before spinning, production of level dyeings (e.g., anthraquinone-blue SR and woolfast-yellow 3G), softening of textiles, dewaxing of cotton,

and aniline-black dyeing. (Cf. B.P. 341,053; B., 1931, 386.) C. HOLLINS.

**Protection of materials from attack by micro-organisms.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,339, 24.10.29).—A mixture of a poly-halogenated phenol (or alkali salt or ester) and a benzoic acid (or alkali salt or alkyl ester) is more effective against fungi and bacteria than either constituent alone. Examples are: trichlorophenyl acetate or tetrachlorophenol with Me *p*-hydroxybenzoate; trichlorophenyl acetate with BzONa; trichlorocresol with BzOH. Materials such as gelatin, glue, fabrics, tanning extracts, etc. may be protected by their application. C. HOLLINS.

**Manufacture of 4-hydroxy-3-ethoxybenzaldehyde.** I. G. FARBENIND. A.-G., and W. W. GROVES (B.P. 344,675, 15.3.30. Addn. to B.P. 219,676; B., 1925, 26).—The product from pyrocatechol monoethyl ether and chloral is boiled with CuO or CuCO<sub>3</sub> in water. C. HOLLINS.

**Manufacture of aromatic *N*-dialkylaminoalkyl-aminoaldehydes and derivatives thereof.** I. G. FARBENIND. A.-G. (B.P. 343,570, 21.11.29. Addn. to B.P. 288,972; B., 1929, 709).—The aminoaldehydes of the prior patent are obtained by action of *N*-dialkyl-aminoalkylarylamines, having a free *p*-position, on *N*-disubstituted formamides in presence of POCl<sub>3</sub> (etc.); or from *N*-formyldialkylaminoalkylarylamines and POCl<sub>3</sub> (etc.). *N*-Methyl-*N*-β-diethylaminoethyl-*p*-aminobenzaldehyde, b.p. 166—168°/2 mm., is produced from *N*-methyl-*N*-β-diethylaminoethylaniline and form-methylanilide; *N*-β-diethylaminoethyl-*p*-aminobenzaldehyde, b.p. 182—183°/4 mm., from formyl-β-diethylaminoethylaniline, b.p. 126—127°/1.5 mm.; *N*-ethyl-*N*-β-diethylaminoethyl-*p*-aminobenzaldehyde, b.p. 168—170°/1.5 mm. from *N*-ethyl-*N*-β-diethylaminoethylaniline, b.p. 118—120°/3 mm., and form-methylanilide; *N*-methyl-*N*-β-diethylaminoethyl-6-amino-*m*-tolualdehyde, b.p. 145—147°/1.5 mm., from *N*-methyl-*N*-β-diethylaminoethyl-*o*-toluidine, b.p. 110—112°/5 mm.; *NN*-bis-β-diethylaminoethyl-*p*-aminobenzaldehyde from *NN*-bis-β-diethylaminoethylaniline, b.p. 163—165°/4 mm.; and *N*-methyl-*N*-β-1-piperidinoethyl-*p*-aminobenzaldehyde, b.p. 202—204°/3 mm., from *N*-methyl-*N*-β-1-piperidinoethylaniline, b.p. 140°/3 mm. C. HOLLINS.

**Manufacture of arylamines.** W. H. WILLIAMS, Assr. to Dow CHEM. Co. (U.S.P. 1,775,360, 9.9.30. Appl., 27.4.27).—The reaction between chlorobenzene (etc.) and NH<sub>3</sub> in presence of Cu<sub>2</sub>O at 150—250°/3—10 atm. is favourably influenced by adding the diphenylamine by-products from a previous batch. C. HOLLINS.

**Replacement of halogen groups in organic compounds by amino-groups.** K. H. T. PFISTER, Assr. to ROHM & HAAS Co. (U.S.P. 1,752,998, 1.4.30. Appl., 23.12.25).—An aromatic halogen compound carrying *o*- and/or *p*-NO<sub>2</sub> groups is fused with carbamide at about 240° and the product boiled with alkali; 1-chloro-2:4-dinitrobenzene yields 2:4-dinitroaniline. C. HOLLINS.

**Manufacture of alkylated 3:7-diaminoacridinium compounds.** I. G. FARBENIND. A.-G. (B.P. 344,627, 12.2.30. Ger., 12.2.29).—3:6-Diaminoacridine is alkylated at 90—120°, especially with alkyl sulphate or

arylsulphonate, in absence of solvent, and, if desired, in presence of an inorganic salt. The products are diamino-acridinium salts alkylated on the ring N and also in one  $\text{NH}_2$  group. The methylated compound dyes cotton orange.

C. HOLLINS.

**Manufacture of aromatic [2-thiolbenz]thiazole compounds.** H. A. MERKLE, ASSR. TO RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,753,898, 8.4.30. Appl. 14.7.27).—The production of 2-thiolbenzthiazole from phenylthiocarbimide and S at 220–240° under pressure is catalysed by 3.5% of water. The yield is increased to 88–92%.

C. HOLLINS.

#### Manufacture of intermediates and [polymethine] dyes from heterocyclic nitrogen compounds.

IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and E. H. RODD (B.P. 344,409, 4.11.29).—Diarylformamidines (1 mol.) react in presence of acid (1 mol. or more, combined with a reactant or free) with cyclic  $\text{NH}_4$  salts or the corresponding  $\psi$ -bases (1 mol.) derived from heterocyclic N compounds having a reactive Me or external  $\text{CH}_2$  group, preferably in a solvent ( $\text{Ac}_2\text{O}$ ), to give intermediates of the type,  $\text{Q}\cdot\text{CH}:\text{CH}\cdot\text{NHR}$ , where Q represents the heterocyclic residue. When  $\text{Ac}_2\text{O}$  is used the product is the Ac derivative. With 2 mols. of the heterocyclic compound a polymethine dye is formed. Examples are: diphenylformamidine with 1 mol. of 2:3:3-trimethylindolenine methiodide, giving on hydrolysis with HCl 2- $\omega$ -anilino-1-vinyl-3:3-dimethylindolenine methiodide, m.p. 243–244° [Ac derivative, m.p. 260° (decomp.)]; with 1 mol. of 2-methylbenzoxazole ethiodide for the 2- $\omega$ -acetanilidovinyl compound, m.p. 227° (decomp.); with 1 mol. of 2-methylbenzthiazole ethiodide for 2- $\omega$ -acetanilidovinyl compound, m.p. 229°; with 1 mol. of quinaldine ethiodide; with 2 mols. of 2-methylene-1:3:3-trimethylindoline for hexamethylindocarbocyanine; with 2 mols. of 2-methylbenzthiazole ethiodide for 1:1-diethylcarbothiocyanine iodide; with 2 mols. of quinaldine ethiodide for pinacyanol.

C. HOLLINS.

**Intermediates of benzanthronepyrazolanthrone series.**—See IV. Sodium glutamate. Zeolites.—See VII.

#### IV.—DYESTUFFS.

**Application of acenaphthene to the production of dyes of the anthanthrone series.** A. CORBELLINI and A. PASSAGGIO (Giorn. Chim. Ind. Appl., 1931, 13, 109–113).—The application of acenaphthene to the synthesis of anthanthrone and its derivatives through the intermediate compounds naphthalic anhydride, naphthalimide, naphthostyryl, 8-amino-1-naphthoic acid, and 1:1'-dinaphthyl-8:8'-dicarboxylic acid is discussed. The properties of anthanthrone and of its dibromo-, diamino-, and diacetyldiamino-derivatives as vat dyes and for printing are considered.

T. H. POPE.

#### PATENTS.

**Production of anthraquinone derivatives [wool dyes].** R. J. LOVELUCK, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 343,449, 9.8.29).—1-Amino (or methylamino)-4-arylaminoanthraquinone-2-sulphonic acids are sulphonated to give wool dyes, which may be isolated or purified by dilution of the  $\text{H}_2\text{SO}_4$

solution. The product from 1-amino-4-anilinoanthraquinone-2-sulphonic acid dyes wool blue. [Stat. ref.]

C. HOLLINS.

**Manufacture of anthraquinone derivatives [dyes for acetate silk or wool, pigments for lacquers].** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 344,117, 27.12.29).—1-Nitroanthraquinones carrying in position-5 or -8 an  $\text{ArNH}_2$  or hydrogenated  $\text{ArNH}_2$  group, and in the other  $\alpha$ -positions  $\text{NH}_2$  or monoalkyl- $\text{NH}_2$  groups, are reduced, and, if desired, the products are sulphonated, alkylated, or acylated. 4:8-Dinitro-1:5-diaminoanthraquinone gives with aniline at 150–160° 8-nitro-1:5-diamino-4-anilinoanthraquinone which on reduction yields 1:5:8-triamino-4-anilinoanthraquinone (blue on acetate silk); the technical mixture of dinitrodiaminoanthraquinones, freed from  $\beta$ -derivatives, gives the same final product. The corresponding 8-nitro-1:5-diamino-4-*p*-anisidino- (m.p. 273–275°), -4-*o*-anisidino-, -4-*p*-toluidino- (m.p. 288–290°), 4-*p*-chloroanilino-, 4-(4'-methoxy-*m*-toluidino)-, and 4-*cyclo*-hexylamino-anthraquinone are similar reduced. 1:5:8-Triamino-4-*p*-anisidino- and -4-*p*-toluidino-anthraquinones, m.p. 220° and 210°, respectively, give green-blue shades on acetate silk. 1:5:8-Triamino-4-anilinoanthraquinone is methylated or hydroxyethylated for green-blue acetate-silk dyes; the *p*-toluidino-compound is sulphonated for a green wool dye. Acylated 1:5:8-triamino-4-anilinoanthraquinone dyes acetate silk blue. 1:5:8-Triamino-4-*p*-anisidinoanthraquinone gives a transparent green-blue nitrocellulose lacquer, fast to light.

C. HOLLINS.

**Manufacture and use of [hydroxylated] compounds of the anthraquinone series [dyes for acetate silk].** BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, E. W. KIRK (B.P. 343,444, 15.11.29).—Part or all of the halogen in halogenated anthrarufins is replaced by OH by heating with  $\text{H}_2\text{SO}_4$ , with or without  $\text{H}_3\text{BO}_3$ . The halogenated products may be aminated or alkylaminated, preferably as leuco-compounds, and OH groups may be alkylated. 6-Chloro-1:4:5-trihydroxyanthraquinone, from 4:6-dichloroanthrarufin, is converted into the 6-methylamino-compound (green-blue on acetate silk), and into a methyl ether (yellow). [Stat. ref.]

C. HOLLINS.

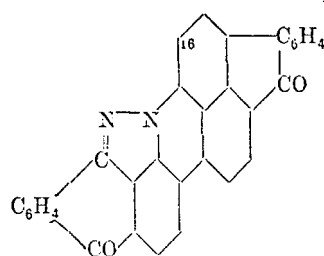
**Manufacture of vat dyes of the N-dihydro-1:2:2':1'-anthraquinoneazine [indanthrone] series which are fast to chlorine.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 343,994, 24.10.29 and 17.4.30).—The condensation products from impure indanthrone and  $\text{CH}_2\text{O}$  are dissolved in  $\text{H}_2\text{SO}_4$  and precipitated by dilution not below 70%; or the condensation products are extracted with  $\text{H}_2\text{SO}_4$  (*d* 1.61 or over). Pure indanthrone gives equally fast products when condensed with  $\text{CH}_2\text{O}$ . The products may be chlorinated if desired.

C. HOLLINS.

**Production of [alkali-soluble] dyes [of the dibenzanthrone series].** C. SHAW, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 344,336, 23.8.29).—A dibenzanthrone is heated at 250–300° with P sulphides or red P and S to give products which dye cotton and wool from an alkaline bath. Examples of starting materials are dibenzanthrone (blue-black at 300°, blue-red at 250°),

and 2:2'-dimethoxydibenzanthrone (blue-grey at 250°, becoming blue-violet by oxidation on the fibre with  $\text{NaBO}_3$ ). C. HOLLINS.

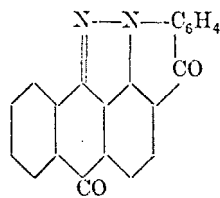
**Manufacture of vat dyes and intermediate products of the benzanthronepyrazolanthrone series.** I. G. FARBENIND. A.-G. (B.P. 344,057 and Addn. B.P. 344,147, [A] 2.12.29, Ger., 1.12.28. [B] 18.1.30, Ger., 27.2.29).—Substituted  $\text{NH}_2$  groups are introduced into benzanthronepyrazolanthrone (annexed



formula) by action of suitable (A) halogeno- or  $\text{NO}_2$  compounds etc. on its  $\text{NH}_2$  derivative, (B)  $\text{NH}_2$  compounds on its halogeno- or  $\text{NO}_2$  derivatives. 16-Aminobenzanthronepyrazolanthrone (by nitration and reduction) is condensed

with  $\text{Ac}_2\text{O}$ ,  $\text{BzCl}$ , cyanuric chloride,  $\text{Me}_2\text{SO}_4$ ,  $\text{CH}_3\text{PhCl}$ , 1-chloroanthraquinone, 1:5-dichloroanthraquinone, 3-bromo-, dichloro-, and hexachloro-benzanthronepyrazolanthrones, 1-amino-2-aldehydoanthraquinone (azo-methine unstable to vatting), and 1:4-naphthaquinone. 3-Bromobenzanthronepyrazolanthrone (from pyrazolanthrone and dibromobenzanthrone of m.p. 256°) and the dichloro-compound are condensed with  $\alpha$ - or  $\beta$ -aminoanthraquinone, and  $\text{NHMe}_2$ . The products are green-blue to grey or olive vat dyes. C. HOLLINS.

**Manufacture of vat dyes [pyrazolanthrone-acridones].** I. G. FARBENIND. A.-G. (B.P. 344,558, 17.12.29. Ger., 17.12.28).—Pyrazolanthrone or 1:5-dipyrazolanthrone is condensed with an *o*-halogeno-



aldehyde and the product is fused with alkali to give an acridone. Pyrazolanthrone with *o*-chlorobenzaldehyde thus yields a yellow-orange vat dye (annexed formula), with 2:5-dichloroterephthalaldehyde (0.5 mol.) a yellow-brown; 1:5-dipyrazolanthrone with *o*-chlorobenzaldehyde gives an olive-green.

C. HOLLINS.

**Manufacture of [azo] dyes and their application [to dyeing of acetate silk etc.].** BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and D. H. MOSBY (B.P. 343,502, 16.9.29).—Diazotised amines of the  $\text{C}_6\text{H}_6$  or  $\text{C}_{10}\text{H}_8$  series are coupled with non-sulphonated hydroxy-alkylated  $\alpha$ -naphthylamines; e.g.,  $\gamma$ -chloro- $\beta$ -hydroxy-*n*-propyl-1-naphthylamine with diazotised *p*-nitroaniline (red-violet on acetate silk), 5-nitro-*o*-anisidine (violet), 5-nitro-*o*-phenetidine (violet), 4-amino-4'-dimethylamino-azobenzene (blue-red), benzencazo- $\alpha$ -naphthylamine (violet-grey). C. HOLLINS.

**Manufacture of [acid and mordant] azo dyes from pyrazolonecarboxylic amides.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 344,250, 24.3.30).—5-Pyrazolone-3-carboxylic amides are used as coupling components. Examples are: 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylamide with diazotised aniline (golden-yellow) or 4-nitro-*o*-aminophenol-6-sulphonic acid (red-brown; after-chromed, red-orange; pre-

chromed, orange-brown); 1-phenyl-5-pyrazolone-3-carboxylic anilide with diazotised 1:2:4-amino-naphtholsulphonic acid (brown-red; after-chromed, violet). C. HOLLINS.

**Acridinium compounds. Polymethine dyes.**—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Optical activity of natural silk.** D. ONGARO (Giorn. Chim. Ind. Appl., 1931, 13, 114—118).—The optical activity is found to be due, not to molecular or crystalline rotatory power, but to a phenomenon of birefractance, the accidental nature of which is revealed by the variability in the orientation of the extinction of different fibres. The fact that the optical axis of the fibre is in the direction of the latter is regarded as evidence that the silk is subjected to pressure during its emission. The appearance of Chardonnet nitrocellulose silk under the polarising microscope differs greatly from that of viscose silk. T. II. POPE.

**Susceptibility of animal fibres to damage by the larvae of two species of clothes moth, *Tineola biselliella*, Hummel, and *Tinea pellionella*, L. R. BURGESS and E. J. POOLE (J. Text. Inst., 1931, 22, T 141—157).**—Vegetable fibres and natural silk are not attacked by the above species, whilst animal fibres such as alpaca, camel hair, cashmere, and the natural fleece of the sheep are readily attacked. The susceptibility of wool is further increased by scouring. Partly processed sheep wools containing vegetable oil are less readily damaged, but are not immune. Fatty acids in concentrations up to 5.2% on the wt. of wool do not afford complete protection and are inferior in this respect to certain mineral oils. "Larvex" and "Eulans" are satisfactory protective agents, whilst "Globol" (*p*-dichlorobenzene) is superior to "Flit" and "Deleoil" when used for the protection of infected wool in a closed atmosphere. B. P. RIDGE.

**Apparatus for measurement of flow and relaxation of textile filaments.** H. D. W. SMITH (J. Text. Inst., 1931, 22, T 158—169).—An improved Polanyi tester is described with which measurements of the rate of flow of single rayon filaments may be made under constant load at various temp. and atm. humidities or in liquid media. For measurement of relaxation, an apparatus has been devised which consists of a balance from one end of the beam of which the filament is suspended, whilst the other end is loaded by means of a float hanging vertically in a reservoir containing  $\text{PrOH}$ . The position of the beam is read by means of a mirror and a distant scale and telescope. Methods of measurement of changes in specimen length and of load changes with the arrangement of the float are discussed.

B. P. RIDGE.

**Flow and relaxation of rayon filaments.** H. D. W. SMITH and R. EISENSCHITZ (J. Text. Inst., 1931, 22, T 170—196; cf. preceding abstract).—The apparatus described above has been used to determine the flow and relaxation curves for viscose rayon filaments; the rate of flow of the filaments under constant load and its relation to the temp. and to the degree of absorption of water have been measured. The relative rate of relaxation

is independent of the load and of the cross-section of the filament. This agrees with Boltzmann's theory of "after-effect." The flow curves, however, deviate from this theory. The rate of flow increases rapidly with rise in temp. and is 30–50 times as great for wet as for dry material. The distribution of the plastic inhomogeneity in the rayon structure (to which the after-effects are ascribed) is apparently unaffected by changes in temp. and in the degree of water absorption.

B. P. RIDGE.

**Determination of the uniformity in thickness of fibres.** E. VIVIANI (Giorn. Chim. Ind. Appl., 1931, 13, 118–120).—Another method is described (cf. B., 1931, 291), in which the thickness of the fibre is measured by determining the variation in pressure of gas passing through a capillary tube through which also the fibre is drawn.

T. H. POPE.

**Properties of cotton fibres.** G. KITA, Y. SHINODA, and S. OHARA (J. Cellulose Inst., Tokyo, 1931, 7, 119–121).—The I values of different varieties of both raw and extracted cottons run parallel to the Cu numbers, hence the former are to be interpreted in the same way as the latter. Differences between the I values of the raw and extracted materials depend on the wax content.

B. P. RIDGE.

**Study of comparative results for lea, single-thread, and ballistic tests on yarns from standard Indian cottons.** A. J. TURNER and V. VENKATARAMAN (J. Text. Inst., 1931, 22, T 197–223).—All tests agree fairly well together, but whereas the differences between the lea strength and single-thread strength results are negligible, differences between these results and those of ballistic strength are more serious. The ballistic test is considered to be a good test of yarn and worthy of a much more extended examination. B. P. RIDGE.

**Determination of hemicelluloses.** R. C. MALHOTRA (Ind. Eng. Chem. [Anal.], 1931, 3, 161–163).—Suggestions are made for a standard technique for the determination of hemicellulose.

T. McLACHLAN.

**Cooking of sulphate-cellulose.** D. JOHANSSON [with G. HELLBORG, W. GILLESPIE, and F. SVENSSON] (Svensk Pappers-Tidn., 1930, 33, 916–928; Chem. Zentr., 1931, i, 1198).—The strength of milled cellulose is maximal at a Cl value (Roc) of 7; 25% Na<sub>2</sub>S solutions have the greatest solvent action on lignin. Soda-cellulose of the same strength as sulphate-cellulose was not obtained by careful cooking or addition of black liquor.

A. A. ELDRIDGE.

**Preparation of cellulose acetate threads in the laboratory.** G. VON FRANK, H. E. KRÜGER, and W. W. WOLFF (Z. angew. Chem., 1931, 44, 342–348).—Suitable apparatus and technique for the dry- and wet-spinning of cellulose acetate solutions on a laboratory scale are described.

F. R. ENNOS.

**Reaction of sulphites with sugar and lignin in the sulphite-pulping process.** E. HÄGGLUND (Papier-Fabr., 1931, 29, 161–164).—Lignosulphonic acids are formed first, which remain in combination with the fibre. Dissolution of these acids depends on the [H<sup>+</sup>] and temp. of the cooking liquor. The aldehydic nature of residual lignin in pulps is demonstrated by means of Schiff's reagent, coloration increasing with lignin con-

tent.  $\alpha$ -Lignosulphonic acid (aldehydic) is reduced to the  $\beta$ -compound (alcoholic). The former acid is combined with aromatic amines, and can be precipitated from digestion liquors. The ultimate product of sugars is gluconic acid.

T. T. POTTS.

**Process water in the pulp and paper industry.** H. HAUPT (Papier-Fabr., 1931, 29, 273–279).—A detailed account of the principal impurities in process water, their effects on manufacture, and methods of purification. Representative analyses of German river waters, with the results of various methods of purification, are given.

T. T. POTTS.

**Rapid measurement of resistance of paper to deterioration by light.** E. H. RIESENFELD and T. HAMBURGER (Papier-Fabr., 1931, 29, 164–166).—Paper is exposed to the rays from a mercury-vapour lamp, the change in fluorescence being taken to indicate the commencement of action. Tests show that many papers behave similarly whether covered with window glass or not. Orange-coloured glass is held to be the only adequate protection. Tests may be carried out in 3 min.

T. T. POTTS.

**Cellulose acetate in U.S.A.**—See III. **Bleaching of wood pulp.**—See VI. **Laminated glass.**—See VIII.

#### PATENTS.

**Manufacture of cellulose.** J. J. DE LE ROZA, SEN. (B.P. 347,297, 30.12.29).—Vegetable fibre is treated with an aq. solution of SO<sub>2</sub> either in two stages, the first being with a cold dil. solution and the second with a hot conc. solution under pressure, or in one stage with the conc. solution. The material is afterwards washed and repeatedly treated with alkali under conditions of increasing temp. and pressure.

F. R. ENNOS.

**Manufacture of high-grade  $\alpha$ -cellulose.** L. I. L. DURCHMAN (U.S.P. 1,781,449, 11.11.30. Appl., 8.5.29).—Chemical pulp, particularly sulphate pulp, is digested with a liquor containing 2–5% of NaOH and 1–3% of Na<sub>2</sub>SO<sub>3</sub> (both on the wt. of pulp) for 4–6 hr. at 20–60 lb./in.<sup>2</sup> and, after washing, is bleached, preferably by a two-stage process, with NaOCl. The bleached pulp is washed, centrifuged to remove as much H<sub>2</sub>O as possible, and treated at room temp. for 3–5 hr. with "white liquor" containing 100–140 g. of NaOH per litre. After further washing, the pulp is soured with 6–8% of (CO<sub>2</sub>H)<sub>2</sub> to remove Fe compounds and is finally washed and dried. 80–90% of the white liquor is recovered and used in the manufacture of sulphate pulp.

D. J. NORMAN.

**Production of high-grade cellulose from soiled and disintegrated vegetable fibres, particularly from cotton wastes such as linters, hull fibre, and spinning-mill wastes.** H. KAMMERL (B.P. 347,444, 1.4.30. Ger., 12.11.29).—The dried material is beaten, mixed with H<sub>2</sub>O, and run through a sieveless centrifuge, whereby the bulk of the impurities are removed; the pectins, waxes, and fats are then dissolved out by a hot solution of 1% K soap plus 1% NaOH (bucking solution).

F. R. ENNOS.

**Production of solutions for spinning artificial threads, bands, films, etc.** I. G. FARBERIND. A.-G. (B.P. 347,428, 27.3.30. Ger., 27.3.29).—Spinning



solutions of high uniformity are produced by combining the filtrates from a number of batches of crude spinning solution, removing the dissolved gas in a buffer vessel, and subjecting the mixture to mechanical agitation.

F. R. ENNOS.

**Manufacture of artificial fibres from viscose.**

W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 345,938, 29.11.29).—Threads of increased tensile strength are produced from viscose containing  $>5\%$  of alkali by spinning into an acid coagulating bath ( $30\text{--}55\%$  of free  $\text{H}_2\text{SO}_4$ ) to which is added more than  $20\%$  of  $\text{NH}_4$  or Na bisulphate.

F. R. ENNOS.

**Preparation of artificial textile products [threads] with reduced lustre from viscose.**

T. KOCH (U.S.P. 1,781,018, 11.11.30. Appl., 3.10.27. Holl., 11.2.27).—Dull-lustre viscose silk is produced by spinning in the usual manner viscose solutions which have been emulsified with small quantities (about  $1\%$ ) of  $\text{PhCl}$  or a mixture of mono- and di-chlorobenzenes. A. J. HALL.

**Wet twisting of threads or yarns.** A. KARSCH (B.P. 346,150, 1.4.30).—During its passage to the twisting device the thread passes through a counter-current of water.

F. R. ENNOS.

**Manufacture of artificial silk.**

I. G. FARBENIND. A.-G. (B.P. 317,417, 21.3.30. Ger., 21.3.29).—The spun cakes threaded on perforated tubular supports are subjected to an alternate or simultaneous spraying and dipping operation with  $\text{H}_2\text{O}$  or after-treating liquids, the dipping troughs being provided with devices for causing rotation of the cakes.

F. R. ENNOS.

**Direct manufacture and working up of artificial filaments.** B. BORZYKOWSKI (B.P. 347,241 and Addn. B.P. 347,259, [A] 20.12.29, [B] 23.1.30. Ger., [A] 22.12.28, [B] 24.1.29).—(A) Spun and twisted filaments in cake form are washed, after-treated, softened or sized after first inserting in the cake a perforated and preferably flexible sleeve of the same length as the cake, and are afterwards dried, as described in (B), while in an upright position on a support so that the lower edge of the cake rests on a plane surface covered with soft material; the filaments are then worked on a knitting or other machine directly into fabrics without rewinding, by insertion of a longer sleeve which projects beyond the ends of the cake. Or untwisted filaments wound on a perforated spinning bobbin larger than the standard size are subjected to further treatment, passed during unwinding through a solution suitable for holding the threads together, and directly worked into fabrics.

F. R. ENNOS.

**Precipitation of cellulose esters.**

U.S. INDUSTRIAL ALCOHOL Co. (B.P. 346,824, 16.1.30. U.S., 10.5.29).—Cellulose material, which has been acylated in presence of liquid  $\text{SO}_2$  as medium and subjected to hydration, is brought near the precipitating point by addition of a coagulant ( $\text{H}_2\text{O}$ ) and the mixture is discharged under pressure in the form of a stream or jet in contact with streams of heated precipitating liquid ( $\text{H}_2\text{O}$ ) and vapour (steam), the  $\text{SO}_2$  being simultaneously liberated as gas within the precipitating ester, thus rendering it loose and fibrous.

F. R. ENNOS.

**Manufacture of mixed esters of cellulose.** KODAK, LTD., Assees. of C. J. MALM (B.P. 346,816, 13.1.30.

U.S., 11.1.29).—Cellulose is acylated with a halogen derivative of an acid of high mol. wt. (stearyl chloride) in the presence of pyridine, and, after separation of the ester, further acyl groups corresponding with acids of low mol. wt. ( $\text{AcOH}$ ) are introduced, preferably in the presence of  $\text{C}_2\text{H}_2\text{Cl}_4$  or  $\text{CHCl}_3$ ; the mixed ester is insol. in  $\text{COMe}_2$  and  $\text{C}_6\text{H}_6$  and sol. in  $\text{C}_2\text{H}_2\text{Cl}_4$  and  $\text{CHCl}_3$ .

F. R. ENNOS.

**Manufacture of cellulose derivatives.** H. DREYFUS (B.P. 345,989, 23.12.29).—Cellulose derivatives containing N are produced from cellulosic material pretreated with mineral and/or organic acids or with alkalis, from incompletely etherified celluloses, or from cellulose carboxylic esters by treatment with organic bases (primary or secondary amines) at  $150\text{--}250^\circ$  under pressure.

F. R. ENNOS.

**Manufacture of organic substitution derivatives of cellulose.**

II. DREYFUS (B.P. 346,806, 18.12.29).—Cellulosic materials are treated in the presence of conc. caustic alkali solution with an aliphatic etherifying agent containing a halogen atom in addition to the etherifying group (epichlorohydrin), or with one containing halogen atoms of different reactivity, so that halogen is retained in the product ( $\text{C}_2\text{H}_4\text{ClBr}$ ); the halogen-substituted derivatives are treated with  $\text{NH}_3$  or amines to yield  $\text{NH}_2$ -ethers of cellulose having an affinity for acid dyes.

F. R. ENNOS.

**Manufacture of organic substitution derivatives of cellulose [(A) nitro- and amino-arylalkyl ethers ;**

(B) amino-esters and aminoalkyl ethers ; (C) cyano-esters and cyanoalkyl ethers ; (D) halogeno-esters and halogenoalkyl ethers]. H. DREYFUS (B.P. 344,420, 344,480, 344,488, and 344,529, [A, B, D] 28.11.29, [C] 2.12.29).—(A) Cellulose is etherified with benzyl chlorides carrying  $\text{NO}_2$ ,  $\text{NH}_2$ , or acylamino-groups, e.g., 2-aminomethylbenzyl chloride, *m*-acetamidobenzyl chloride, *p*-nitrobenzyl chloride.  $\text{NH}_2$  groups may be acylated or alkylated,  $\text{NO}_2$  groups reduced. (B) Hydroxyacyl esters or hydroxyalkyl or carboxyalkyl ethers of cellulose are aminated by successive treatment, e.g., with thionyl chloride and  $\text{NH}_3$  or amines. Suitable starting materials are the glycollate,  $\beta$ -hydroxyethyl ether, malonate, maleate, phthalate, and oxalate of cellulose. (C) Cellulose is esterified with cyano-acid halides, or etherified with cyanoalkyl halides, e.g., cyanoacetyl chloride or chloroacetonitrile; the products may be hydrolysed to carboxylic derivatives or reduced to  $\text{NH}_2$  compounds. (D) A carboxylated or hydroxylated ester or ether of cellulose (glycollate,  $\beta$ -hydroxyethyl ether, malonate, phthalate, etc.) is treated with thionyl chloride or other agent to give halogen derivatives which may be aminated. Amino- and acylamino-compounds prepared according to (A—D) have affinity for wool dyes.

C. HOLLINS.

**Reducing the electric charge of textile fibres.**

ACETA GES.M.B.H. (B.P. 346,912, 24.2.30. Ger., 11.3.29).—While being worked up, textile fibres, particularly artificial fibres from cellulose esters and ethers, are impregnated with a mixture of oils or fats and fatty acids to which is added an organic base forming an oil-soluble soap (dibutylamine, "sapamines," etc.).

F. R. ENNOS.

**Recovery of fibrous material from sugar cane.**

J. J. DE LA ROZA, SEN., ASSR. to BAGASSE PRODUCTS CORP. (U.S.P. 1,782,869, 25.11.30. Appl., 25.3.26).—The fibrous part of the cane is digested with approx. neutral water at about 160° and 6.35 kg./cm.<sup>2</sup> in absence of air.

F. R. ENNOS.

**Digestion of fibrous material.**

T. L. DUNBAR (U.S.P. 1,781,447, 11.11.30, Appl., 1.8.29).—The hot acid relief liquor and gases from one digester are fed into the top of another digester which is being filled to facilitate even packing of the chips.

D. J. NORMAN.

**Manufacture of cellulosic material.**

S. P. CANE, H. HEATHER, and L. G. S. HEBBS (B.P. 346,813, 3.1.30).—After soaking felled trees of *Broussonetia papyrifera* in water or dil. alkali at room temperature or above, the bark and wood are separated by hand or machinery and each is separately converted into pulp by digestion with NaOH in the usual manner; the waste liquor from the digestion is used for the first treatment of a fresh batch of trees.

F. R. ENNOS.

**Cyclic process of fibre liberation.**

G. A. RICHTER, ASSR. to BROWN CO. (U.S.P. 1,781,733, 18.11.30. Appl., 9.3.29).—Cellulosic material is digested first with Na<sub>2</sub>CO<sub>3</sub> solution, *e.g.*, for 3—4 hr. at 160° with a 5—10% solution, and then with Na<sub>2</sub>S solution, *e.g.*, for about 3 hr. at 150—170° with a liquor containing the equivalent of 3.2—4% of Na<sub>2</sub>O. The waste liquors may be regenerated separately, or may be smelted together, the Na<sub>2</sub>CO<sub>3</sub> being then separated from the Na<sub>2</sub>S by crystallisation. The Na<sub>2</sub>CO<sub>3</sub> remaining in the sulphide liquor may be causticised in the usual way. Very little CaO is required in the whole process and the pulp is of high quality.

D. J. NORMAN.

**Manufacture of paper from plant fibre.**

H. EGGERS, ASSR. to A. ORGOVAN, O. S. LESZAY, and I. MATUSOFF (U.S.P. 1,782,382, 18.11.30. Appl., 4.1.29. Renewed 17.10.30).—The material, *e.g.*, leaves, stalks, roots, etc. of potatoes, corn, beans, etc., is cut into small pieces, washed, and mixed with 3% of its vol. of waste papers and 1% of powdered CaCO<sub>3</sub>. This mixture is digested for 3 hr. at about 3 atm. with a 2% solution of NaOH. The resulting pulp may be bleached by treating it with 5% of bleaching powder and 2% of powdered CaCO<sub>3</sub>, and, after about ½ hr., adding NaHCO<sub>3</sub> in sufficient quantity to remove the residual colour.

D. J. NORMAN.

**Manufacture of hard paper or hard fabric.**

INTERNAT. GEN. ELECTRIC CO., INC., ASSCES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 346,411, 6.11.29. Ger., 6.11.28).—Condensation products of polyhydric phenols, *e.g.*, resorcinol, with aldehydes are used as binders in the manufacture of ply fabrics or boards. The artificial resins produced are hardened by heat (about 1 min. at 160°). Up to 50% of the polyhydric phenol may be replaced by monohydric phenols without unduly prolonging the time of hardening.

D. J. NORMAN.

**Manufacture of (A) reinforced filter paper,**

**(B) filter papers and filter fabrics.** F. W. MANNING, ASSR. to FILTER FABRICS, INC. (U.S.P. 1,782,784—5, 25.11.30. Appl., [A] 11.4.27, [B] 29.11.27. Renewed [B] 30.9.30).—(A) The paper is made with a fabric, *e.g.*,

scrim backing, on a cylinder mould, and provision is made for removing the pulp-suspending medium by passing through the sheet suitable liquid or gaseous reagents (*cf. infra*). The paper is finally festoon-dried in a current of warm air. A paper of high porosity is obtained. (B) The paper etc. is made from a suspension of the fibrous material in an inert liquid of sufficiently high surface tension to keep the fibres properly dispersed during sheet formation. Preferably, the surface tension of the suspending liquid should approximate to that of the liquid which is subsequently to be filtered through the paper. Suitable suspending liquids are sugar liquors of  $d > 1.176$  at 80°, glycerin, or oil. Thus cellulose fibres optionally in admixture with other fibrous material, *e.g.*, asbestos and granular treating solids, *e.g.*, bone char, are dispersed in oil and the resulting mixture spread evenly over a fabric support, *e.g.*, scrim, as this is wound on to a rotating foraminous drum. Suction is applied to the interior of the winding drum during the sheeting process and when the reel is full the adhering suspending liquor is removed by a reverse current of naphtha, *i.e.*, from the drum outwards. The naphtha is similarly displaced by superheated steam followed by hot air. The finished paper may be stripped from the fibrous support if desired.

D. J. NORMAN.

**Paper-coating composition.**

D. B. BRADNER and C. H. MILLIGAN, ASSRS. to CHAMPION COATED PAPER CO. (U.S.P. 1,781,716, 18.11.30. Appl., 31.12.27).—The adhesive consists of a chlorinated protein, *e.g.*, casein, which has been treated either before or during the preparation of the coating mixture with 1—2% of active Cl added in a form which will not cause flocculation of the casein, *e.g.*, as NaOCl. The  $p_H$  of the final coating mixture should not exceed 9. The use of chlorinated proteins improves the keeping properties of the composition, and permits a 10—20% reduction in the quantity of adhesive used without impairing the printing qualities of the coated paper.

D. J. NORMAN.

**[Weaving of] filter cloths.**

A. LEMAIRE (B.P. 348,016, 28.1.30. Belg., 29.1.29).

**[Machine for producing] yarns and the like formed from paper [strip].**

R. BROWNLOW, and LATENT LABORATORIES, LTD. (B.P. 348,197, 29.7.30).

**Production of artificial threads by [twisting].**

J. BRANDWOOD (B.P. 348,161, 6.11.29).

**[Nozzle for] wetting of threads.**

ACETA GES.M.B.H. (B.P. 348,407, 23.5.30. Ger., 23.5.29).

**Viscous mixtures.**—See I. Cellulose fibre.—See VI. Glass.—See VIII. Wall-board.—See IX. Plasticised products.—See XIII. Saccharification of cellulose.—See XVII.

**VI.—BLEACHING; DYEING; PRINTING; FINISHING.****Multiple-stage bleaching [of wood pulp].**

L. RYS (Papier-Fabr., 1931, 29, 209—210).—Two-stage bleaching, *viz.*, Cl water followed by dil. NaOCl solution, is recommended. Treatment with dil. alkali, before or after the use of oxidising bleaches, may be advantageous, as many of the incrustants are soluble in alkali alone. It is held that the lignin residues in sulphite pulp are sulphonated to differing degrees, the higher forms being

the more readily treated with Cl water, and the lower in the presence of alkalis. It is claimed that 20–30% less Cl is required than when single-stage bleaching with NaOCl is carried out.

T. T. POTTS.

**Heat of combustion of acid-treated wool in reference to the theory of dyeing.** W. PÄSSLER and W. KÖNIG (*Z. angew. Chem.*, 1931, **44**, 288–291, 304–309).—It has been established by Knecht and Meyer that the dyeing of wool with acid dyes consists in the combination of the acid with free  $\text{NH}_2$  groups in the protein. The present authors have investigated the energy relations of these reactions from the differences between the heats of combustion of treated and untreated wool, applying corrections for the reactions between the amines and  $\text{H}_2\text{O}$  to form bases. The average heat of combustion of washed and dried wool was found to be 5498.8 g.-cal. The acid treatment was carried out on a boiling water-bath and the treated wool washed, although it was recognised that this involves hydrolysis. The acid neutralised was determined by wt. In this way  $\text{H}_2\text{SO}_4$  treatment was found to involve a heat of neutralisation of 33.4 g.-cal. per g. of wool substance converted, a figure so high as to suggest that subsidiary reactions occur. Similar determinations were made of the heats of neutralisation of AcOH, oxalic acid, picric acid, naphthol-yellows, and some other organic acids. In almost all cases the values found were far above those to be expected from theory. Picric acid and naphthol-yellow gave increased figures if  $\text{H}_2\text{SO}_4$  was also present. The results of other workers on the heat balances of the hydrolysis of peptides are discussed; the acid treatment of wool and silk may involve similar reactions of varying degree according to the  $p_{\text{H}}$  of the acid used. In the case of picric acid etc. with addition of  $\text{H}_2\text{SO}_4$ , the formation of "molecular compounds" may occur. To sum up, chemical changes must occur in all cases, and in the case of complex organic acids these may lead to molecular or adsorption compounds being formed. This last is, however, not a complete explanation of the heat values by itself.

C. IRWIN.

**The "küpometer," for determining the reducing value of vat-dye liquors.** J. HAUSNER (*Chem.-Ztg.*, 1931, **55**, 363–364).—A sample of the liquor is pipetted into a small flask, the neck of which is then closed by a tube with side opening, into the bottom of which tube extends the elongated delivery tube of a burette. The tube and burette are filled to the zero mark with water, and by this means the volume contraction due to O absorption when the flask is shaken is measured. The result indicates the total reducing value of unconverted hyposulphite and leuco-compound. The method is not suitable for liquors containing glucose or finished with "rongalite." Otherwise it gives results accurate enough for process control.

C. IRWIN.

**Action of detergents on cotton and linen fabrics.** E. NORLIN and K. I. SKÄRBLÖM (*Ingen. Vetensk. Handl.*, 1930, No. 100, 92 pp.; *Chem. Zentr.*, 1931, i, 1196).—NaOH solution (1–2 g./litre) causes an unexpectedly small diminution in strength if air is excluded. Na silicate solutions caused with cotton a small, and with linen a marked, loss of strength. The action of  $\text{NaBO}_3$

solution depends on the velocity of decomposition. Mg silicate is a good stabilising agent. Marked loss of strength was caused by soaps containing highly unsaturated fatty acids, particularly if the drying is slow, but is avoided by the use of soft water. Retention of the Ca and Mg soaps is evidently followed on drying by autoxidation.

A. A. ELDRIDGE.

**Anthanthrone dyes.**—See IV. **Larval attack of animal fibres.**—See V. **Rubber-impregnated fabrics.**—See XIV.

#### PATENTS.

**[Cellulose] fibre liberating and bleaching process.** G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN CO. (U.S.P. 1,780,943, 11.11.30. Appl., 19.4.27).—A somewhat impure cellulose fibrous product (85–95% cellulose) obtained by digesting chipped wood with a  $\text{NaHSO}_3$  liquor containing about 4% each of combined and free  $\text{SO}_2$  is bleached by successive treatment with a solution of Cl and one containing 5–10% of NaOCl or  $\text{CaOCl}_2$  and 1.5–2.0% of NaOH; the product is highly suitable for the manufacture of paper having a high bursting strength and permanent whiteness.

A. J. HALL.

**Printing of cotton [with vat dyes].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,527, 343,596–7, [A–C] 21.8.29).—Vat dye printing pastes are made up with a selected hydrotropic agent, viz., (A) an alkali dialkylanilinesulphonate; (B) an alkali sulphonate of  $\text{C}_6\text{H}_6$ ,  $\text{C}_{10}\text{H}_8$ , hydrogenated  $\text{C}_{10}\text{H}_8$ , or homologue; (C) an alkali benzoate or hydroxybenzoate or homologue. The pastes may be highly conc., do not settle on storing, and have high tinctorial power. Examples are the Na salts of dimethylmetanilic, dimethyl-*o*-toluidine-4-sulphonic, naphthalene-1- or -2-sulphonic, benzene-sulphonic, tetrahydronaphthalene-2-sulphonic, benzoic, salicylic, and *o*-toluic acids. [Stat. ref.] C. HOLLINS.

**Preparation of emulsions for sizing [vegetable] textile fibres.** G. LEFRANC (B.P. 346,055, 20.1.30).—Oils and fats are emulsified in an aqueous dispersion of casein or gelatin, shellac, etc. in which neutral alkaline borates are used as dispersing agents, and Na silicate solution is finally added. Treatment with this sizing solution may be followed by a deglutinating bath if desired, e.g., in the case of very fine threads.

E. LEWKOWITSCH.

**Making emulsified compositions [for waterproofing of fibrous sheets].** L. KIRSCHBRAUN (U.S.P. 1,781,645, 11.11.30. Appl., 6.5.27. Cf. U.S.P. 1,302,810; B., 1919, 494 A).—An aqueous emulsion of a hot, liquid, waterproof binder dispersed with soap or prepared by adding alkali to a solution of, e.g., oleic acid, in a liquid binder such as asphalts, pitches, drying oils, rubber solutions, etc. is added to and mixed with an aq. paste of colloidal clay etc.

E. LEWKOWITSCH.

**Gasproof balloon fabric.** GOODYEAR TIRE & RUBBER CO., Assees. of C. McK. CARSON (B.P. 347,735, 27.1.30. U.S., 9.3.29).—See U.S.P. 1,779,389; B., 1931, 535.

**Wetting agents etc. Protection of materials.**—See III. **Dyeing of acetate silk.**—See IV. **Cellulose from soiled fibres. Cellulose derivatives. Hard fabrics.**—See V. **Bleaching agent.**—See XIX.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Performance of the Kessler apparatus [in sulphuric acid manufacture].** V. N. SCHULTZ, U. M. VOLCHENKO, I. V. IOSELOVSKI, and L. V. SUPONITZKI (*J. Chem. Ind., Russia*, 1929, 6, 928—940).—The heat utilisation was distributed as follows: concentration of  $\text{H}_2\text{SO}_4$  77.9, outgoing gases 13.2, surrounding medium 8.9%.

CHEMICAL ABSTRACTS.

**Special control apparatus in the U.S. Naval sulphuric acid plant.** H. M. COSTER (*Ind. Eng. Chem.*, 1931, 23, 563—565).—The Schröder-Grillo contact plant in the U.S. Naval Powder Factory, Indian Head, includes a  $\text{SO}_2$  recorder, a conductivity measuring apparatus for determining the concentration of oleum, and an automatic acid diluter. It has been in operation since 1918, except for an idle period of 11 months, and has shown an average conversion and yield of 95 and 94%, respectively.

W. J. WRIGHT.

**Causticisation [of sodium carbonate] and filtration of caustic lime.** MANGER (*Chem.-Ztg.*, 1931, 55, 361—363).—The causticisation of  $\text{Na}_2\text{CO}_3$  with CaO is usually carried out in Europe by batch-working. The CaO sludge is generally washed by decantation. Filter presses and vacuum filters with asbestos cloth have been used, as also sand filters. In the case of continuous working these are replaced by rotary vacuum filters, which are much more economical. Such a filter is described which effects filtration, washing, and the removal of the precipitate in each solution. Mother-liquor and wash-water can be kept separate. Woven metal cloths are used and the CaO sludge is delivered with 0.4—0.5% NaOH content only. The capacity of such filters varies greatly with the quality of the CaO used, and tests on this point are desirable before designing an installation.

C. IRWIN.

**California desert soda.** G. R. ROBERTSON (*Ind. Eng. Chem.*, 1931, 23, 478—481).—The soda content of the water of Owens Lake, Cal., is increasing owing to the withdrawal of water from inflowing rivers for public supply. "Trona" (native Na sesquicarbonate) is dissolved in fresh  $\text{H}_2\text{O}$  and mixed with brine concentrated by solar evaporation so as to give a uniform raw material. This is treated with  $\text{CO}_2$ , prepared by igniting dolomite, and the  $\text{NaHCO}_3$  precipitated is filtered, washed, partly dried, and calcined in a rotary kiln, about 50% of the  $\text{CO}_2$  being recoverable. The product is "light soda ash" of about 98% purity. If necessary, it is converted into "dense ash" by fusion and atomisation or partial hydration and calcination.

C. IRWIN.

**Utilisation of natural Glauber's salt from the Gulf of Karabugaz.** N. D. ZELINSKI, M. A. RAKUZIN, P. P. BORISOV, and A. N. TIROVA (*Bull. Acad. Sci. U.S.S.R.*, 1930, 707—714).—To obtain information regarding the practicability of utilising  $\text{Na}_2\text{SO}_4$  by the ammonia-soda process (cf. Gerlach, A., 1877, ii, 26), laboratory experiments have been made on the electrolysis of  $(\text{NH}_4)_2\text{SO}_4$  solutions at  $80^\circ$  in a cell with a porous diaphragm, a current of air being kept passing through the solution in the cathode chamber. Propor-

tions up to 76% of the  $(\text{NH}_4)_2\text{SO}_4$  may be decomposed in this way and only traces of  $\text{H}_2\text{SO}_4$  are formed. Similar experiments with  $\text{Na}_2\text{SO}_4$  solutions yield  $\text{H}_2\text{SO}_4$  containing only small proportions of  $\text{Na}_2\text{SO}_4$  and only traces of persulphuric or Caro's acid.

T. H. POPE.

**Trisodium phosphate—its manufacture and use.** F. D. SNELL (*Ind. Eng. Chem.*, 1931, 23, 470—474).—A suspension of  $\text{Na}_2\text{CO}_3$  in hot  $\text{H}_2\text{O}$  or mother-liquor is treated with  $\text{H}_3\text{PO}_4$  in slight excess and the solution finally boiled to eliminate all  $\text{CO}_2$ . The solution of  $\text{Na}_2\text{HPO}_4$  is rendered more alkaline by addition of mother-liquor from  $\text{Na}_3\text{PO}_4$  crystallisation and filtered in a plate-and-frame press at  $85$ — $100^\circ$ . The filtrate is treated with slight excess of NaOH at  $90^\circ$ , diluted to  $d_{20}^{20}$  1.34—1.40, filtered hot, and crystallised in water-cooled Swensen-Walker pans, the liquor entering at  $60^\circ$  and leaving at  $30^\circ$ , or it may be spray-congealed by atomisation. Except in the latter case the crystals tend to cake and occasionally other salts such as NaCl, NaOCl, etc. are added to minimise this.  $\text{Na}_3\text{PO}_4$  is used as a detergent on account of its high  $p_{\text{H}}$  value and emulsifying power to oils. It is also used for cleaning metals before electroplating, and as a water softener and paint remover. It is employed in the leather and sugar industries and has a variety of minor uses.

C. IRWIN.

**Tricalcium phosphate and phosphate rock.** T. S. BUIE (*Amer. Fertiliser*, 1931, 74, [9], 13—16).—A review and discussion of recent work.  $\text{Ca}_3(\text{PO}_4)_2$  has a greater citrate solubility and assimilability (Neubauer) than rock phosphate. Partial reversion of superphosphate during the manufacture of ammoniated superphosphates should not materially reduce its efficiency as fertiliser.

A. G. POLLARD.

**Rate of calcination of limestone.** C. C. FURNAS (*Ind. Eng. Chem.*, 1931, 23, 534—538).—Calcination of limestone proceeds from the outer heated surface of the piece at a rate (cm./hr.) given by the empirical equation  $\log_{10} R = 0.003145t - 3.3085$ , where  $R$  is the rate and  $t$  is in  $^\circ\text{C}$ . There is a sharp line of demarcation between calcined and uncalcined material. Two sets of experiments made over a low and a high temp. range showed that in the former the temp. of the centre of a piece rose in the usual manner for the heating of a solid body, but calcination lagged behind this temp. At the higher temp. the centre rose to a definite temp. and remained there while calcination proceeded; this phenomenon is associated with the endothermic nature of the reaction. From observation of the rate of gas evolution a method was developed of estimating the surface area of the irregularly shaped lumps used in the experiments; some of these areas were found to be 50% greater than the area of a sphere of the same vol.

H. INGLESON.

**Gypsum as a raw material of the chemical industry.** E. TERRES (*Z. angew. Chem.*, 1931, 44, 356—363).—There are 3 types of process which, commencing with gypsum, lead to the useful recovery of S. or  $\text{SO}_2$ . In the first,  $(\text{NH}_4)_2\text{SO}_4$  is prepared by double decomposition and then heated with  $\text{Na}_2\text{SO}_4$ . The  $\text{NaHSO}_4$  produced on further heating evolves  $\text{SO}_3$ . The second method is the thermal decomposition of

gypsum or kieserite. The latter is completely dissociated at 1150°, but the presence of Cl and HCl in the gases causes difficulty in their utilisation for  $\text{H}_2\text{SO}_4$  making. The decomposition of gypsum only begins at 1200°, and it is therefore necessary to add  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , or  $\text{Fe}_2\text{O}_3$ . The influence of the three decreases in the order mentioned. A series of dissociation pressure curves for mixtures of gypsum with each is given. This process is still worked at Leverkusen, yielding saleable cement as a by-product. Another process of this type consists in the treatment of gypsum or mixtures containing it with coke in a blast furnace, the product being sulphur. The third group of processes consists in the reduction of gypsum to sulphide by roasting with coke or coal in a rotary kiln. Further treatment may consist in decomposition with  $\text{CO}_2$  and recovery of sulphur by the Chance-Claus process. Or the  $\text{CaS}$  may be decomposed with superheated steam in a shaft furnace:  $\text{CaS} + \text{H}_2\text{O} \rightleftharpoons \text{CaO} + \text{H}_2\text{S}$ . This reaction commences at 1000°, and with rise in temperature the equilibrium moves to the right;  $\text{SO}_2$  is also formed by subsidiary reactions. These are very complex, and as they occur under the conditions present in a gas producer they explain the occasional presence of free S in producer clinker. C. IRWIN.

**"Modified" Caro apparatus for evaluation of calcium carbide.** R. VONDRÁČEK (Chem.-Ztg., 1931, 55, 344).—The apparatus is criticised, in that the  $\text{CaC}_2$  falls directly into the  $\text{H}_2\text{O}$  of the gasholder and the  $\text{C}_2\text{H}_2$  passes into the holder without further washing; the temp. of the gas is said to be the mean of the readings of thermometers in the  $\text{H}_2\text{O}$  and in the gas at the top of the holder. The mean gas temp. is, however, 2–4° higher than the value obtained by this method, whereas the original Caro apparatus gives the correct temp.

H. F. GILLBE.

**Volumetric determination of iodides by silver. Application to the determination of potassium iodide in iodine-iodide tinctures.** P. FLEURY and J. COURTOIS (J. Pharm. Chim., 1931, [viii], 13, 478–484).—The blue colour given to starch solution by I is due to the presence of soluble iodides, and on the precipitation of these by Ag the colour is immediately discharged. In the absence of free HI the tincture may be diluted with water and boiled to remove all but a trace of I, when starch and a little  $\text{HNO}_3$  are added before titration with  $\text{AgNO}_3$ . In the presence of free HI the tincture is evaporated to dryness on the water-bath to remove the acid before proceeding as above. T. McLACHLAN.

**Economics of recovering by-product carbon dioxide.** C. L. JONES (Ind. Eng. Chem., 1931, 23, 519–523).—Factors governing the economic value of various sources of  $\text{CO}_2$  as raw material for the solid  $\text{CO}_2$  industry are discussed. W. J. WRIGHT.

**Machinery to make solid carbon dioxide.** T. MITCHELL (Ind. Eng. Chem., 1931, 23, 523–525).—A diagrammatic sketch of the plant is given, with details of the condensers, liquid coolers, and snow chambers. An installation in Philadelphia has a capacity of 50 tons per day. W. J. WRIGHT.

**Utilisation of fluorine in bone factories.** G. D. LIUBARSKI (Ukraine Chem. J., 1930, 5 [Tech.], 163–165).

—Bone meal and superphosphate contain about 0.08% F. R. TRUSZKOWSKI.

**Gas purification.**—See II. Limestone for road surfaces.—See IX. Iron scale. Chloride volatilisation.—See X. Sulphides and filter cloths.—See XVII.  $\text{NH}_4\text{NO}_3$  from amatol.—See XXII.

## PATENTS.

**Manufacture of sulphuric acid.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,000, 12.9.30. U.S., 13.9.29).—The catalyst used for the oxidation of  $\text{SO}_2$  to  $\text{SO}_3$  is a granular mass of  $\text{MgSO}_4$  containing about 1% of  $\text{Th}(\text{SO}_4)_2$ ,  $\text{Ce}_2(\text{SO}_4)_3$ ,  $\text{BaSO}_4$ ,  $\text{Fe}_2(\text{SO}_4)_3$ , or  $\text{CuSO}_4$ , and impregnated with about 0.3% Pt.

A. R. POWELL.

**Production of sulphuric acid and metals simultaneously, by the electrolysis of sulphuric acid and metallic sulphates.** T. DE PALACIO (B.P. 346,667, 8.1.30).—An outer vessel contains a solution of the metal sulphate as the negative electrolyte, and an inner one the  $\text{H}_2\text{SO}_4$  as the positive electrolyte.  $\text{SO}_2$ , or a liquid saturated with it, is introduced into the anode cell, acting as a depolariser and forming  $\text{H}_2\text{SO}_4$ , and the metal from the sulphate solution is deposited in the cathode cell.

W. J. WRIGHT.

**Treatment [clarification] of phosphoric acid.** C. F. BOOTH, ASSR. to SWANN RESEARCH, INC. (U.S.P. 1,777,548, 7.10.30. Appl., 19.3.28).—A 1.6% solution of  $\text{As}_2\text{O}_3$  in pure  $\text{H}_3\text{PO}_4$  is treated with  $\text{H}_2\text{S}$  until about 90% of the As is converted into colloidal sulphide, and this suspension is added to the  $\text{H}_3\text{PO}_4$  to be clarified (e.g., that obtained from smelting phosphate rock) after previously saturating it with  $\text{H}_2\text{S}$ . The final mixture should contain 200–400 p.p.m. As; on setting aside for a few days, all the As settles as sulphide and collects the suspended impurities.

A. R. POWELL.

**Ammonia oxidation [catalyst].** E. SMYTH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,800, 11.12.29 and 30.1.30).—Sagging of the catalyst gauze through softening of the  $\text{SiO}_2$  rods is prevented by supporting the latter on metal tubes through which  $\text{H}_2\text{O}$  passes. The latter are provided with collars at the points where they cross the  $\text{SiO}_2$  rods, so that if slight sagging of the gauze should occur the latter will not come in contact with the metal.

W. J. WRIGHT.

**Catalytic conversion of ammonia into nitric oxide.** F. JOST (B.P. 346,247, 24.7.30. Ger., 3.8.29).—Fine-meshed nets of Pt or other suitable substance, which have been subjected to pressure, hammering, or rolling, are used as catalysts for the oxidation process.

H. ROYAL-DAWSON.

**Manufacture of sodium hydroxide and ammonia.** A. MENTZEL (B.P. 347,426, 26.3.30. Ger., 26.3.29).— $\text{NaHCO}_3$  is heated with C in an atm. of N at 1000° to form  $\text{NaCN}$ , which is then treated with steam at 400° to produce  $\text{NaOH}$ ,  $\text{NH}_3$ , and  $\text{CO}$ . The  $\text{NaOH}$  is purified by dissolution in  $\text{H}_2\text{O}$ , filtration, and evaporation.

A. R. POWELL.

**Fixation of ammonia-nitrogen.** N. CARO and A. R. FRANK (B.P. 347,641, 29.1.30. Ger., 30.1.29).—A mixture of  $\text{NH}_3$  and air with or without steam and/or  $\text{CO}_2$  is passed at 50–300° over sawdust, peat, lignite,

or other vegetable material containing or resembling humic acids or capable of forming humic acids, or into aqueous suspensions of these materials, whereby partial oxidation of the organic material takes place with the absorption of 10–20% of  $\text{NH}_3$ . The products are suitable for use as fertilisers and for case-hardening steel. A. R. POWELL.

**Preparation of ammonium sulphate.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 346,508, 10.2.30. Ger., 16.2.29).—Dry  $\text{SO}_2$  and  $\text{NH}_3$  in the mol. ratio 1 : 2 are passed downwards into a tube internally heated at 100–120° and cooled externally, whereby the compound  $\text{NH}_2 \cdot \text{SO}_2 \cdot \text{NH}_4$  condenses on the walls of the tube. This compound is removed continuously as a fine powder by means of a suitable rotating stirrer and falls downwards against a rising current of moist air, which oxidises it to  $(\text{NH}_4)_2\text{SO}_4$ . A. R. POWELL.

**Production of potassium nitrate and sodium carbonate.** CHEMIEVERFAHREN-GES.M.B.H. (B.P. 347,935, 4.6.30. Ger., 13.6.29).—The mother-liquor from the  $\text{NaHCO}_3$  obtained by the Solvay process is treated with  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , to obtain  $\text{CaCO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$ , the ppt. is removed, and the solution treated with sylvinit and  $\text{NH}_3$ , whereby  $\text{KCl}$  and glaserite are deposited. The crystals are drained and treated with  $\text{H}_2\text{O}$  to obtain a solution of  $\text{NaCl}$  and  $\text{NH}_4\text{Cl}$ , which is returned to the Solvay process, and a residue of  $\text{K}_2\text{SO}_4$ . The latter is mixed with the  $\text{CaCO}_3$  previously obtained and the mixture treated with  $\text{HNO}_3$  to form  $\text{KNO}_3$  and also  $\text{CaSO}_4$  and  $\text{CO}_2$ , which are used again in the process. A. R. POWELL.

**Manufacture of monosodium glutamate and like glutamic acid salts.** H. D. HARDIE & Co., LTD., and A. C. STIRRAT (B.P. 347,258, 23.1.30).—Gluten flour is hydrolysed with  $\text{HCl}$ , the solution filtered, evaporated to half its vol. *in vacuo*, and treated with an equal vol. of conc.  $\text{HCl}$ , and the ppt. of glutamic acid hydrochloride is washed with  $\text{HCl}$  and dissolved in boiling  $\text{H}_2\text{O}$ . The solution is treated with sufficient  $\text{NaOH}$  to neutralise the  $\text{HCl}$ , and the ppt. of glutamic acid is collected, washed, dried, and dissolved in sufficient dil.  $\text{NaOH}$  to produce mono- $\text{Na}$  glutamate. A. R. POWELL.

**Manufacture of dilithium alkali citrates.** W. W. TRIGGS. From J. A. WÜLFING (B.P. 346,208, 17.5.30).—A solution of citric acid in an aliphatic alcohol (preferably 96–98%  $\text{EtOH}$ ) is treated with 1 mol.  $\text{Li}_2\text{CO}_3$  and, after removal of  $\text{CO}_2$  by boiling, with 1 mol.  $\text{KOH}$  or  $\text{NaOH}$  dissolved in a little  $\text{H}_2\text{O}$ ; the white cryst. product is filtered off, washed with abs.  $\text{EtOH}$ , and dried. L. A. COLES.

**Purification of complex compounds containing phosphorus and tungsten.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,074, 25.7.30. U.S., 25.7.29).—Mother-liquors containing  $\text{Na}$  phosphotungstate are treated with  $\text{HCl}$  and an alkyl-substituted arylamine, e.g.,  $\text{NPhMe}_2$ . The precipitate of amine phosphotungstate is collected and distilled with sufficient  $\text{NaOH}$  to recover the amine and re-form  $\text{Na}$  phosphotungstate, which is then crystallised out. A. R. POWELL.

**Manufacture of phosphorus or oxygen compounds thereof and gaseous mixtures.** Soc.

D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTRI., Assees. of E. VOITURON (B.P. 346,038, 9.1.30. Ger., 9.1.29).—A mixture of  $\text{Ca}_3(\text{PO}_4)_2$ , coal, and  $\text{SiO}_2$  or  $\text{Al}_2\text{O}_3$  is heated in a blast of air enriched in  $\text{O}$  and, after removal of  $\text{P}$  or its compounds, the resultant gas mixture is treated with steam, under raised pressure if desired, to yield a mixture which, after removal of  $\text{CO}_2$  etc., is suitable for the synthesis of  $\text{NH}_3$  or for the successive syntheses of  $\text{MeOH}$  and  $\text{NH}_3$ . L. A. COLES.

**Manufacture of calcium carbide.** E.M.S. INDUSTRIAL PROCESSES, LTD., and A. T. DE MOUILPIED (B.P. 347,763, 4.2.30).—Carbide sludge is mixed with powdered coal and the mixture heated at a temp. sufficient to carbonise the coal and convert the  $\text{Ca}(\text{OH})_2$  into  $\text{CaO}$ . The resulting cokey product containing 100 pts. of  $\text{CaO}$  to 65 pts. of  $\text{C}$  is charged directly into the carbide furnace. A. R. POWELL.

**Production of cyanamides of alkaline-earth metals.** Soc. D'ETUDES CHIM. POUR L'IND., and E. DE LUSERNA (B.P. 346,463, 24.12.29. Ger., 24.12.28).—A mixture of  $\text{CaCO}_3$  and  $\text{C}$  is heated at 950° in a current of  $\text{NH}_3$ . [Stat. ref.] A. R. POWELL.

**Manufacture of strontium oxide.** I. G. FARBEN-IND. A.-G. (B.P. 346,404, 10.1.30. Ger., 12.1.29).—Moist  $\text{SrCO}_3$  heated to 1200° in a rotary furnace.

H. ROYAL-DAWSON.

**Manufacture of chlorine compounds [anhydrous metal chlorides].** A. CARPMAEL. From I. G. FARBEN-IND. A.-G. (B.P. 347,137, 22.1.30).—The ore is heated in a rotating furnace with the flame produced by the combustion of a mixture of  $\text{CO}$ ,  $\text{O}$ , and  $\text{Cl}$  in the ratio 2 : 1 : 1.  $\text{AlPO}_4$  yields  $\text{POCl}_3$  and  $\text{AlCl}_3$ , bauxite  $\text{AlCl}_3$  and a residue of  $\text{SiO}_2$ , ilmenite  $\text{FeCl}_3$  and  $\text{TiCl}_4$ , and  $\text{Ca}_3\text{P}_2\text{O}_8$   $\text{POCl}_3$  and  $\text{CaCl}_2$ . A. R. POWELL.

**Dehydration of [magnesium and cerium] chlorides.** Soc. DE PROD. CHIM. DES TERRES RARES (B.P. 347,182, 18.1.30. Fr., 18.1.29).—Cryst.  $\text{MgCl}_2$  containing 0.05–0.5%  $\text{CaCl}_2$  is thrown in a thin layer on to heavy metal plates heated at 450°, whereby the  $\text{H}_2\text{O}$  is rapidly evolved leaving a porous mass which can be readily converted into the pure anhyd. salt by subsequent heating in a current of  $\text{HCl}$  gas. The process is applicable to the dehydration of cerium and other rare-earth chlorides. A. R. POWELL.

**Purification of anhydrous aluminium chloride containing iron.** J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 347,510, 26.5.30).—Crude  $\text{AlCl}_3$  containing  $\text{FeCl}_3$  is treated with  $\text{COCl}_2$  and sufficient  $\text{Fe}$  or  $\text{Al}$  to reduce  $\text{FeCl}_3$  to  $\text{FeCl}_2$ , whereby a clear solution is obtained; this is then stirred under pressure with sufficient  $\text{Cl}$  to re-form  $\text{FeCl}_3$ , which settles out on keeping for some time. The clear solution is decanted and the  $\text{COCl}_2$  removed by distillation. The remaining  $\text{AlCl}_3$  contains <0.1%  $\text{FeCl}_3$ . A. R. POWELL.

**Manufacture of a decolorising clay.** W. S. BAYLIS, ASSR. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,781,265, 11.11.30. Appl., 14.5.29).—Bentonite is thoroughly mixed with 25–50% of its wt. of  $\text{H}_2\text{SO}_4$  and a further quantity of clay equal to 3–5 times the weight originally used is mixed with the charge, which is

then kept for 24 hr. to allow the reactions to complete. The mass is then heated to expel excess acid and to dehydrate the colloidal material without decomposing the salts formed in the acid treatment. A. R. POWELL.

**Zeolite.** A. O. JAEGER (ASSR. to SELDEN CO.) and J. A. BERTSCH (U.S.P. 1,782,353, 18.11.30. Appl., 6.4.26).—Catalytically active zeolites containing at least one complex basic compound in non-exchangeable form together with diluents, one of which acts as an activator of the base, are claimed. One or more of the constituents may be added in a low-valency state and converted into a higher-valency state during the manufacture. Eleven examples are given, suitable for the reduction of aromatic  $\text{NO}_2$  compounds, as hydrogenation or chlorination catalysts, for oxidation of alcohol vapours, for converting water-gas-steam mixture into  $\text{CO}_2$  and  $\text{H}_2$ , etc. A. R. POWELL.

**Reviving kieselguhr and like material.** C. F. ARMSTRONG (B.P. 346,472, 18.1.30).—Kieselguhr that has been used for clarifying sugar solutions is revived by boiling it with 10%  $\text{HCl}$  for 30 min. and washing with hot  $\text{H}_2\text{O}$  until free from chlorides. A. R. POWELL.

**Manufacture of pure zinc oxide and carbonate.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,849, 5.4.30).—The grey paste of  $\text{ZnO}$  or  $\text{ZnCO}_3$  obtained as a residue in the manufacture of  $\text{Na}_2\text{S}_2\text{O}_4$  or of alkali formaldehyde-sulphoxylates is agitated with water and a mild oxidising agent, *e.g.*,  $\text{H}_2\text{O}_2$  or  $\text{NaOCl}$ . A. R. POWELL.

**Manufacture of zinc sulphide.** W. W. TRIGGS. From ST. JOSEPH LEAD CO. (B.P. 347,799, 24.2.30).—Blende is heated in a current of steam in an electric furnace, whereby pure  $\text{ZnS}$  sublimes in a finely-divided crystalline form. A. R. POWELL.

**Treatment of silica or compounds or combinations thereof, and production of products therefrom.** I. P. LLEWELLYN, T. J. I. CRAIG, A. KIRKHAM, and P. SPENCE & SONS, LTD. (B.P. 347,223, 24. and 29.1.30).—Finely-divided or gelatinous  $\text{SiO}_2$  is treated in suspension in  $\text{H}_2\text{O}$  with small quantities of  $\text{NaOH}$  and then with a solution of a  $\text{Mg}$  salt, *e.g.*,  $\text{Mg}(\text{HCO}_3)_2$ ,  $\text{MgCl}_2$ , or  $\text{MgSO}_4$ . The product is filtered, washed, and dried; it forms a useful heat-insulator. A. R. POWELL.

**Production of nickel carbonyl.** MOND NICKEL CO., and C. M. W. GRIEB (B.P. 347,208, 16.1. and 5.7.30).—In the reduction of roasted  $\text{Ni-Cu}$  matte by producer gas in the Mond process, a small quantity of  $\text{NiSO}_4$  is added to the charge or a small quantity of  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ , or  $\text{COS}$  to the reducing gas so as to produce a reduced material containing 0.2–5%  $\text{S}$  as sulphide. Subsequent treatment of this material in the volatilisers gives a very high yield of  $\text{Ni}(\text{CO})_4$  even in the presence of much  $\text{CO}$ . A. R. POWELL.

**Manufacture of cyanogen halides.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,989, 15.8.30. U.S., 16.8.29).—A 4% solution of  $\text{HCN}$  is treated at 20–30 lb. pressure with 95% of the theoretical  $\text{Cl}$  at below  $30^\circ$ . A. R. POWELL.

**Purification of sulphur.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,835, 18.1.30).—The  $\text{S}$  at  $120$ – $150^\circ$  is treated with a strong mineral acid,

*e.g.*,  $\text{H}_2\text{SO}_4$  (40–50%), any tarry matter being subsequently removed by treatment with an oxidising agent, *e.g.*,  $\text{HNO}_3$ . W. J. WRIGHT.

**Production of chlorine and hydrogen.** IMPERIAL CHEM. INDUSTRIES, LTD., C. L. HIGGINS, and J. T. BARKER (B.P. 346,470, 17.1.30).— $\text{HCl}$  produced by the action of  $\text{H}_2\text{SO}_4$  on  $\text{NaCl}$  is absorbed in  $\text{H}_2\text{O}$  and the solution electrolysed between  $\text{C}$  electrodes in a divided cell to give  $\text{H}$  and  $\text{Cl}$ . The effluent from the cell, containing 8–11%  $\text{HCl}$ , is returned partly to the condensers and partly to the stills. A. R. POWELL.

**Fixation or recovery of chlorine [from electrolytic cells].** E. O. BARSTOW, ASSR. to DOW CHEM. CO. (U.S.P. 1,781,830, 18.11.30. Appl., 30.10.26).—Hot  $\text{Cl}$  from the electrolysis of  $\text{NaCl}$  or  $\text{MgCl}_2$  is cooled by dilution with air and the gases are mixed with  $\text{SO}_2$  and passed through a  $\text{H}_2\text{O}$  scrubber to obtain  $\text{H}_2\text{SO}_4$  and  $\text{HCl}$ . A. R. POWELL.

**Electrolytic manufacture of gaseous fluorine.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,774, 9.1. and 27.5.30).—The U-shaped electrolysis vessel has tubular attachments below, through which the electrodes pass, the jointing material consisting of solid alkali di- or poly-fluoride used as electrolyte. The electrodes may also be inserted through the top or sides, the jointing material being then contained in a cup. W. J. WRIGHT.

**Production of phosphorus by the volatilisation process.** VICTOR CHEM. WORKS, ASSEES. of H. W. EASTERWOOD (B.P. 346,539, 6.3.30. U.S., 23.3.29).—Phosphate rock, coal, and siliceous material are mixed with sulphite-cellulose liquor and 1–5% of the fume obtained in a previous volatilisation process, the mixture is briquetted, and the briquettes are smelted in a blast furnace. A. R. POWELL.

**Production of phosphorus and phosphorus-oxygen compounds from raw phosphates.** METALLGES. A.-G. (B.P. 347,937, 4.6.30. Ger., 11.6.29).—A damp mixture of coarsely ground phosphate rock, sand, and 6–10% of coke dust is sintered in a Dwight-Lloyd machine and the sinter roughly broken and smelted with coke in a blast furnace having an auxiliary oil heater at its lower end. A. R. POWELL.

**Hydrogen chloride.**—See II. Catalysts for oxygenated compounds.—See III. Galvaniser's waste. **Oxidation catalyst.**—See X. Electrolysis of alkaline salts.—See XI. Pigments.—See XIII. Tanning preparations.—See XV. Fertiliser.—See XVI. Purifying air.—See XXIII.

## VIII.—GLASS; CERAMICS.

**Argillaceous colloids and their applications in ceramics.** R. DUBRISAY (Bull. Soc. d'Encour., 1931, 130, 176–182).—Suspensions of purified kaolin were treated with solutions of  $\text{HCl}$  and alkalis in varying concentrations, and the conditions of stabilisation and flocculation were noted. Considerable variations in the vol. of the deposit were observed, depending on whether the suspension was stable or flocculent. A study of the deposits tended to support Le Chatelier's view that clay particles have a lamellar structure. F. SALT.



**Manufacture and characteristics of laminated glass.** W. L. MORGAN (Ind. Eng. Chem., 1931, 23, 505—508).—The process of the Triplex company is described. Elaborate precautions for cleanliness are necessary, spraying with gelatin is done under cover, and the air of the pressing room is filtered and washed. Developments in the industry consist mainly of greater mechanisation. C. IRWIN.

**Refractory materials for the [iron or steel] induction furnace.** J. H. CHESTERS and W. J. REES (Iron & Steel Inst., May, 1931. Advance copy. 16 pp.).—In addition to withstanding the furnace temp., the ability of the lining to sinter and prevent crumbling, the resistance to corrosion, and the burning shrinkage are all necessary properties required. Batches of varying mixture and grading have been tested in the form of rammed crucibles which were filled with slag and maintained at 1500°. Austrian magnesite possessed good slag resistance, but cracked owing to thermal shock. Electrically fused magnesite showed high resistance, although severe slag entry took place along small cracks. In zircon-ball clay mixtures the latter was eroded and a pot failed apparently through softening. MgO-zircon mixtures were less satisfactory than was MgO alone, and the inclusion of crushed SiO<sub>2</sub> brick did not effect any improvement in the acid linings. The best grading of an acid lining of calcined ganister and boric acid was 45% on 20-mesh (I.M.M.), 10% on 20—60-mesh, 45% through 60-mesh (approx. half of this passing 120-mesh). The use of pre-burned bricks would simplify the problem of making linings.

C. A. KING.

**Slagging of refractory materials. V. Relation between slagging of grog bricks and flux content and porosity.** H. SALMANG and O. HEBESTREIT (Feuerfest, 1931, 7, 1—8; cf. B., 1931, 491).—Small crucibles were made from synthetic 1:2 mixtures of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>, with and without the addition of fluxes, and were tested for resistance to simple slag mixtures. The addition of the fluxes appeared to have no effect, probably owing to the high porosity of the bodies. The addition of 2% of flux to crucibles made of 70% of fired and 30% of unfired Zettlitz kaolin made little difference; 4% of flux increased the slag attack slightly. Slag attack was greatest when the oxides TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CaO were present in the crucible only or in the slag only; the attack was less when the same oxide was present both in the crucible and in the slag. Na<sub>2</sub>O produced little effect. Experiments with normal bricks, blast-furnace slag high in CaO, and reheating-furnace slag showed that slag attack was not entirely dependent on porosity. The structure of the refractory is an important factor; material having practically closed pores is attacked equally by the more viscous CaO slags and the thin Fe slags. F. SALT.

## PATENTS.

**Manufacture of (A) reinforced, (B) splinterless, glass.** BRIT. CELANESE, LTD. (B.P. 347,777 and 347,972, [A] 10.2.30, [B] 16.7.30. U.S., [A] 12.2.29, [B] 31.7.29).—(A) A glass sheet is coated successively with an adhesive (gelatin or a synthetic resin and, preferably, a cellulose

derivative with plasticisers or softeners) and with one or more layers of a solution of a cellulose derivative; after evaporation of the solvent, the cellulose layer is coated with the adhesive, and a covering glass sheet is united to it by heat and pressure. (B) A reinforcing layer comprising a cellulose derivative and at least 50% of a plasticiser (triacetin, ethyl or butyl tartrate or phthalate, etc.) is softened by soaking in a high-boiling liquid (BuOH, cyclohexanol, dioxan, etc.) and united to two sheets of glass by heat and pressure without the use of an adhesive. L. A. COLES.

**Manufacture of laminated glass.** J. H. SHERTS and R. E. HAMILL, Assrs. to DUPLATE CORP. (U.S.P. 1,781,084, 11.11.30. Appl., 18.10.29).—The edges of a laminated plate made up of alternating sheets of glass and cellulose acetate are sealed by immersing them in a bath of a pyroxylin plastic solvent, e.g., tolyl phosphate, and heating the solvent under pressure until the cellulose acetate softens and is forced into the joints.

A. R. POWELL.

**Manufacture of ceramic articles such as insulators [having a glazed, sanded surface].** BULLERS, LTD., and J. E. HARRIS (B.P. 347,612, 28.1.30).—The articles are dipped in a solution of a combustible adhesive (gum tragacanth), covered with sand, sprayed with glaze, and fired. L. A. COLES.

**Plastic compositions [of refractories].** BRIT. THOMSON-HOUSTON Co., LTD., Assces. of L. NAVIAS (B.P. 346,683, 29.10.29. U.S., 13.11.28).—Finely-ground ceramic or refractory material, e.g., ZrO<sub>2</sub>, MgO, or feldspar, is made into a paste with flour and a small quantity of NH<sub>3</sub> solution, the paste shaped as desired, dried at 100°, and fired at about the m.p. of the refractory. A. R. POWELL.

**Cutting or abrading compositions [cobalt-tungsten carbide].** A. E. WHITE. From FIRTH-STERLING STEEL Co. (B.P. 346,473, 18.1.30).—Powdered WC is plated with 3.5—13% of its wt. of Co, the mass is dried, pressed into the desired shape, sintered at 1000°, cooled, forged to the final shape, and again sintered at 1450° in H. A. R. POWELL.

**Glass-annealing lehrs.** PILKINGTON BROTHERS, LTD., and E. B. LE MARE (B.P. 347,587, 28.12.29).

**Lehrs, furnaces, etc. for annealing glassware.** UNITED GLASS BOTTLE MANUFRS., LTD., F. A. HURLBUT, and W. A. MOORSHEAD (B.P. 348,354, 28.4.30).

**[Baking and annealing] lehrs for [decorated] glassware.** BRIT. HARTFORD-FAIRMONT SYND., LTD., Assces. of H. L. EASTMAN (B.P. 348,481, 15.8.30. U.S., 15.8.29).

**Manufacture of [wired] glass sheets.** CHANCE BROS. & Co., LTD., and A. L. FORSTER (B.P. 348,291, 18.3.30).

**[Apparatus for] coating of surfaces with [powdered] glass.** S. ARNOLD (B.P. 348,187, 20.2.30).

**[Apparatus for] manufacture of bricks or the like.** T. JONES, R. BARKER, and D. C. MURRAY (B.P. 347,845, 3.4.30).

**Filter presses.**—See I.

## IX.—BUILDING MATERIALS.

**Quick-setting silicate of soda cements for acid-proof tank and tower construction.** F. D. SNELL and H. FARKAS (Ind. Eng. Chem., 1931, 23, 525—529).—A mixture of Na silicate and inert material normally requires 7—10 days to set and never sets in a saturated atm. For rapid hardening (24 hr.) an acid material is added. A cement containing a neutral or alkaline material hardens comparatively quickly, but remains soft sufficiently long to give time for working it. It is flexible enough to allow for the strains incidental to building, but when set is able to carry the load of additional structure, and adheres to brick, metal, wood, or glass. The details and requirements of acid-proof masonry work are described. W. J. WRIGHT.

**Action of gypsum and other setting retarders on Portland cement clinker.** L. FORSÉN (Zement, 1930, 19, 1130—1134, 1155—1160; Chem. Zentr., 1931, i, 1149).—A discussion. A. A. ELDRIDGE.

**Action on concrete of water containing carbon dioxide.** N. SUNDIUS (Beton u. Eisen, 1930, No. 2, 14 pp.; Chem. Zentr., 1931, i, 1149).—The amount of CO<sub>2</sub> in water plays a subordinate part in the attack of pervious concrete. With dense concrete the rate of flow of the water is important; with a slow rate a high CO<sub>2</sub> content has little action. A. A. ELDRIDGE.

**Silication of limestone for road surfaces.** L. WASILEWSKI and K. CZARNECKI (Przemysl Chem., 1931, 15, 156—164).—The resistance of limestones to compression and erosion as well as their impermeability to H<sub>2</sub>O are greatly increased by treatment with Na silicate, the best results being obtained using material of 13—17% porosity and not less than 16% silicate solutions, in which the ratio SiO<sub>2</sub> : Na<sub>2</sub>O > 3. Where this ratio greatly exceeds 3 silication is incomplete. The durability of the product increases up to 5% of adsorbed silicate, above which little further difference is observed. The quantity of solutions necessary varies with the porosity of the limestone and with the proportion of finely-ground limestone to coarse grains; the best results are obtained using 0.3 m.<sup>3</sup> of powdered per m.<sup>3</sup> of coarsely broken limestone. Limestone containing over 5% of clay is not suitable for silicate road-surfacing; whilst sand does not interfere with the process. Solid Na silicate containing 25% H<sub>2</sub>O, and soluble in cold H<sub>2</sub>O, can be prepared in a special autoclave.

R. TRUSZKOWSKI.

**Chemistry of Australian timbers.** W. E. COHEN and H. E. DADSWELL (J. Counc. Sci. Ind. Res., Australia, 1931, 4, 45—48).—A programme for their systematic chemical examination is outlined. The results so far achieved indicate that modifications to existing methods for determining cellulose, lignin, etc. will be necessary. Thus the eucalypt woods are very resistant to the action of Cl, and the subsequent determination of  $\alpha$ -cellulose by treatment with MOH is rendered impossible. The lignin values also are too high owing to incomplete extraction of extraneous matter by C<sub>6</sub>H<sub>6</sub>—EtOH. Karri and jarrah woods may be differentiated by chemical means; the cellulose contents being 55.69—63.74% and 38.86—51.98%, respectively, whilst the alkalinities of the ash

are 0.43—0.91 and 0.01—0.16 c.c. of 0.1N-acid, respectively. H. J. DOWDEN.

**Fresh-air chambers.**—See I. Gypsum.—See VII.

## PATENTS.

**Production of porous concrete insulating material.** TORKRET GES.M.B.H. (B.P. 346,572, 10.4.30. Ger., 15.5.29).—Kieselguhr is added in such amount to the pouring mass as to render it plastic without loss of expansion capacity. H. ROYAL-DAWSON.

**Rendering concrete surfaces acid-proof.** I. KOTANI (B.P. 346,053, 20.1.30).—The surface is coated first with a mixture of asbestos and porcelain, talc, steatite, or agalmatolite powder, or with a mixture of the powders, and then with aq. Na silicate, and allowed to dry. H. ROYAL-DAWSON.

**Manufacture of porous [building] material.** H. W. GREIDER, Assr. to PHILIP CAREY MANUFG. CO. (U.S.P. 1,782,384, 18.11.30. Appl., 27.6.29).—Moulded heat-insulators are formed by mixing light, finely divided, and/or fibrous inorganic materials, e.g., MgCO<sub>3</sub> and asbestos, with H<sub>2</sub>O containing alkali, and generating gas therein by incorporating a gas-forming substance, e.g., CaC<sub>2</sub>, which is coated with a resinous, oily, waxy, or a fatty acid substance, e.g., oleic acid, which on interaction with the alkali will produce a permanent froth. The mass is then moulded under pressure with expulsion of H<sub>2</sub>O, but without destroying the minute bubbles.

B. M. VENABLES.

**Production of slabs for walls etc.** A. P. LAURIE (B.P. 346,516, 20.2.30).—Granulated cork (4 pts. by wt.) is mixed with 5 pts. of conc. Na (or K) silicate, moulded, and dried. The slab is then placed in a solution of CaCl<sub>2</sub> or MgCl<sub>2</sub> and when reaction is complete the soluble salts are washed out. To enable the mixture to be rolled into slabs, 2 pts. of Na silicate (viscosity 90 c.g.s. units) should be mixed with 1 pt. by wt. of cork. C. A. KING.

**Manufacture of mastic asphalt and similar materials [for roads etc.].** W. E. BRAHAM (B.P. 346,515, 19.2. and 8.3.30).—Earthy materials, previously untreated by drying etc., are mixed cold with an asphaltic binder and then are gradually heated while being constantly stirred, e.g., in a rotary drum.

C. A. KING.

**Treatment of bagasse for manufacture of wall-board and the like.** W. L. S. WILLIAMS, Assr. to HAWAIIAN CANE PRODUCTS, LTD. (U.S.P. 1,782,755, 25.11.30. Appl., 9.8.29).—After separation of the material into fibrous and pith portions, the former is digested with alkali (hydrated CaO, NaOH, etc.) under pressure to produce a clear fibre and refined, whilst the latter is cooked in water to effect the desired degree of hydration, the resultant stocks being subsequently mixed. F. R. ENNOS.

**[Lumber] kiln.** J. A. WORDEN (U.S.P. 1,783,030, 25.11.30. Appl., 24.6.29).—Adjacent the kiln proper is a return passage for air running from end to end and containing a heater and fan, and beneath the kiln are two outer and one middle longitudinal passages with ports communicating with the interior of the kiln. If

the outer passages are used for supply of heated air they decrease in depth from the end where the warm air enters, whilst the centre passage for used air slopes the other way, the effect being that the circulation within the kiln is up the sides and down the centre, mainly in a transverse direction. B. M. VENABLES.

[Tractor device for] treatment of railways, roads, and other surfaces with chemical solutions. M. GRATTEPAIN, and FILS D'ALBERT COLLET (B.P. 348,294, 19.3.30. Fr., 20.3.29).

Preparing damp mixtures. Centrifugal extractor.—See I. Bituminous solutions.—See II. Fibre board.—See V. Vulcanisation of rubber[—wood joints].—See XIV.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Basic Bessemer process. Considerations of its possibilities in England. V. HARBORD (Iron & Steel Inst., May, 1931. Advance copy. 17 pp.).—Consideration of the justification of the erection of a basic Bessemer plant would rule out the Cleveland and S. Wales districts, due to lack of local ore. Northamptonshire and Lincolnshire are more favoured and with an admixture of foreign high-P ore the total costs would compare well with those of Belgian or Luxemburg works. The plant would have to restrict its production to mild steel for re-rolling into miscellaneous semi-finished products, as the process is not controlled sufficiently accurately for high-class structural work. Some of the most important factors in favour of the Bessemer process are the lower labour costs, fuel costs, and depreciation, and the higher credit for slag; the total saving is estimated at 7s. per ton. Typical analyses of Northampton, Rutland, Lincoln, Luxemburg, and Lorraine ores, and comparative costs of basic Bessemer and basic open-hearth processes are given. C. A. KING.

Blast-furnace data and their correlation. II. E. C. EVANS, L. REEVE, and M. A. VERNON (Iron & Steel Inst., May, 1931. Advance copy. 68 pp.).—The mathematical expression of Evans and Bailey (B., 1928, 405) has been confirmed, the figure for C used other than at the tuyères being calc. from gas analyses. The curve showing the Fe-C relation is of hyperbolic form, which indicates that beyond a certain output further advantage is counterbalanced by increased C requirements. The mean time of stock descent for optimum fuel consumption and output is lower with rich than with poor burdens, and the most important factor in efficiency is effective contact between ore and gas. This leads particularly to careful control in treatment of ore and quality of coke. Owing to the disparity in external heat losses in winter and summer, investigation into any advantage in insulation of the shaft is necessary. C. A. KING.

Blast-furnace slag and its utilisation. E. I. ORLOV (Ukraine Chem. J., 1930, 5, [Tech.], 139—153).—An exposition of the methods of treatment of slag for conversion into industrial products. R. TRUSZKOWSKI.

Fuel control in the iron and steel industry. G. V. SLOTTMAN (J. Inst. Fuel, 1931, 4, 275—280).—The manufacture of 1 ton of steel from iron ore requires

the energy of 2 tons of coal. Economy is obtained by utilising the heat of blast-furnace gas, coke-oven gas, and flue gas, the fluctuations of load being carried by a gasholder, and auxiliary coal firing. Present tendencies in combustion control, gas cleaning, and regenerator furnace and burner design are discussed.

D. K. MOORE.

Production economy in iron and steel works. I. O. CROMBERG (Iron & Steel Inst., May, 1931. Advance copy. 17 pp.).—The desirability of a systematic method of production-costing by timing the various work stages of manufacture, and of the co-operated working between "order" and "stock" departments, with a view to a more continuous process output, is examined.

C. A. KING.

Effect of carbon and silicon on the growth and scaling of grey cast iron. A. L. NORBURY and E. MORGAN (Iron & Steel Inst., May, 1931. Advance copy. 22 pp.).—Dilatometer measurements on grey cast irons containing 4—2.1% total C and 1.6—7.6% Si indicated that as the Si increases up to 3—4% the growth increases. With higher Si content decrease in growth resulted, due to the Si raising the crit. point and also increasing the resistance to oxidation. Accelerated growth tests in an atm. of moist CO<sub>2</sub> confirmed these results, and the increase in Si is so effective that growth and scaling may be reduced to negligible quantities even under severe service conditions. A mixture of CO<sub>2</sub> and SO<sub>2</sub> was found to be particularly severe in its action. Growth is due to conversion of combined C into graphite, both external and internal oxidation, and the formation of cracks, which are caused probably by the lack of ductility in the metal or surface-oxidised layers. Irons containing 4—10% Si in conjunction with finely-divided graphite are being developed commercially.

C. A. KING.

Constitution of [iron] scale. L. B. PFEIL (Iron & Steel Inst., May, 1931. Advance copy. 19 pp.; cf. B., 1929, 437).—The microstructure and cooling curves of the range of composition of Fe oxide, as found in iron scale, has been studied. Pure oxides of desired composition were prepared by mixing oxides of high and low Fe content followed by prolonged heating *in vacuo* to attain homogeneity. Most of the refractory oxides were definitely soluble in Fe<sub>2</sub>O<sub>3</sub> and Pt boats were used for melting the oxide. In the FeO-Fe<sub>2</sub>O<sub>3</sub> system the f.p. decreased with increasing Fe content, but with more than 77% Fe contamination of the couple rendered the results valueless. Double arrests on the curve occurred with 75.5, 74.84, and 74.5% Fe, only one arrest with 73.86% Fe, and double arrests appeared again below this value. The Fe<sub>2</sub>O<sub>3</sub> phase probably dissociates at high temp. with formation of magnetite. The equilibrium composition at 1000° of Fe<sub>2</sub>O<sub>3</sub> was 70.3% Fe, of magnetite 72.35—72.5% Fe, and of the ferrous phase 76.9%. The last-named never corresponds to the compound FeO (77.75% Fe) without the presence of metallic Fe. The microstructure of scale produced by the oxidation of pure Fe at high temp. in air consists of all three phases. C. A. KING.

Formation of ferrite from austenite. (SIR) H. C. H. CARPENTER and J. M. ROBERTSON (Iron & Steel Inst., May, 1931. Advance copy. 35 pp.).—Experiments with

three C steels cooled at four different rates show that the mode of formation of ferrite from austenite is controlled by the rate of cooling and the C content of the austenite. The two factors act in a similar way, by influencing the growth of ferrite through their effect on the diffusion of C in the austenite. These factors do not completely determine the final structure, however. Three types of ferrite crystals are distinguished: (1) smooth crystals, sometimes rounded and sometimes sharp in outline, (2) irregular crystals, and (3) elongated crystals that form along the crystallographic planes in the austenite. A certain rate of cooling and concentration of C in the austenite are necessary in order to produce elongated crystals, but an increase in the rate of cooling or C content brings about a progressive change in the shape of the ferrite crystals. Ferrite always appears, in the first instance, at the boundaries of the austenite grains, but the manner of its growth in these regions varies with the rate of cooling and the C content. Large masses of residual austenite always transform into pearlite, but films may give rise to cementite. By decreasing the size of the particles of residual austenite an increase in the rate of cooling favours this formation, but by tending to promote the formation of more nuclei in a given vol. it causes even the smallest particles to transform into pearlite. An increase in the rate of cooling has two opposing effects, therefore. These results are embodied in a theory of the structural change. The factors, C content and rate of cooling, exercise their control through their effect on (1) the temp. at which the change begins, (2) the diffusion of C in the austenite, (3) the amount of C taken up by the ferrite, (4) the extent to which ferrite crystals coalesce. The work described includes many types of ferrite-pearlite structures and provides further information on the Widmanstätten and banded structures, the sub-boundaries in ferrite, and the cementite boundaries in low-C steel.

E. S. HEDGES.

**Sub-crystalline structure of ferrite.** C. O. BANISTER and W. D. JONES (Iron & Steel Inst., May, 1931. Advance copy. 15 pp.).—Three different types of sub-crystalline structure in ferrite have been observed in the microscopical examination of wrought Fe. Type I is associated with the presence of P in the metal and has been observed in wrought Fe and mild steel. It is detected after deeply etching a polished surface with 5%  $\text{HNO}_3$  in EtOH. Etching reagents containing  $\text{CuCl}_2$  also develop the structure readily. No trace of the structure can be detected after annealing for 5 hr. at  $820^\circ$ . Type II has been found in bars of wrought Fe, but is most readily distinguished in specimens after prolonged annealing in an atm. of H. This structure consists of a distinct veining of the ferrite crystals. Type III has been found in wrought Fe specimens after prolonged etching with  $\text{HNO}_3$  and is a modification of type II structure. It is generally found in isolated crystals, although it sometimes affects a considerable area as fine markings. Annealing the specimens, followed by mild quenching, causes disappearance of this structure. It is suggested that types II and III are manifestations of microscopical or sub-microscopical inclusions in the metal. The effects of such inclusions are discussed.

E. S. HEDGES.

**X-Ray investigations on the crystal structure of hardened steel.** E. ÖHMAN (Iron & Steel Inst., May, 1931. Advance copy. 19 pp.).—X-Ray spectrographic examination of hardened steels has confirmed that tetragonal martensite ( $\alpha'$ ) is a supersaturated solution of C in  $\alpha$ -Fe. On effective quenching, the  $\alpha'$ -phase has the same C content as the  $\gamma$ -phase from which it was formed. The most probable distribution of the C atoms is a complex substitution of a group of two C atoms for one Fe atom. Decomposition of the  $\alpha'$ -phase on tempering takes place continuously with a progressive decrease of the axial ratio. Causes of the hardness of quenched steel are discussed, and it is concluded that all the known causes producing hardness may contribute to this end.

E. S. HEDGES.

**Accelerated cracking of mild steel (boiler plate) under repeated bending.** W. ROSENHAIN and A. J. MURPHY (Iron & Steel Inst., Mar., 1931. Advance copy. 20 pp.).—When bending occurs at intervals of 24 hr. with intervening rests, the metal being continuously submerged, visible cracking and fracture occur at earlier stages in tap water and brine than in air or a 50% solution of NaOH. The greatest reduction in endurance is produced by tap water. When there is no interval between successive deflexions the endurance is the same in all four media.

C. A. KING.

**Surface decarburisation of steel at heat-treating temperatures.** W. E. JOMINY (Dept. Eng. Res. Univ. Michigan, 1931, Bull. No. 18, 51 pp.).—Of all the constituent gases of a gas-furnace atm. moist H had by far the greatest decarburising action, which was apparent on heating steel for 24 hr. at  $540^\circ$ . Dry H had little effect in 1-hr. heating at  $870^\circ$ , whilst pure N caused no decarburisation. Steel was decarburised by  $\text{CO}_2$  at  $730^\circ$  and above, though there was less activity when mixed with 50% of steam, which by itself had only a slight effect at  $788^\circ$ , at which temp. air produced little decarburisation in 5 hr. Variations in pressure ( $\frac{1}{2}$ — $1\frac{1}{2}$  atm.) or in the rate of flow of the gases were negligible. The more strongly oxidising is the furnace atmosphere (as measured by the percentage of free O), the less the tendency to decarburise; conversely, increasing the excess of gas increased this tendency, and consequently no relief from decarburisation is to be expected by using a high ratio of gas to air, although scaling is reduced by such means. Heating in a so-called neutral or slightly oxidising atm. is recommended; e.g., with 4% of free O ordinary carbon steel may be heated at  $840^\circ$  for 1 hr., whereas 10 min. is the limit if 2% of CO is present. Tables of limiting times and compositions of furnace atm. are given.

C. A. KING.

**X-Ray investigation of certain nickel steels of low thermal expansion.** G. PHRAGMÉN (Iron & Steel Inst., May, 1931. Advance copy. 10 pp.).—An X-ray spectrographic examination of iron-nickel alloys containing about 36% Ni has shown that the low thermal expansion of these alloys is a property of the face-centred cubic phase. The low coeff. of thermal expansion is not due to a two-phase reaction, which compensates the normal dilatation of the two phases, and there does not appear to be any reason to assume the presence of a body-centred cubic phase in the

low-expansion alloys. In alloys containing 20–40% Ni the size of the elementary cube increases with the Ni content, but with greater amounts of Ni it decreases with the Ni content.

E. S. HEDGES.

**Alloys for use at high temperatures. Complex iron-nickel-chromium alloys. III. Effect of composition and exposure to high temperatures.**

C. H. M. JENKINS and H. J. TAPSELL (Iron & Steel Inst., May, 1931. Advance copy. 29 pp.; cf. B., 1931, 24).—The results of short and prolonged stress tests at 800° have been studied in relation to composition with alloys containing Ni 30, Cr 30, W 4, and (Fe + Si + C) 36%. After heating between 600° and 1100°, alloys of this type undergo an increase in Brinell hardness and a change in microstructure. The most suitable alloys for stress resistance appear to be those which maintain, after the initial period, a fairly constant Brinell hardness after exposure to high temp. The greatest resistance to prolonged stress occurs in the alloy containing C 1.5% and Si 1%. In view of the high percentage of Ni and Cr, these alloys remain in the austenitic condition throughout the temp. range between their m.p. and room temp. The hardening action cannot be ascribed, therefore, to the suppression of the transformation in Fe. An alloy otherwise similar, but free from W, exhibits similar changes of microstructure, but undergoes no appreciable increase of hardness on heat-treatment. The strength of the W-free alloy under prolonged tensile tests is low. The phenomena described bear some resemblance to the age-hardening of Al alloys. The hardness changes appear to occur simultaneously with the separation of a new constituent, but it is possible that hardness changes due to other phase separations may occur and not be accompanied by a visible structural change. The extent and velocity of the changes differ widely in the various alloys examined. On account of the gradual changes which produce hardening, these materials retain their high initial strength for long periods at 800°. The somewhat narrow range of composition in which the most favourable properties occur appears to be associated with the separation of a special compound or phase. The tendency of a material to harden at the service temp. is probably accelerated by deformation, such as is likely to occur in an alloy subjected to high stress in service.

E. S. HEDGES.

**Resistance of copper-nickel steels to sea action.**

J. N. FRIEND and W. WEST (Iron & Steel Inst., May, 1931. Advance copy. 5 pp.).—Test-bars were subjected to the action of sea-water as in previous work (B., 1927, 844) for 2 years. The addition of Cu up to 3.7% increased the resistance of steel to corrosion. The most resistant alloy contained 1.16% Cu and 3.75% Ni. In general, forged alloys suffered slightly greater corrosion than corresponding annealed bars, but there was no general correlation with fineness of grain.

C. A. KING.

**Resistance of metals and alloys to the action of salt solutions. II. Solid salts. III. Aqueous solutions.** E. MAASS and W. WIEDERHOLT (Korrosion u. Metallschutz, 1930, 6, 241–250, 265–277; Chem. Zentr., 1931, i, 1161–1162).—II. The action on Ni,

Cu, Fe, and their alloys, and on Al alloys depends on the moisture-attracting power and the nature of the anion.  $\text{MgCl}_2$  had the greatest, and  $\text{MgSO}_4$  the least, action; carnallite, "Hartsalz," KCl, and NaCl were also used. Ni-Cr steels were most resistant;  $\alpha$ - and  $\beta$ -brass gave white corrosion products.

III. In sylvine, carnallite,  $\text{Na}_2\text{SO}_4$ , and  $\text{MgSO}_4$  solutions, Fe suffers the greatest, and Ni-Cr steels the least, corrosion. Sylvine causes the greatest corrosion. The degree of corrosion is, in general, less than with the solid salts. Materials containing Cu acquire a protective layer when treated with  $\text{MgSO}_4$ . Access of O increases the corrosion.

A. A. ELDRIDGE.

**Effect of sodium chloride solutions on the course of corrosion.** E. K. O. SCHMIDT (Korrosion u. Metallschutz, 1930, 6, 250–255; Chem. Zentr., 1931, i, 1161).—The effects of repeatedly dipping laural rods in NaCl solutions (1–20%) on the physical properties are described.

A. A. ELDRIDGE.

**Unusual corrosion problems.** F. B. PORTER (J. Amer. Water Works' Assoc., 1931, 23, 534–537).—Failure of boiler tubes and oil-well piping has been attributed to internally generated electric currents produced when steel is in contact with an electrolyte and when there are differences in temp. and composition.  $p_{\text{H}}$  values of 9.0 or over are recommended as a means of preventing corrosion in pipe lines and in boiler steam drums below the water line. It is advisable to mix some boiler water with a feed of turbine condensate and distilled water make-up to reduce corrosion in the economisers.

C. JEFSON.

**Chloride volatilisation process [for ores].** R. H. BRADFORD and C. M. MACFARLANE (Univ. Utah Dep. Min. Met. Res. Tech. Paper, 1928, 3, 21 pp.).— $\text{CaCO}_3$  was increasingly deleterious to chloride volatilisation as the amount in the gangue increased above 20%;  $\text{H}_2\text{O}$  vapour was beneficial with ores having a high calcareous gangue. HCl, which was a more active chloridiser than NaCl or  $\text{CaCl}_2$ , was prepared by spraying a solution of  $\text{CaCl}_2$  or NaCl into the roasting furnace,  $\text{SiO}_2$  being necessary with NaCl. For a Cu ore an atm. containing 4–11% O is desirable.

CHEMICAL ABSTRACTS.

**Development of processes for treatment of crude ore, accumulated dumps of tailing and slime at Broken Hill, N.S.W.** MEMBERS OF THE BROKEN HILL BRANCH OF THE AUSTRAL. INST. MIN. MET. (Proc. Austral. Inst. Min. Met., 1930, 379–444).—An historical account is given of the development of flotation processes on the Zn-Pb ores of the Broken Hill district.

A. R. POWELL.

**Composition and examination of some blends.** G. FREE (Chem.-Ztg., 1931, 55, 353–354).—Notes on the various impurities which occur in blende concentrates in different parts of the world and their effect on the determination of Zn and Pb in the blende.

A. R. POWELL.

**Electrolytic determination of copper in steel.** L. ANDERSON (Chemist Analyst, 1931, 20, No. 2, 7–8).—The metal (5 g.) is dissolved in 3N- $\text{H}_2\text{SO}_4$  (100 c.c.); the residue is treated with hot dil.  $\text{HNO}_3$  and the filtrate is electrolysed.

CHEMICAL ABSTRACTS.

**Electrogalvanising of wires and strips at high current densities.** D. V. STEPANOV, B. D. KABANOV, and N. T. KUDRIAVTSEV (*Tsvet. Met.*, 1930, 1151—1158).—Circulation of the bath permits the use of higher c.d. than usual (200—400 amp./dm.<sup>2</sup>). At 50 amp./dm.<sup>2</sup> H<sub>2</sub>O (30 litres), H<sub>2</sub>SO<sub>4</sub> (1 kg.), and H<sub>3</sub>BO<sub>3</sub> (10 g.) must be added to a 1000-litre bath per hr.; the best temp. is 45°. At higher c.d. the temp. should be higher. Cooling is effected by air-stirring. Tables show the change of voltage with temp. and with the distance between the electrodes, and optimal conditions for c.d. of 50, 100, and 200 amp./dm.<sup>2</sup> are specified. **CHEMICAL ABSTRACTS.**

**Gas producing in steel works.**—See II. Na<sub>3</sub>PO<sub>4</sub>.  
—See VII. Refractories for furnaces.—See VIII.

## PATENTS.

**Cupola furnace operation.** V. S. DURBIN (U.S.P. 1,778,524, 14.10.30. Appl., 11.2.29).—The furnace is provided with two sets of tuyères, one some distance above the other. The melting is started with the lower set open and the upper set closed, and after tapping the metal produced the furnace is run with the upper set open and the lower set closed. **A. R. POWELL.**

**Operation of blast furnaces.** J. C. HOPKINS and A. OSOLIN (U.S.P. 1,780,485, 4.11.30. Appl., 13.5.29).—The furnace is tapped with the blast on and plugging is effected by forcing a stream of moist clay from a "mud gun" against the stream of molten Fe. **A. R. POWELL.**

**Annealing furnace.** G. B. SHIPLEY and H. ALINDER, Assrs. to INTERNAT. NICKEL CO., INC. (U.S.P. 1,782,481, 25.11.30. Appl., 4.1.29).—A continuous furnace adapted for annealing tubes, rods, etc. is claimed. **C. A. KING.**

**[Metal]-heating furnaces.** R. E. ELLIS. From RUST FURNACE CO. (B.P. 346,381, 6.12.29).—A furnace for re-heating bars, billets, etc. is described. **B. M. VENABLES.**

**Recovering [volatile] metals [from slag].** U. A. GARRED, Assr. to ANACONDA COPPER MINING CO. (U.S.P. 1,782,418, 25.11.30. Appl., 8.4.27).—Pulverised coal, oil, or gas is introduced with air under high pressure into molten slag. The fuel should be in sufficient excess to reduce the metallic oxide to the volatile metal, which is later re-oxidised by the admission of air at low pressure. Cold materials or slag may be added to blast-furnace slag before this treatment. **C. A. KING.**

**Heat-treatment of magnetisable metals [steel].** H. C. KNERR (U.S.P. 1,779,604, 28.10.30. Appl., 21.4.21. Renewed 11.3.30).—The steel is suspended within a magnetic field by means of a counterbalance wt. somewhat less than the wt. of the steel and heated until it loses its magnetic properties, whereupon it automatically falls into a quenching bath. **A. R. POWELL.**

**Manufacture of metals [iron, chromium, copper] from their halides.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 346,921, 27.2.30).—The chlorides are heated in an externally heated, revolving Fe drum through which a current of H is passed. FeCl<sub>2</sub> is reduced at 700°, CrCl<sub>2</sub> at 950°, and Cu<sub>2</sub>Cl<sub>2</sub> at 450°, yielding the corresponding metals in fine crystals of high purity. **A. R. POWELL.**

**Preparation of metal [steel] for painting.** J. H. GRAVELL, Assr. to AMER. CHEM. PAINT CO. (U.S.P. 1,781,507, 11.11.30. Appl., 2.10.28).—The steel is heated or treated with a caustic alkali to remove oil and grease, then washed with or immersed in a solution of H<sub>3</sub>PO<sub>4</sub> containing CS(NH<sub>2</sub>)<sub>2</sub> to remove scale and coat the surface with Fe<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, and finally washed in a 5% solution of Na<sub>2</sub>HPO<sub>4</sub> to neutralise the acid. **A. R. POWELL.**

**Ferrous alloy [cast iron resistant to scaling].** B. D. SAKLATWALLA (U.S.P. 1,781,527, 11.11.30. Appl., 29.10.27).—Cast iron containing 0.5—5% Cu, 0.5—6% Si, 0.1—2% Al, and 2.0—3.5% C is claimed. **A. R. POWELL.**

**[Nickel-chromium-iron alloy for] roller-mill guide.** M. GORHAM, Assr. to MICHIGAN STEEL CASTING CO. (U.S.P. 1,783,148, 25.11.30. Appl., 7.5.28).—The use of an alloy containing approx. Ni 32—38, Cr 13—17, C 0.35—0.65, Si 0.9—1.4, Mn 0.85—0.95%, remainder Fe, is claimed for the wearing parts of guides for use in the rolling of hot or cold metals. **B. M. VENABLES.**

**Manufacture of a magnetic material [nickel-cobalt-iron alloy].** STANDARD TELEPHONE & CABLES, LTD., G. LAWTON, and W. H. DEARDEN (B.P. 346,788, 17.1.30).—An alloy of 45% Ni, 25% Co, and 30% Fe is melted with 0.1—0.5% C to remove O and, after casting, is rolled and drawn into wire. The wire is annealed in H at 900—1250° to remove excess C. **A. R. POWELL.**

**Inhibitor [for metal-pickling baths].** R. E. LAWRENCE, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,780,595, 4.11.30. Appl., 9.10.29).—Mustard oils, particularly allylthiocarbimide, are claimed. **A. R. POWELL.**

**[Inhibitor for] the pickling of metals etc.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & CO. (B.P. 346,992, 15.4.30).—A mercaptan of the type R·SH, where R is a hydrocarbon radical or an anthraquinonyl group, either of which may contain alkyl, NH<sub>2</sub>, and/or alkoxyl groups, but no other substituents, is claimed. To accelerate the action of H<sub>2</sub>SO<sub>4</sub> pickles, NaCl is also added to the bath. **A. R. POWELL.**

**[Inhibitor for the] acid treatment [pickling] of metal [steel] articles.** R. E. LAWRENCE, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,780,594, 4.11.30. Appl., 9.10.29).—Thioglycollic acid or one of its derivatives, e.g., the Et ester, is claimed. **A. R. POWELL.**

**Utilisation of galvaniser's waste.** W. G. WARING (U.S.P. 1,780,323, 4.11.30. Appl., 6.7.29).—The material is ground in H<sub>2</sub>O in a ball mill and the metallic particles are separated by screening or gravity methods. The remaining slurry is treated with Ca(OH)<sub>2</sub> sufficient to liberate the NH<sub>3</sub> and precipitate the metals as hydroxides, the mixture is distilled to recover the NH<sub>3</sub>, and the residual slurry aerated to oxidise the Fe. The ppt. is collected, washed free of chlorides, and digested with (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> solution at 10—25° under reduced pressure to extract the Zn and Cd, leaving a residue of PbCO<sub>3</sub> and Fe(OH)<sub>3</sub>. The Zn solution is treated with Zn dust to remove Cd and distilled to recover NH<sub>3</sub> and

precipitate  $\text{ZnCO}_3$ . The Pb is extracted from the carbonate residue by  $\text{AcOH}$ . A. R. POWELL.

**Treatment of [crushed] copper ores preparatory to [percolation-]leaching.** C. E. CARSTENS, ASSR. to ANACONDA COPPER MINING CO. (U.S.P. 1,782,841, 25.11.30. Appl., 17.8.25).—The oxide ores (including the fines) are moistened with 4–5% of  $\text{H}_2\text{O}$  or previous wash solutions, before introduction into the leaching vat. H. ROYAL-DAWSON.

**Forming (A) [aluminium], (B) magnesium, or (C) zinc, ingots for working.** T. D. STAY and W. HOLZHAUER, ASSRS. to (A) ALUMINUM CO. OF AMERICA, and (B) AMER. MAGNESIUM CORP. (U.S.P. 1,777,657–9, 7.10.30. Appl., [A] 11.2.29, [B, C] 4.6.29).—The metal is poured into a mould which is heated above the m.p. of (A) Al, (B) Mg, or (C) Zn, and then lowered from the heating zone gradually into a zone in which the metal is cooled rapidly from the bottom upwards, so as to obtain a uniform, equiaxed crystal structure free from porosity and segregations. A. R. POWELL.

**Lead alloy amenable to hardening by heat treatment.** M. G. CORSON (U.S.P. 1,780,261, 4.11.30. Appl., 8.6.28).—Pb alloys containing 0.5–15% Sb and 0.5–15% Cd are claimed. Hardening is effected by annealing at 270–280° for 1–2 hr. followed by air-cooling or quenching and ageing. A. R. POWELL.

**Hard metallic compositions [tungsten carbide-cobalt alloys].** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of R. L. ADAMS (B.P. 346,632, 23.7.30. U.S., 31.7.29).—Lampblack is heated at 1500° in H to remove volatile matter, the product is mixed with  $\text{WO}_3$ , and the mixture made into a paste with a solution of  $\text{Co}(\text{OAc})_2$ . The paste is dried and heated to decompose the acetate, and the resulting powder is heated in a graphite crucible in H at 1150°, cooled, pressed into shape, and sintered as usual. A. R. POWELL.

**Improving the properties of silver alloys containing manganese.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,451, 13.1.30).—An alloy of Ag consisting of > 75% Ag, < 4% Cu, 4.5–25% Mn, 0–5% Al, and 0–8% of one or more other metals forming solid solutions with Ag is heated above 600° but below the m.p., quenched in water, and aged at 100–400°. A. R. POWELL.

**Separation of gold and antimony contained in sulphide of antimony ores.** H. W. C. ANNABLE, and FERRO ALLOY CO. OF AFRICA (PROPRIETARY), LTD. (B.P. 347,680, 27.1.30).—The ore is roasted with  $\text{NaHSO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{MgSO}_4$ ,  $\text{Al}_2(\text{SO}_4)_3$ , NaCl, and  $\text{H}_2\text{SO}_4$  or similar materials or mixtures thereof, until all As and sulphide-S are expelled; the product is then subjected to amalgamation and/or cyanidation. A. R. POWELL.

**Oxidation catalyst [containing rhenium].** SIEMENS & HALSKE A.-G., and W. and I. NODDACK (B.P. 346,652, 8.10.29).—A mixture of 90% of W powder and 10% of Re as a film on quartz is an efficient catalyst for the oxidation of  $\text{SO}_2$ , and a solution of Re oxide in HCl acts as a catalyst in the oxidation of nitrite to nitrate by air at 70–90°. A. R. POWELL.

**[Steel] welding rods for electric arc-welding.** QUASI-ARC CO., LTD., and A. P. STROHMENGER (B.P.

346,587, 1.5.30).—The rods are made by drawing down a composite billet having a core of an alloy steel rich in Cr, Mn, and/or Ni and an outer sheath of mild steel.

A. R. POWELL.

**Rod for use in arc-welding or cutting.** J. B. GREEN, ASSR. to F. M. WARDEN (U.S.P. 1,783,013, 25.11.30. Appl., 10.2.27).—The rod is coated with a paste of 24 pts. of U oxide and 13 pts. of Na tungstate mixed with  $\text{H}_2\text{O}$ .

A. R. POWELL.

**Apparatus for electrodeposition of iron.** R. D. PIKE (U.S.P. 1,782,909, 25.11.30. Appl., 20.7.26).—A cell for the purpose comprises a number of alternate anode and cathode compartments separated by porous diaphragms, insoluble anodes suspended in the anode compartments, and rotating-disc cathodes mounted on a shaft and partly dipping into the electrolyte in the cathode compartments. Means for introducing fresh electrolyte into the cathode compartments, an overflow weir for maintaining a constant level of catholyte, means for heating and causing a downward circulation of anolyte in the anode compartments, means for withdrawing the net flow of anolyte from the cell and maintaining its level slightly below that of the catholyte, and a thermally insulated hood covering the exposed parts of the cathode discs are also provided.

J. S. G. THOMAS.

**Chromium plating.** W. M. PHILLIPS, ASSR. to GEN. MOTORS RES. CORP. (U.S.P. 1,780,535, 4.11.30. Appl., 30.6.26).—The bath comprises a solution of  $\text{H}_2\text{CrO}_4$  containing < 12.5 g. of sulphate per litre, and the anode (of Fe low in C) is preferably of a similar shape to that of the article to be plated.

A. R. POWELL.

**Producing a two-toned finish [chromium plate] on metal surfaces.** M. J. UDY (U.S.P. 1,780,212, 4.11.30. Appl., 28.7.27).—The surface is polished, a design stencilled on it by means of "re-etching ink," and the article then chromium-plated in the usual bath. The parts not inked are plated bright, but plating of the inked parts is retarded until the bath etches the surface in these parts so that the subsequent plating has a frosted effect.

A. R. POWELL.

**Electrolytic production of magnesium.** MAGNESIUM PRODUCTION CO., LTD. (B.P. 347,001, 24.4.30. Ger., 24.4.29).—In the process described in B.P. 345,518 (B., 1931, 539), part of the spent electrolyte rich in KCl is added to the  $\text{MgCl}_2$  undergoing dehydration so as to prevent the accumulation of too high a proportion of KCl in the electrolytic bath.

A. R. POWELL.

**Refining antimony by electrolysis of acid electrolytes.** H. ROSCHER, ASSR. to NORDDEUTS. AFFINERIE (U.S.P. 1,780,944, 11.11.30. Appl., 5.10.27. Ger., 18.7.27).—The electrolyte contains 200 g.  $\text{Sb}_2(\text{SO}_4)_3$ , at least 100 g. of free  $\text{H}_2\text{SO}_4$ , and 60 g. HF per litre. Sb containing more than 5% Pb and 8% Sn may be used as anode, but As must first be removed by fusion with an alkali.

A. R. POWELL.

**[Automatic] casting of metals [e.g., iron or steel].** WETHERILL-MORRIS ENG. CO. (B.P. 348,058, 28.1.30. U.S., 19.2.29).

**Moulds for casting copper.** H. WADE. From AMER. METAL CO., LTD. (B.P. 348,333, 9.4.30).



Roasting apparatus.—See I. Fixation of ammonia-nitrogen. Metals by electrolysis.—See VII. Furnaces for zinc smelting. Metal-melting furnace. Iron anodes.—See XI. Pigments for painting etc.—See XIII. Printing plates.—See XXI.

### XI.—ELECTROTECHNICS.

Applications of electrical heating elements in the chemical industry. H. WINKELMANN (Chem. Fabr., 1931, 4, 217).—A description of some of the uses of totally enclosed and insulated heating elements, particularly for the production of large crystals, the manufacture of many pharmaceutical preparations, and the heating of culture media, with notes on installation, operation, and operating costs. H. F. GILLBE.

Cheaper power. Boiler feed-water control.—See I. Dielectric const. of petroleum etc. Petroleum emulsions. Analysis of petroleum.—See II. Glauber's salt.  $\text{Na}_2\text{PO}_4$ .—See VII. Refractories for induction furnaces.—See VIII. Determining Cu in steel. Electrogalvanising.—See X.

### PATENTS.

[Surrounded-pool-type] induction furnaces. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of P. II. BRACE (B.P. 347,986, 12.8.30. U.S., 14.8.29).—Part of some or all of the turns of the energising coil are deformed out of the normal path of the turn so that the greater part of the turns remains in close contact with the crucible during heating and cooling of the furnace. J. S. G. THOMAS.

[Vertical-shaft] electric furnaces [for smelting zinc ore etc.]. W. W. TRIGGS. From ST. JOSEPH LEAD CO. (B.P. 347,718, 17.12.29).—Corresponding numbers of pairs of spaced electrodes, connected with a source of current supply, enter the upper and lower portions of a reduction chamber comprising independently supported sections between which outlets are arranged for passage of gas from the reduction chamber. J. S. G. THOMAS.

Electric melting furnace [for light metals]. F. LINNHOFF, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,782,359, 18.11.30. Appl., 7.2.29. Ger., 11.2.28).—In a furnace comprising an upper portion surrounded by an induction coil, and a lower portion of smaller cross-sectional area surrounded by a resistance heater, metal is first melted by induction-heating, and then heated to casting temp. by resistance-heating. J. S. G. THOMAS.

Photoelectric pyrometer. Soc. C.E.M.A. (B.P. 346,620, 25.6.30. Fr., 16.7.29).—A process and apparatus for comparing the effects produced on a photoelectric cell by the radiation from the body, the temp. of which is to be measured, and from a standard source of radiation, is claimed. J. S. G. THOMAS.

Carbonised iron anode [for mercury-vapour rectifier]. L. SMEDE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,781,305, 11.11.30. Appl., 11.10.27).—An iron anode, the surface of which is case-hardened or carbonised, preferably by heating in a carbonaceous vapour, is claimed. J. S. G. THOMAS.

Unilateral conductor for rectifying alternating current. H. G. ANDRÉ (U.S.P. 1,782,129, 18.11.30. Appl., 29.4.25. Fr., 26.12.24. Renewed 11.4.30).—The conductor comprises a tubular Ag anode containing colloidal material mixed with  $\text{H}_2\text{SO}_4$  ( $d$  1.83) and  $\text{SO}_3$ , and a helical cathode of Ni alloy with particles of Ag between the convolutions of the helix. J. S. G. THOMAS.

Zinc-lead electric accumulators. A. POUCHAIN (B.P. 347,010, 29.4.30. Ger., 8.5.29. Addn. to B.P. 290,665; B., 1927, 217).—A portion only of the constituents of the electrolyte is initially introduced into the accumulator, other solid constituents being stored on the electrodes or in the container. Thus, *e.g.*,  $\text{H}_2\text{SO}_4$  may be introduced into the accumulator and additional Zn placed on the negative plates. J. S. G. THOMAS.

[Dielectric for] electrical condenser. S. RUBEN, Assr. to RUBEN PATENTS CO. (U.S.P. 1,781,033, 11.11.30. Appl., 6.5.27).—A mixture of finely-powdered, cryst. dielectric material, *e.g.*,  $\text{TiO}_2$ , and a binder, *e.g.*, petroleum jelly, is claimed. J. S. G. THOMAS.

Manufacture of electrical insulating material. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 346,951, 19.3.30. Ger., 21.3.29).—A mixture of  $\text{Al}_2\text{O}_3$  or  $\text{Al}(\text{OH})_3$ , K silicate,  $\text{H}_2\text{O}$ , and, if desired, asbestos is allowed to set and heated to reduce the  $\text{H}_2\text{O}$  content to about that of natural mica. J. S. G. THOMAS.

Electrical insulating materials [for submarine conductors]. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 346,382, 6.12.29).—Material composed of wholly or partly desiccated gutta-percha and/or balata together with not more than 50% of petroleum jelly is claimed. The material is preferably heat-treated, as in B.P. 326,481 (B., 1930, 522). J. S. G. THOMAS.

Impregnation of carbon electrodes [for use in the electrolysis of alkaline salts]. O. A. LAUBI, Assr. to BOZEL MALETRA SOC. INDUSTR. DE PROD. CHIM. (U.S.P. 1,779,242, 21.10.30. Appl., 10.9.29. Fr., 21.12.28).—The electrodes are impregnated with a paraffin hydrocarbon containing  $\text{C}_{20}$  or over per mol., *e.g.*, paraffin, partly or wholly halogenated by substitution and/or addition. J. S. G. THOMAS.

[Gas-filled] electric [negative] glow-discharge devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of T. E. FOULKE (B.P. 346,808, 19.12.29. U.S., 19.12.28).—Two similar, spaced, sheet-metal electrodes, *e.g.*, of Ni, partly backed with insulating and/or refractory material, are arranged in the discharge vessel so that their adjacent longitudinal edges are parallel to the axis of the vessel. A mixture of Al and Mg, applied near the junction of the leads and electrodes, is used as a getter, and a mixture of CaO and red P is applied to the lamp stem. J. S. G. THOMAS.

Apparatus for the electrical precipitation of suspended particles from gases. LODGE-COTTELL, LTD. From METALLGES. A.-G. (B.P. 346,635, 29.7.30).—Two precipitating chambers are interconnected by a gas flue having an openwork gable-shaped top, arranged above the dust-collecting space and provided with baffles, vanes, etc. for distributing the gas, so that gas

can pass from one chamber to the other without traversing the dust-collecting space below the collecting electrodes.

J. S. G. THOMAS.

**Electrically conducting coils or windings.** I. G. FARBENIND. A.-G. (B.P. 345,944, 24.12.29. Ger., 28.12.28).—The coils etc., which are insulated with asbestos, are coated with the cement mass described in B.P. 267,396 (B., 1927, 678), then allowed to harden at room temp., and dried at 200°. Coils to be used at about 400° should be boiled in water for about 20 hr. to remove alkali and then dried.

H. ROYAL-DAWSON.

**Electric furnaces [for dental etc. work].** W. I. ROBERTSON (B.P. 348,119, 7.2.30).

**Manufacture of electric accumulators.** SOC. ANON. DES ACCUMULATEURS MONOPLAQUE (B.P. 348,112, 5.2.30. Fr., 30.4.29).

**Formaldehyde from methane.**—See III. **Metals by electrolysis.** Cl and H. Cl from electrolytic cells. **Fluorine.**—See VII. **Insulators.**—See VIII. **Magnetisable metals.** **Magnetic alloy.** **Cobalt-tungsten carbide.** **Arc-welding.** **Deposition of Fe.** **Chromium-plate.** **Magnesium.** **Antimony.**—See X. **Rubber goods.**—See XIV. **Surgical dressing.**—See XX.

## XII.—FATS; OILS; WAXES.

**Deacidification [of fats] with alcohol.** E. SCHLENKER (Chem. Umschau, 1931, 38, 108—110).—Deacidification of highly acid fats by extraction with EtOH is complicated by the fact that appreciable quantities of neutral fat are dissolved by EtOH in the presence of free fatty acids. Dilution of the EtOH (cf. G.P. 371,813; B., 1923, 666 A) reduces the amount of fatty acid extracted.

E. LEWKOWITSCH.

**Apparatus for experiments on deodorisation of small quantities of fats.** G. DE BELSUNCE (Bull. Mat. Grasses, 1931, 15, 43—47).—The steam and oil temp. and the amount of steam necessary to deodorise the material under vac. or CO<sub>2</sub> can be determined on 100 g. of fat in the apparatus (which is described in detail and by means of diagrams) employed in the Institut Colonial de Marseille; the Wecker effect can be reproduced.

E. LEWKOWITSCH.

**Alkaline- or acid-reacting soaps.** E. I. BETTER (Chem. Umschau, 1931, 38, 110—112).—Methods for the determination of free acid or alkali in soaps are discussed. If considerable amounts of free alkali and of unsaponified fat are present, secondary saponification occurring during dissolution of the soap may vitiate the results of direct titration in EtOH solution; with only small amounts of free alkali atmospheric CO<sub>2</sub> may cause an indeterminate end-point. The occasional occurrence of an acid value in cold-process soaps cannot be explained (? due to the presence of easily saponifiable neutral fat): such a soap after exhaustive extraction with Et<sub>2</sub>O still showed an apparent acid value (0.5) in EtOH solution.

E. LEWKOWITSCH.

**Improved Spitz and Hönig method [of determining unsaponifiable matter in fats].** I. DAVIDSON and E. I. BETTER (Chem.-Ztg., 1931, 55, 363).—The unsaponifiable matter is extracted with light petroleum and the extract twice washed with 50% EtOH

to remove dissolved soap. Very little soap is dissolved in the petroleum in this test and it can be completely removed together with its accompanying water by allowing the solvent to remain in contact with anhyd. Na<sub>2</sub>SO<sub>4</sub>. This procedure is quicker and simpler.

C. IRWIN.

### Determination of lecithin in certain fats and oils.

E. FOYN (J. Pharm. Chim., 1931, [viii], 13, 465—474).—The oil is oxidised by fuming HNO<sub>3</sub> on the water-bath until a homogeneous solution is obtained, when it is evaporated to dryness. The residue is taken up with a little 10% HNO<sub>3</sub>, the mixture cooled on ice and filtered to remove fats, and phosphates are precipitated and weighed as ammonium phosphomolybdate. Tables are given showing the N and P contents of various fats and oils; these are not constant for different samples of the same oil, nor is there any relationship between the two elements, the N being always in excess of that required for lecithin. This confirms the belief that lecithin is present as a lecithalbumin.

T. McLACHLAN.

**Measurement of rate of formation of oxidative decomposition products in fats and oils.** D. P. GRETTE and R. C. NEWTON (Ind. Eng. Chem. [Anal.], 1931, 3, 171—173).—Since rancidity is due to the formation of volatile oxidation products, the fat is oxidised in air under standardised conditions and the distillation products are absorbed in acid KMnO<sub>4</sub> solution, the amount of KMnO<sub>4</sub> reduced being determined by titration with (CO<sub>2</sub>H)<sub>2</sub>.

T. McLACHLAN.

### Preparation and utilisation of sulphur olive oil.

I, II. I. BOHLE (Chem.-Ztg., 1931, 55, 321—322, 342—344; cf. B., 1929, 363).—The production and refining of sulphur olive oil are discussed from technical and economic viewpoints. Residues from modern pressing contain only 8—10% of oil. Oils containing over 6% of free fatty acids cannot be deacidified economically with NaOH; for more acid oils the Wilhelm NH<sub>3</sub>-EtOH process is suitable. The Wecker process is technically effective, but is unsuitable for Spanish installations as the input is seasonal and varies widely in amount and quality. Even when neutralised and refined, sulphur oils are considered to be less stable towards heat and certain reagents.

E. LEWKOWITSCH.

**Future of high-pressure hardening in the oil and margarine industries.** J. A. VAN DIJK, R. T. A. MEES, and H. I. WATERMANN (Chem. Weekblad, 1931, 28, 319—320).—Hardening at 30—60° under high pressure does not destroy the carotene of palm oil or the vitamin-A of cod-liver oil.

H. F. GILLBE.

**Types of Malayan copra.** F. C. COOKE (Malay. Agric. J., 1931, 19, 166—172).—The yields of copra and oil from various commercial grades of nut are detailed. For the best yields naturally-fallen, ripe, and slightly-germinated nuts should be collected. The copra should be graded and dried uniformly at a moderate rate with free ventilation to a moisture content of 6%, and protected from attack by insects during transport and storage (cf. B., 1931, 551).

E. LEWKOWITSCH.

**Manometric determination of the velocity of oxidation of unsaturated oils.** J. JÁNY (Z. angew. Chem., 1931, 44, 348—351).—The Barcroft manometer

is adapted to determine the rate of oxidation of a number of unsaturated oils, as well as the influence of catalysts and other factors on this reaction. As a result, the following oils are placed in order of increasing oxidation velocity: mineral oil, olein, fish oil, rape oil, linseed oil, turpentine oil; owing to the volatility of turpentine oil its exact oxidation velocity could not be determined.

F. R. ENNOS.

Action of detergents on fabrics.—See VI.  $\text{Na}_3\text{PO}_4$ .  
—See VII. Yellowing of rabbits' fat.—See XIX.

#### PATENTS.

Regeneration of used cooking fats. M. BRANDT, Assr. to DARCO CORP. (U.S.P. 1,781,661, 11.11.30. Appl., 25.10.26).—A device suitable for domestic use is described.

E. LEWKOWITSCH.

Treatment of cashew nut-shell liquid. HARVEL CORP., Assces. of M. T. HARVEY (B.P. 300,654, 16.11.28. U.S., 17.11.27).—See U.S.P. 1,725,796; B., 1929, 947.

Oxidation products of oils. Lubricating oil.—See II. Hard-water soaps.—See III. Electric charge on fibres.—See V. Emulsions.—See VI. Resinous condensation products.—See XIII. Rubber-like masses. Treatment of rubber compounds.—See XIV. Tanning agent.—See XV. Insecticide.—See XVI.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oil content of ready-mixed paints. II. WOLFF (Farben-Ztg., 1931, 36, 1425—1426).—Determinations of the viscosity, flow characteristics, etc. of  $\text{ZnO}$ -antimony white-linseed oil mixtures showed that the proportions of the two pigments in the mixtures did not affect the properties thereof additively. An attempt is made to obtain a mathematical expression governing the behaviour of the mixtures (cf. B., 1929, 825).

S. S. WOOLF.

[Improvement of lacquers etc. by addition of] ursolic acid and other plant products. H. A. GARDNER, G. G. SWARD, and A. W. VAN HEUCKEROOTH (Amer. Paint & Varnish Manufs.' Assoc., April, 1931, Circ. 379, 154—160).—Ursolic and oleanolic acid (cf. Sando, A., 1931, 491), both found in the leaves and fruit skins of various plants, appear to offer possible improvements of gloss, water-resistance, and brushing properties of cellulose lacquers and other coatings.

S. S. WOOLF.

Use of "albertols" in printing inks. E. FON-ROBERT (Farben-Ztg., 1931, 36, 1383—1385).—A general discussion is given on the use of copal, amber, rosin, ester gum, hardened rosin, and coumarone resin in printing inks, the drawbacks of each type being indicated. The suitability of albertols, especially 116 Q, for this purpose is suggested and typical formulations are cited.

S. S. WOOLF.

Cellulose acetate.—See III.  $\text{Na}_3\text{PO}_4$ .—See VII. Oxidation of oils.—See XII.

#### PATENTS.

Manufacture of cold-water paint. L. H. LARSON, Assr. to KALBFLEISCH CORP. (U.S.P. 1,781,019, 11.11.30. Appl., 29.1.27).—The paint, comprising substantially

Ca aluminate and Ca caseinate in alkaline aqueous suspension, is prepared by adding 20% of aq. casein suspension (1:1) to a mixture of  $\text{Al}_2(\text{SO}_4)_3$  and excess of hydrated  $\text{CaO}$ . Preservative, e.g.,  $\text{CH}_2\text{O}$ , is added to prevent decomposition of the casein, and pigments may be incorporated.

S. S. WOOLF.

[Diluent for] coating compositions. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,252, 12.9.30. U.S., 27.9.29).—The portion of petroleum naphtha soluble in liquid  $\text{SO}_2$  is used as diluent for paints and lacquers in larger quantities than is permissible when normal petroleum naphtha is employed.

S. S. WOOLF.

Manufacture of white titanium pigments. I. G. FARBENIND. A.-G. (B.P. 346,116, 28.2.30. Ger., 2.3.29).— $\text{TiO}_2$  or a pigment containing it is washed repeatedly with cold  $\text{H}_2\text{O}$ , then with 0.5—5.0%  $\text{H}_2\text{SO}_4$  at 50—60°, and finally with cold  $\text{H}_2\text{O}$ , to remove the Fe compounds.

L. A. COLES.

Composite titanium pigments. TITAN Co. A./S. (B.P. 346,801, 11.12.29. Nor., 12.12.28).—Composite  $\text{TiO}_2$ - $\text{BaSO}_4$  pigments containing 40—60% (preferably 45—55%)  $\text{TiO}_2$  in crypto- and/or micro-cryst. form are prepared by controlled precipitation, the ppt. being washed and calcined in the presence of neutralising agents, e.g., alkaline-earth oxides, and, if desired, sintering agents, e.g.,  $\text{ZnCl}_2$ .

S. S. WOOLF.

Pigments for painting or staining purposes. G. F. THOMPSON (B.P. 345,987, 29.11.29).—Blast-furnace flue dusts, e.g., black or grey from down-draught, brown from under boilers, light grey from hot-air stoves, after extraction and recovery of K salts present, if desired, are used as pigments in conjunction with other colouring materials, e.g., the green oxide obtained from galvanisers' waste acid.

S. S. WOOLF.

Manufacture of resinous products. TOLEDO SCALE MANUFG. CO. (B.P. 345,935, 26.11.29. U.S., 15.5.29).—Urea (<1 mol.) is added to aq.  $\text{CH}_2\text{O}$  (2.25 mols.) the  $p_{\text{H}}$  of which has been adjusted to 6—7 by addition of an organic base, e.g., triethanolamine, and the mixture is heated under reflux until spontaneous boiling ceases, when more urea is added and the product is concentrated to a thick syrup by evaporation in the open. The  $p_{\text{H}}$  is now adjusted to 7—7.5 and thiourea is added in the ratio of 1 mol. to 2 mols. of the remaining uncombined  $\text{CH}_2\text{O}$  (as determined by analysis), and the product is again boiled and concentrated. A "glyptal" resin or other suitable plasticiser is added, and the  $p_{\text{H}}$  adjusted to 3—5 either by this addition, or, if such plasticisers as  $p$ -toluenesulphonethylamide be used, by the incorporation of an organic acid, e.g., phthalic or tartaric, therein. The product is dried *in vacuo* and ground.

S. S. WOOLF.

Preparation of [resinous] condensation products. H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. CO. (U.S.P. 1,779,367 and 1,783,168, [A] 21.10.30, [B] 25.11.30. Appl., [A] 8.10.28, [B] 25.9.30).—(A) The reaction products of polyhydric alcohols, e.g., glycerol or diethylene glycol, with straight-chain aliphatic dibasic acids, e.g., adipic or sebacic acid, are claimed. (B) Products as in (A), but incorporating fatty-oil acids and, if desired, fatty oils themselves, are claimed.

S. S. WOOLF.

[Resinous] moulded articles [e.g., dentures]. H. WADE. From BAKELITE CORP. (B.P. 346,661, 1.1.30).—A fusible resin of the glycerol-polycarboxylic acid type is heated to incipient gelation, and is then moulded and converted into the infusible state under pressure at 140–200°, the heating being continued until the articles are substantially non-plastic at 36°.

S. S. WOOLF.

Shellac composition. R. E. SUMNER, ASST. to CALCO CHEM. CO., INC. (U.S.P. 1,781,711, 18.11.30. Appl., 5.4.28. Renewed 31.12.29).—The composition, suitable for phonograph records etc., consists of shellac (23%), nigrosine or other azine-base dye (2%), cotton flock (4%), and a mineral filler (70–25%).

H. ROYAL-DAWSON.

Manufacture of plasticised products from cellulose acetates. I. G. FARBERIND. A.-G. (B.P. 346,827, 17.1.30. Ger., 26.2.29).—Liquid ethylene oxide or a homologue is used as solvent for cellulose acetates, alone or in conjunction with liquids, e.g.,  $\text{COMe}_2$  or  $\text{EtOH}$ , which when used alone swell the cellulose ester without dissolving it, the alkylene oxide being removed from the solution, if desired, before the product is worked up.

S. S. WOOLF.

Manufacture of solutions, plasticised products, and the like from cellulose ethers. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 346,040, 9.1.30).—The solvent comprises a mixture of  $\text{CCl}_4$  and  $\text{CH}_2\text{Cl}_2$ ; an alcohol, dyes, fillers, plasticisers, etc. may also be added.

L. A. COLES.

Grinder for pigments.—See I. Ketone-formaldehyde products. Pentaerythritol ethers.—See III. Pigments for lacquers.—See IV. Hard paper etc.—See V. Emulsions for sizing.—See VI. Preparing steel for painting.—See X. Sulphur-resin compound.—See XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Röntgenographic investigation of gutta-percha and balata. C. W. STILLWELL and G. L. CLARK (Kautschuk, 1931, 7, 86–89).—The X-ray diagram results with balata and gutta-percha can be explained by the possibility that the latter is a mixture of balata and another crystalline constituent which at the so-called transformation temp. is dispersed so finely in the balata as to yield no interference diagram. The impossibility of a simple reversal of the transformation by cooling supports this view. Some samples of balata also give an interference diagram different from that for gutta-percha (cf. B., 1931, 74).

D. F. TWISS.

Impregnation [of fabric with rubber]. E. A. HAUSER (Kautschuk, 1931, 7, 89–94).—The degree of penetration of rubber into a fabric is examined by cutting sections from a thread or cord embedded in paraffin wax or gelatin, removing the wax and cotton by successive treatment with xylene and  $\text{H}_2\text{SO}_4$  or the gelatin and cotton simultaneously with  $\text{H}_2\text{SO}_4$ , and obtaining a photomicrograph with the aid of "ultropak" illumination. Rubber solutions are found to penetrate the cords far more deeply than latex and thereby to effect better impregnation. Rubber applied by "frictioning" penetrates the cords only during the subsequent

vulcanisation process. Impregnation by latex rubber is not appreciably improved by vulcanisation on account of the unworked nature of the rubber. The degree of penetration with latex can be increased by dilution and addition of dispersing agents or of emulsified benzene.

D. F. TWISS.

Effect of selenium in rubber mixings. H. RIMPEL (Kautschuk, 1931, 7, 94–97).—In the presence of S, Se accelerates vulcanisation and the reduced time of vulcanisation leads, as with organic accelerators and to a comparable extent, to improvement in the resistance to wear. Rubber vulcanised with Se and no S shows no advantage over S-vulcanised rubber in wear-resistance. No justification could be discovered of the claims for marked improvement in abrasion-resistance with the use of Se (cf. B., 1927, 149).

D. F. TWISS.

Combination of organic accelerators for rubber vulcanisation. I. S. MINATOYA, K. KOJIMA, and I. NAGAI (J. Soc. Chem. Ind., Japan, 1931, 34, 138–140 B).—Diphenylguanidine and mercaptobenzthiazole form a molecular compound and two eutectics. The compound has a greater accelerating power than have combinations of the accelerators in other proportions; the eutectics are more active than the components, but are not otherwise remarkable.

D. F. TWISS.

Determination of sulphur in india-rubber products and artificial rubber. M. SAGAJILO, J. BOBINSKA, and A. PIKULSKI (Rocz. Chem., 1931, 11, 289–300).—The comminuted substance (0.5–1.0 g.) is dissolved in 10 c.c. of  $\text{HNO}_3$  ( $d$  1.4), to which 1 g. of  $\text{MgO}$  has been added, and a further 10 c.c. of  $\text{HNO}_3$  ( $d$  1.5) are then added. The solution is boiled for 10–20 min., 15–20 c.c. of 20%  $\text{HClO}_4$  are added, and the mixture is boiled until colourless. The solution is evaporated to dryness, and the residue, after ignition until fumes of N oxides cease to be evolved, is dissolved in  $\text{HCl}$ , and sulphates are precipitated with  $\text{BaCl}_2$ . Free S is determined by extracting the sample with  $\text{COMe}_2$ , evaporating off the  $\text{COMe}_2$ , and heating the residue at 100° with 200 c.c. of  $\text{Br}$  water and 6 c.c. of  $\text{B}_2$  until the solution becomes colourless, when 1 c.c. of  $\text{HCl}$  is added, the solution is filtered off from resinous substances, and sulphate is determined as usual. The residual resins are oxidised with  $\text{HNO}_3$  and  $\text{HClO}_4$  as above, and the result gives the content of S combined with unpolymerised oils or resins.

R. TRUSZKOWSKI.

#### PATENTS.

Coagulation of latex-like emulsions. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 346,785, 17.1.30).—The artificial latex-like liquids obtained by polymerisation of emulsions of butadienes or analogous hydrocarbons are advantageously coagulated by the addition of alkali salts, e.g.,  $\text{NaCl}$  or  $\text{AcONa}$ . Artificial latices obtained from diolefine emulsions, stabilised, e.g., with Na isobutyl naphthalenesulphonate, are not coagulated by acid; even in cases where acid is effective, the acid renders recovery of the emulsifying agent difficult and costly.

D. F. TWISS.

Manufacture of rubber. W. A. BOUGHTON (U.S.P. 1,781,492, 11.11.30. Appl., 28.7.23).—By passing an excess of  $\text{H}_2\text{S}$ , or of the hydride of a higher member of

the S group, into ammoniacal latex and evaporating to dryness, a partly vulcanised product is obtained; vulcanisation can then be completed by heat. A similar result can be obtained by introducing into latex a solution of S, Se, or Te in aq.  $\text{NH}_4\text{HS}$  and evaporating.

D. F. TWISS.

**Manufacture of products having high stability to attrition from rubber and the like.** J. Y. JOHNSON. From I. G. FARRENIND. A.-G. (B.P. 347,108, 16.1.30).—Natural products such as rubber or gutta-percha are treated with oxonium compounds free from aldehydic or phenolic OH groups, such as are obtainable by the addition of Al halide,  $\text{SnCl}_4$ , or  $\text{H}_2\text{SO}_4$  to a halide or anhydride of an appropriate carboxylic acid, *e.g.*,  $(\text{Ac})_2\text{O}$ , phthalyl chloride, or  $\text{COCl}_2$ . This treatment leads to a conversion of the single chains of the mols. into a net-like structure, with development of reduced unsaturation and enhanced resistance to attrition. Even in a highly extended state the new products give little or no indication of a definite X-ray diagram.

D. F. TWISS.

**Production of micro-porous rubber.** H. BECKMANN (B.P. 346,588, 1.5.30. Ger., 3.5.29. Addn. to B.P. 240,430; B., 1925, 1000. Cf. also B.P. 262,179; B., 1927, 119).—Rubber latex is thickened, without coagulation, by the addition of salts with univalent cations, *e.g.*,  $\text{NaCl}$  or Na salicylate, after which it is moulded, converted into a cohesive jelly, and vulcanised. The additional presence of small amounts of soap assists the thickening process.

D. F. TWISS.

**Manufacture of rubber articles [from latex].** ANODE RUBBER CO., LTD., ASSEES. OF C. L. BEAL (B.P. 346,673, 14.1.30. U.S., 15.1.29).—In the coagulation of a layer of rubber on a shaped mould by means of the diffusion of coagulating ions from the surface to be coated or by means of anodic deposition, the danger of formation of streaks by surplus uncoagulated latex adherent to the deposit is obviated by washing this latex from the deposit with an alkaline solution free from coagulating ions and preferably containing a protective colloid, *e.g.*, with a solution of an  $\text{NH}_4$  soap.

D. F. TWISS.

**Manufacture of goods of rubber or similar material [from latex].** DUNLOP RUBBER CO., LTD., and E. W. MADGE (B.P. 346,810, 20.12.29).—Porous or micro-porous rubber articles, *e.g.*, battery diaphragms or filter cones, are produced by converting an aq. dispersion into a gel or continuous coagulum by one or more operations such as electrophoresis, dipping, or moulding, and vulcanising under such conditions that evaporation of the liquid in the pores of the coagulum is prevented.

D. F. TWISS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., and P. D. PATTERSON (B.P. 346,446, 13.1.30).—An artificial aq. emulsion of rubber-like material, particularly reclaimed rubber, which may have been mixed with natural latex in such proportion that the rubber content of the latter is less than half that of the former, is mixed with substantially dry powders (clay or whiting) and then coagulated by adding, *e.g.*,  $\text{PbCl}_2$  while the dispersion is being agitated in a suitable mixer. The powdery product is

suitable for moulding purposes or for compounding rubber.

D. F. TWISS.

**[Applying a nap to] rubber articles.** O. G. BOHLIN (B.P. 346,279, 3.1.30).—The surface of the rubber article which has been rendered temporarily tacky, *e.g.*, by heat or by means of a rubber solvent or solution, is treated with cellulose fibres such as dry disintegrated wood pulp or straw pulp which may be applied in the form of a suspension in air. After ensuring fixation of at least some of the fibres by pressure or rolling, the article is hot-vulcanised. A suede-like nap is thus produced.

D. F. TWISS.

**Sulphur and resin compound [for rubber-compounding].** S. P. MILLER, ASSR. TO BARRETT CO. (U.S.P. 1,782,693, 25.11.30. Appl., 2.12.24).—Paracoumarone resin, m.p. 78–95°, is improved for rubber-compounding purposes by dissolving in the molten resin 10–20% of S. The tendency of the resin to "bloom" is thereby greatly reduced.

D. F. TWISS.

**Rubber derivative and its manufacture.** H. L. FISHER and H. A. WINKELMANN, ASSRS. TO B. F. GOODRICH CO. (U.S.P. 1,782,140, 18.11.30. Appl., 29.12.26).—Rubber is converted into a heat-plastic product by heating, *e.g.*, at 140° for 8 hr., with a dialkyl sulphate, *e.g.*, with 10% of  $\text{Et}_2\text{SO}_4$ .

D. F. TWISS.

**Heat-plastic composition.** H. GRAY, ASSR. TO B. F. GOODRICH CO. (U.S.P. 1,782,036, 18.11.30. Appl., 27.4.27).—If the heat-plastic isomers of rubber having a lower chemical saturation than has rubber are mixed with a small proportion, *e.g.*, 10%, of various substances, such as Pb oleate, tetramethylthiuram disulphide, K stearate, selenium, or wood-flour, the heat-plastic material acquires increased tensile strength and a greater resistance to flow when hot.

D. F. TWISS.

**Preparation of rubber-like masses.** H. A. BRUSON, ASSR. TO RESINOUS PRODUCTS & CHEM. CO. (U.S.P. 1,783,167, 25.11.30. Appl., 8.7.30).—Elastic products are obtained by heating castor oil with an aliphatic dibasic acid ( $\text{C}_6$ – $\text{C}_{10}$ ) and a polyhydric alcohol with more than 2 OH groups, with or without a drying oil. A mixture of glycerin (55 pts.), sebacic acid (181.8 pts.), and castor oil (189.6 pts.) when heated for about 10 hr. at 190–200° gives a rubbery product soluble in BuOH or toluene; if heated for 5 or 6 hr. longer, it becomes insoluble, but is rendered completely soluble on mastication on a rubber mill.

D. F. TWISS.

**Vulcanisation of rubber.** H. O. CHUTE (U.S.P. 1,782,713, 25.11.30. Appl., 29.5.26).—Vulcanisation is accelerated by the reaction products of a cyanamide,  $\text{CH}_2\text{O}$ , and an amine such as aniline.

D. F. TWISS.

**Vulcanisation of rubber.** B. E. MAREAN (B.P. 347,478, 24.4.30).—Rubber in a mould is vulcanised by immersion in a bath of glycerin (flash point not below 195°) heated to a temp. above 132°, but at least 27.8° below the flash point. Rubber tubing or hose, extruded in and with a lead sheath, wound on a drum and filled with water, may be vulcanised by immersing the drum on a bath of electrically-heated glycerin as described.

D. F. TWISS.

**Vulcanisation of indiarubber or the like.** F. W. FARR (B.P. 346,894, 6.2.30. Addn. to B.P. 324,545;

B., 1930, 385).—Individual sheets of thin crêpe rubber are cut into suitable shape and piled loosely in a mould. The pile is then saturated with a vulcanising liquid, *e.g.*, a  $\text{CS}_2$  solution of  $\text{S}_2\text{Cl}_2$ , any surplus liquid is removed, and pressure is applied to compact the mass. For joining two sheets of other material, *e.g.*, wood or leather, these are pressed together after interposing a sheet or sheets of crêpe rubber so treated. D. F. TWISS.

**Manufacture of vulcanised rubber.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 346,853, 23.12.29).—Rubber is vulcanised with the aid of a derivative of mercaptobenzthiazole in which the thiol H atom is replaced by acyl (*e.g.*, Bz or stearyl). Accelerators of this type have a reduced tendency to scorching or premature vulcanisation.

D. F. TWISS.

**Regenerating process for vulcanised rubber.** O. G. BOHLIN (B.P. 346,290, 6.1.30).—In the conversion of vulcanised rubber into a plastic and vulcanisable condition by heat-treatment and/or mechanical working, a small proportion, *e.g.*, 0.5%, of Hg or a Hg compound such as  $\text{Hg}(\text{NO}_3)_2$  mixed into the comminuted rubber, exercises a catalytic effect in accelerating the disaggregation or depolymerisation process. D. F. TWISS.

**Adhesive composition [containing rubber].** W. A. GIBBONS, ASSR. to GEN. RUBBER CO. (U.S.P. 1,779,808, 28.10.30. Appl., 6.3.25).—Surfaces of materials such as leather are treated with an artificial aq. dispersion of rubber and also, possibly simultaneously, with an alkaline material such as  $\text{NH}_3$  solution capable of facilitating penetration of the dispersion. The surfaces are then placed in contact. D. F. TWISS.

**Treatment of rubber compounds [to reduce adhesiveness].** GOODYEAR TIRE & RUBBER CO., ASSEES. of P. A. DAVIS (B.P. 346,489, 27.1.30. U.S., 8.4.29).—Soaps of metals with at. wt. between 24 and 27, *e.g.*, Mg or Al oleate, stearate, or palmitate, are used as dusting compounds to prevent adhesion between unvulcanised rubber surfaces at room temp. D. F. TWISS.

**Aldehyde-amines.** 2-Thiolbenzthiazole. — See III. Waterproofing of sheets.—See VI. Insulating materials.—See XI.

## XV.—LEATHER; GLUE.

**Microscopic technique with special reference to micro-tannology.** F. O'FLAHERTY (J. Amer. Leather Chem. Assoc., 1931, 26, 257–263).—The reagents and methods employed in cutting, staining, and mounting skin sections are described. D. WOODROFFE.

$\text{Na}_3\text{PO}_4$ .—See VII.

## PATENTS.

**Treatment of hides, skins, and pelts.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 346,836, 18.1. and 24.2.30).—The epidermal layer of the hides etc., which have been pretreated with an aq. solution of an alkali, a dil. acid, and/or a neutral water-soluble salt, is removed and the skins are simultaneously bated by treatment in a liquor at 40° and at  $p_{\text{H}}$  6.7–12.0 with the enzymes found in *Carica papaya*, with or without other proteolytic enzymes. D. WOODROFFE.

**Tanning agent.** A. SCHMIDT, ASSR. to I. G. FARBERIND. A.-G. (U.S.P. 1,780,983, 11.11.30. Appl., 4.1.29. Ger., 7.9.26).—Fish oil (100 pts.) or other animal oil or fat is emulsified in 200 pts. of aq. (5% solution) alkylcellulose (*e.g.*, methyl- or dimethyl-cellulose).

D. WOODROFFE.

**Manufacture of titanium tanning preparations.** I. G. FARBERIND. A.-G. (B.P. 346,009, 1.1.30. Ger., 2.1.29).—Solutions containing Ti sulphate are neutralised by the addition of, *e.g.*,  $\text{Na}_2\text{CO}_3$  in quantity such that the solutions contain 0.3–0.5 mol.  $\text{H}_2\text{SO}_4$  per mol.  $\text{TiO}_2$ , and are then concentrated or evaporated to dryness. The product is soluble in  $\text{H}_2\text{O}$ . L. A. COLES.

**Production of [dry] adhesives.** G. MÖRING (B.P. 346,264, 25.10.29).—Glue, casein, or other albuminous substance is hydrolysed by means of alkalis, enzymes, or enzyme mixtures mixed with a hygroscopic substance and, if desired, with fillers and/or rubber latex, applied to bands, plates, rollers, etc. and dried.

D. WOODROFFE.

**Wetting agents. Protection of materials.**—See III. Fibrous sheet. Paper-coating composition. —See V. Vulcanisation of rubber[leather joints]. Adhesive.—See XIV.

## XVI.—AGRICULTURE.

**Nitrogen-fixing bacteria in soils.** M. SELIM (Zentr. Bakt. Par., 1931, 83, II, 311–325).—Comparative determinations of the amounts of N fixed by numerous soil organisms are recorded. The addition of soil and of certain mineral-soil constituents to cultures of the organisms stimulated N fixation. A. G. POLLARD.

**Presence of butyric acid bacteria in silage and their significance.** G. RUSCHMANN and L. HARDER (Zentr. Bakt. Par., 1930, 83, II, 325–349).—The proportion of free and combined butyric acid and of AcOH in various types of silage is recorded, together with the *Amylobacter* content. The number of *Amylobacter* in silage is a criterion of its quality.

A. G. POLLARD.

**Effect of the C : N ratio on the decomposition of organic matter in soil.** I. Effect of nitrogen compounds on the decomposition of carbon compounds. II. Effect of carbon compounds on soluble nitrogen compounds. S. OSUGI, S. YOSHIE, and J. KOMATSUBARA (Mem. Coll. Agric., Kyoto, 1931, [12], 1–39).—I. The decomposition of organic matter in soil, under conditions met with in dry and wet rice fields, is accelerated by the addition of available N [as  $(\text{NH}_4)_2\text{SO}_4$ ], the effect being greater for difficultly decomposable material under unfavourable conditions. The optimum C : N ratio for the decomposition of dextrose and filter paper is about 10 : 1 under dry and 5 : 1 under wet conditions, though it is noted that these ratios change during decomposition due to liberation of  $\text{CO}_2$  and to the microbiological assimilation of C and N compounds. The optimum ratios for rice straw are 30 : 1 and 20 : 1, respectively, these ratios being influenced by the fact that rice straw contains difficultly decomposable C compounds and some available N.

II. Soluble N added to the soil is converted into insoluble forms by the addition of organic material, the

rate being dependent on the amount of the addition. This action is very different under dry and wet conditions, the action in the latter case being slow. N rendered insoluble in this way may not be available to the plant even after 240 days. E. HOLMES.

**Effect of hydrogen peroxide on soil organic matter.** W. McLEAN (J. Agric. Sci., 1931, 21, 251—261).—The oxidation of soil organic matter proceeds in two stages, viz., (a) oxidation of organic matter containing C and N, (b) oxidation of nitrogen-free matter. If 6%  $\text{H}_2\text{O}_2$  be used for determining the degree of humification of soils, (a) is succeeded by (b), which is continued to an extent varying with amount of reagent used and the original C content of the soil. Use of 3%  $\text{H}_2\text{O}_2$  restricts the oxidation to (a). Material involved in (a) has a C:H ratio of 10:1 and is built up of a protein complex in association with a carbohydrate complex of high C content. The unattacked residue from (a) consists of a nitrogenous portion not affected even by 6%  $\text{H}_2\text{O}_2$  and a N-free portion (possibly cellulose) which can be destroyed almost completely by 6%  $\text{H}_2\text{O}_2$ . The "oxidisable matter" (a + b) in mineral, carbonate-free soils includes 85% and 83%, respectively, of the total C and N. In carbonate soils the figures are lower, but increase with repeated treatment with  $\text{H}_2\text{O}_2$ . High percentages of "oxidisable organic matter" are associated with soils of high fertility. A. G. POLLARD.

**"Sticky-point" water of soils. II.** B. DE C. MARCHAND (J. Agric. Sci., 1931, 21, 324—336; cf. B., 1931, 311).—The water content (vol.%, but not wt.%) of soil at the "sticky point" is a rectilinear function of the colloidal content of the soil. Additions of NaCl reduced the sticky-point moisture of soils, the effect being small with solutions more dil. than 0.1N. The reduction in the case of  $\text{Na}_2\text{CO}_3$  was considerably greater. The "loss on ignition" is not a satisfactory indication of the colloidal status of a soil.

A. G. POLLARD.

**Effect of the alternation of layers of different texture on soil-moisture conditions, especially during drying.** J. H. ENGELHARDT (Soil Res., 1931, 2, 204—219).—Apparatus is described for determining the max. and min. negative capillary pressures of moist soils. The formation of "canals" within the soil mass and their influence on the capillary rise of water are examined.

A. G. POLLARD.

**Aggregating effect of frost on soils.** E. JUNG (Kolloidchem. Beih., 1931, 32, 320—373; cf. B., 1931, 454).—Sedimentation and permeability measurements of the degree of aggregation of soil particles after freezing the suspensions in aqueous acetone have shown that the aggregating effect is greater the slower is the rate of cooling. This result is related to the size of the ice particles produced. Cooling by liquid air produces ice crystals of such small size that no aggregating effect is observed. The effect varies also with the amount of water present, suggesting that there is an equilibrium between the adsorption forces of the soil and the crystallisation tendency of the water. An increase in viscosity after freezing was observed only with clays.

E. S. HEDGES.

**Methods for determining the hydrogen-ion concentration of soils.** R. J. BEST (J. Agric. Sci., 1931, 21, 336—365).—The H, quinhydrone, and Sb electrodes are compared in  $p_{\text{H}}$  determinations of numerous Australian soils. The Sb electrode exhibits the same "adaptation lag" as does the quinhydrone electrode, the effect in both cases being more pronounced in soil suspensions than in simple solutions. Higher oxides of Mn are responsible for unsatisfactory results obtained with the quinhydrone electrode for certain basaltic soils. A. G. POLLARD.

**Rapid method for determining the sand content of soils.** L. WOLF, H. SCHLATTER, and W. JUNG (Soil Res., 1931, 2, 220—231).—A centrifugal machine is described in which the coarser soil particles are separated on a series of screens and the finer fractions by centrifugal action.

A. G. POLLARD.

**Seasonal variations in composition of pasture under different manurial treatment.** A. A. HALL and J. HARGRAVE (J.S.C.I., 1931, 50, 167—169 t).—The chemical changes in certain soils from the Northumberland County Council Experiment Farm brought about by thirty years' manuring, and the seasonal variations in the composition of the pasture under varied manurial treatments, are described.

**Nutritive value of pasture. VII. Influence of the intensity of grazing on the yield, composition, and nutritive value of pasture herbage. III.** H. E. WOODMAN, D. B. NORMAN, and M. H. FRENCH (J. Agric. Sci., 1931, 21, 267—324; cf. B., 1931, 83).—Herbage cut at varying intervals is examined. During April and May the composition of the grasses remained the same whether cut at 1-, 2-, 3-, or 4-weekly intervals, significant differences appearing only during the flush period of growth. At this stage there was a higher crude protein content, lower crude fibre, and slightly low N-free extractive content in weekly- as against monthly-cut grass. The intensive use of fertilisers did not affect the composition of the grasses examined during the year of application. The interval of grazing in the early part of the season under intensive treatment may be increased to 4 weeks without loss of digestibility, digestible protein, or of starch equivalent, except under drought conditions when lignification is intensified. Later in the season the digestible protein content of monthly-cut grass falls below that of grass cut at shorter intervals, lowest values being reached in mid-June. Little difference in starch equivalent was, however, observed. Until April monthly-cut herbage had a nutrient ratio corresponding with a conc. nitrogenous food. During May-June the ratio widened, making the ration too bulky for heavy-milking cows (>5 gals.), unless supplemented with concentrates. Composition of herbage during severe drought showed a sharp decline in protein content, slight increases in N-free extractives and crude fibre, considerable increase in Ca, and a corresponding fall in  $\text{P}_2\text{O}_5$  content, nutritive ratio, and digestibility. Effects of fertilisers on the yield and composition of hay and aftermath are recorded and discussed.

A. G. POLLARD.

**Intensive system of grassland management. II. Mineral content of intensively treated pasture**



and a relationship between the nitrogen and phosphorus contents. A. W. GREENHILL and H. J. PAGE. III. Seasonal variation in the mineral content of pasture, with particular reference to drought. W. S. FERGUSON (J. Agric. Sci., 1931, 21, 220—232, 233—240; cf. B., 1931, 82).—I. Analyses of intensively manured pasture cut at 3—5-week intervals are recorded. Variations in the Ca content were considerable, but showed no definite seasonal change. The P content decreased during drought and through the early summer flush with a subsequent recovery in each case, the variations being closely correlated with the N content. The total ash and  $\text{SiO}_2$  contents of the herbage showed less definite seasonal changes, average values being practically the same in all trials examined. A pasture receiving only phosphate had lower total ash and P contents than samples intensively treated, but similar proportions of Ca and  $\text{SiO}_2$ .

II. The mineral composition of pasture herbage was markedly affected by the drought conditions of 1929, the Ca, P, and N contents decreasing, Cl increasing, Na remaining constant. During the subsequent wet autumn and winter the Ca and Cl contents decreased and the  $\text{P}_2\text{O}_5$ , Na, and N contents increased.  $\text{K}_2\text{O}$  variations were irregular, a fairly high level being maintained during the drought period, with a decrease in December. The N content was closely dependent on rainfall.

A. G. POLLARD.

**Fertility of the fundamental soil zones of the U.S.S.R. and their fertiliser requirements.** A. N. LEBEDIANTZEV (Udobr. Urozhai, 1930, 2, 351—363).—Chemical analyses of various types of soil are recorded graphically and in tables. Pot and field experiments on fertility are also recorded. All zones show deficiencies decreasing in the order N, P, K. The podzols and watered desert soils, containing the least amount of K, respond best to fertilisers in general. The southern portion of the chernozem zone shows least response, the northern chernozems being intermediate.

CHEMICAL ABSTRACTS.

**Effect of fertilisers on the phosphorus content of plants.** J. H. MITCHELL (Phosphorus Digest, 1931, Mar., 6—7).—Application of complete fertilisers containing 10—12%  $\text{P}_2\text{O}_5$  gave average increases of 27, 42, and 47% in the  $\text{P}_2\text{O}_5$  content of oats, soya beans, and grass, respectively.

CHEMICAL ABSTRACTS.

**Effect of increasing dressings of phosphates on Riesling and Burgundy vines.** C. DREYSPRING and H. KURTH (Superphosphate, 1931, 4, 93—103).—Superphosphate markedly increased the growth of vines. The thickness of the stems, the number of nodes, and the internodal lengths were all increased in proportion to the amount of superphosphate applied.

A. G. POLLARD.

**Absorption and utilisation of potassium by plants.** R. P. BARTHOLOMEW and G. JANSSEN (Proc. Assoc. Southern Agric. Workers, 31st. Conv., 1930, 242—245).—The min. K concentration for optimal growth is for lucerne and Hubam clover 0.5, for cow peas, oats, soya bean, and cotton 2.0, and for Sudan grass 3.0 p.p.m.; good growth was made at 0.5 p.p.m. Although plants absorb K rapidly, symptoms of K starvation in the later stages of growth are infrequent,

since the element is transferred from older to embryonic regions.

CHEMICAL ABSTRACTS.

**Effects of varied dressings of ground limestone in the field.** II. I. NICHOLSON (J. Agric. Sci., 1931, 21, 262—266).—Results of applications of CaO are compared with lime-requirement determinations (Hutchinson and McLennan). On arable land, equilibrium of soil with added CaO may be attained 1—2 yrs. after the application. The reduction of lime requirement corresponding to a given dressing of CaO is approx. the same for different soils.  $\text{CaCO}_3$  added to the soil is rapidly transformed into exchangeable Ca to an extent depending on the nature of the soil.

A. G. POLLARD.

**Extraction of papain, the active digestive principle from papaya.** H. D. SEN (J. Agric. Sci., 1931, 21, 209—219).—Effects on the yield of papain of varying the number and period of lancements, of manuring, and of varietal differences are recorded. Potash deficiency markedly reduces yields. The effect of phosphate deficiency is less definite.

A. G. POLLARD.

**Constituents of manures and rate of their decomposition in soil.** S. OSUGI and S. YOSHIE (Mem. Coll. Agric., Kyoto, 1931, [12], 41—57).—The composition and behaviour in the soil of the following manures are detailed: soya-bean cake, rape-seed cake, genge (*Astragalus loloides*), saatwicken, rice straw, herring cake, and steamed bone dust. The greater part of the constituents of these manures was decomposed in the soil in 17 days, the ether- and water-soluble constituents being easily decomposed. The amounts of water-soluble residues rendered insoluble were most marked with the vegetable manures. The decomposition of cellulose and hemicellulose was affected appreciably by the amount of N present; if sufficient were present, 77—80% of cellulose and 60—70 or even 90% of hemicellulose were decomposed in 45 days. The maximum amount of organic nitrogenous compounds decomposed was 60%; lignin was scarcely decomposed in any case.

E. HOLMES.

**Preservation of manure under arid climatic conditions.** H. N. WATENPAUGH (New Mexico Agric. Exp. Sta. Bull., 1931, No. 190, 8 pp.).—Min. losses of N and dry matter from manure stacks occur when the wet manure is compacted immediately on storage and as little surface as possible exposed. Under these conditions decomposition is small, but proceeds rapidly after incorporation in the soil. The action of such manure is somewhat slower, but more lasting, than that applied in a more advanced stage of decomposition.

A. G. POLLARD.

**Determination of barium fluosilicate spray residue.** R. H. CARTER (Ind. Eng. Chem. [Anal.], 1931, 3, 146—147).—Figures show that the av. residue varies from 0.031 to 0.144 grain/lb. on apples. This residue is removed and determined by washing first with boiling NaOH solution and then with dil. acid, filtering, and precipitating the Ba. These residues refer to approx. 80% of the total, whereas As is always reported as  $\text{As}_2\text{O}_3$ , which in the case of Pb arsenate is about 30% of the total residue.

T. McLACHLAN.

**Rapid determination of mercury in insecticides.** F. WESSEL and M. KESSLER (Chem.-Ztg., 1931, 55,

318).—The Hg is extracted from the material by shaking it with slightly alkaline KI solution, the filtered solution is shaken with NaOH and  $\text{CH}_2\text{O}$  to precipitate the Hg as metal, and, after acidification with AcOH, the Hg is dissolved in an excess of 0.1N-I solution; the excess I is titrated with  $\text{Na}_2\text{S}_2\text{O}_3$ . A. R. POWELL.

**Cheaper power.**—See I. Calcium phosphate etc.  
—See VII. Cattle excreta.—See XIX.

## PATENTS.

**Manufacture of [nitrogenous and phosphatic] fertilisers.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,974, 18.7.30. U.S., 20.7.29).—Superphosphates (preferably triple) are treated with  $\text{NH}_3$  in two or more stages, preferably with aq. and with liquid anhyd.  $\text{NH}_3$  in successive stages, and the solid product is cooled between each treatment; the percentage of citrate-insoluble  $\text{P}_2\text{O}_5$  in the final product is less than when the treatment is effected in one stage. L. A. COLES.

**Manufacture of phospho-nitro-potassium fertiliser.** L'Air Liquide Soc. Anon. pour l'Étude et l'Exploit. des Proc. G. CLAUDE, Assees. of Soc. Belge de l'Azote Soc. Anon. (B.P. 347,729, 2.1.30. Holl., 3.1.29).—The final mother-liquor of the process is treated with mixture of  $\text{CO}_2$  and oxides of P, and the filtered liquor is charged with a mixture of NaCl and KCl, with more of the gas mixture, and with  $\text{NH}_3$  to produce a mixture of KCl,  $\text{NH}_4\text{Cl}$ , and  $(\text{NH}_4)_3\text{PO}_4$  for use as a fertiliser. Mg compounds, if present, are recovered as Mg phosphates in the fertiliser mixture. L. A. COLES.

**Insecticides.** I. G. FARBERIND. A.-G. (B.P. 343,938, 25.11.29. Ger., 26.11.28).—Aralkyl thiocyanates carrying a thioether group, e.g., 2-methylthiol-5-methylbenzyl thiocyanate, m.p.  $55^\circ$  (from  $\text{NH}_4\text{CNS}$ , and the product from methylated thio-*p*-cresol,  $\text{CH}_2\text{O}$ , and HCl), are dissolved in a water-miscible organic solvent ( $\text{COMe}_2$ ), preferably with addition of wetting agent (alkylnaphthalenesulphonic acid), and diluted for use against green fly; 0.015% is effective. C. HOLLINS.

**Insecticide [for exterminating *Tetranychus telarius*, L.].** A. HARTZELL, Asst. to BOYCE-THOMPSON INST. FOR PLANT RESEARCH, INC. (U.S.P. 1,781,841, 18.11.30. Appl., 9.7.26).—S vapour is condensed in a mixture of oil, fish-oil soap, and water. L. A. COLES.

**Disinfecting and stimulating seeds.** K. WERBA (B.P. 346,014, 27.9.29).—The seeds before sowing are treated with disinfecting gases or vapours, e.g., chromyl halides, halogen-substituted hydrocarbons,  $\text{CH}_2\text{O}$  or its derivatives, which may be liberated by burning cartridges containing substances, e.g.,  $\text{KMnO}_4$ , for regulating the rate of liberation of the gases etc. [Stat. ref.] L. A. COLES.

**Roller pulveriser.**—See I. Paper from plant fibre.  
—See V. Fixation of ammonia-nitrogen.—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Action of sulphides on [sugar-factory] filter-cloth.** O. SPENGLER (Deut. Zuckerind., 1931, 56, 17—18).—Alkaline sulphides are shown to have a very destructive effect on filter-cloth, their presence being traced to the S sublimed from the raw materials of the

lime kiln, which ultimately passes into the press muds of the first carbonatation. In contact with the filter-press cloths, the sulphides are fixed by the cellulose, later oxidising with the liberation of  $\text{H}_2\text{SO}_4$  in the fibre.

J. P. OGILVIE.

**Influence of pre-liming on [beet] juice working and sugar quality.** E. NAEHRING (Deut. Zuckerind., 1930, 55, 1353).—Raw juice in the measuring tanks is pre-limed with 0.15—0.2% CaO, and after being rapidly heated to  $90$ — $95^\circ$  enters a storage tank through an overflow pipe, in front of the first tank of the first carbonatation station, into which milk of lime to the amount of 1.25% CaO is admitted. Two tanks are used at the second carbonatation. In the first 0.05% of CaO is added and the juice carbonatated, while  $\text{SO}_2$  from a bomb is admitted into the over-flow between the first and second tanks. Soda for de-liming is added to the juice in the second tank, the final alkalinity being 0.017—0.012%. Press muds remarkably low in sugar, a superior first-product sugar, and higher yield are among the advantages observed in this method of working. J. P. OGILVIE.

**Digestibility of the protein scums obtained on defecating beet diffusion juice.** V. ŠAZAVSKÝ, K. ŠANDERA, and C. A. RŮŽIČKA (Z. Zuckerind. Czechoslov., 1931, 55, 415—419).—Samples of scums obtained by different methods of coagulating beet diffusion juice in laboratory and factory were found to contain as much as 42% of indigestible N compounds (raw proteins).

J. P. OGILVIE.

**Impurities in white sugars. I. Determination of phosphorus.** S. BYALL and J. A. AMBLER (Ind. Eng. Chem. [Anal.], 1931, 3, 136—137).—Traces of impurities affect caramelisation on heating and fermentation on storage. Lecithin, present in the cane, is hydrolysed to glycerophosphoric acid by CaO, nucleic acid being hardly affected; neither of the latter is removed in the refinery. P, total and inorganic, is determined by Brigg's modification of the Bell-Doisy method (cf. A., 1922, ii, 718). T. McLACHLAN.

**Measurement of colour in solutions of white sugars.** J. A. AMBLER and S. BYALL (Ind. Eng. Chem. [Anal.], 1931, 3, 135—136).—The colour of white sugar is due to occluded and adhering films of the liquors from which it has been crystallised, hence it can be measured usually by comparison with a standard caramel solution. If the colour is abnormal, e.g., grey or green, it can be measured only by use of the spectroscope. There is no relationship between the initial colour of a sugar and that of the candy prepared from it.

T. McLACHLAN.

**Stability of stored starch syrup.** C. LUCKOW (Pharm. Zentr., 1931, 72, 321—322).—Starch syrups can be stored without showing growth of fungi or fermenting provided they are pure, are not diluted with water, and are kept at a low temp. in lac-lined drums. Tinplate may be used for the construction of the drums, but the use of galvanised Fe or wood is excluded, owing to the hygroscopic nature of the syrups.

C. RANKEN.

**Relation between the tenacity of potato flour and the size of the starch grains.** SPROCKHOFF and

WOLFF (Z. Spiritusind., 1931, 54, 101—102).—The results of Parlow and Düll (B., 1931, 217), who attribute a low value of starch to the content of small grains, could not be confirmed. Starch flours are classified into two types. Type A has a lower initial viscosity, and on prolonged heating shows a small viscosity loss; type B has a very high initial viscosity and a high decrease in viscosity on heating. Contrary to the views of Parlow and Düll, type B has a higher value, since its final viscosity is higher than that of type A when treated similarly, and if the starches are fractionated into large, medium, and small grains, the small grains are more valuable than the large, since the fractions containing the small grains show not only a high initial viscosity, but the viscosity remains after prolonged heating higher than that of the large-grained fractions subjected to similar treatment. C. RANKEN.

**Measurement of consistency of starch solutions.** J. C. RIPPERTON (Ind. Eng. Chem. [Anal.], 1931, 3, 152—154).—Starch solutions are prepared under standard conditions and the vol. of the swollen grains is measured directly. Starches differ in the relation between the swell and the viscosities of their solutions, but curves showing viscosity against the product of the concentration and the swell practically coincide for all starches. T. McLACHLAN.

**Identification of the common gums.** M. B. JACOBS and L. JAFFE (Ind. Eng. Chem. [Anal.], 1931, 3, 210—212).—A method is described and tables are given for the identification of gum arabic, tragacanth, agar-agar, karaya, Irish moss, quince seed, locust kernel, Hull emulsifier, and galagum by various group reagents and specific tests. T. McLACHLAN.

**Reaction of sulphites with sugar.**—See V.  $\text{Na}_3\text{PO}_4$ .—See VII. Lævulose in condensed milk.—See XIX.

#### PATENTS.

**Extraction of sucrose from cane molasses.** INTERNAT. INDUSTRIAL & CHEM. CO., LTD., and S. WITTOUCK (B.P. 347,217, 21.1.30).—Cane molasses, diluted with twice its wt. of washings from later stages, is treated at 90° with 1 mol. of  $\text{Ba}(\text{OH})_2$  per mol. of reducing sugar to decompose the latter, and filtered from insol. Ba salts, the washings being used for dilution as above. The filtrate is treated at 90° with 2 mols. of  $\text{Ba}(\text{OH})_2$  per mol. of sucrose and the saccharate ppt. separated, washed with 2%  $\text{Ba}(\text{OH})_2$  solution, and carbonated. The filtrate from the  $\text{BaCO}_3$ , freed from remaining traces of Ba by means of alkali sulphates or carbonates, contains sucrose at a purity of about 98%, and is evaporated as usual. The process is cyclic,  $\text{Ba}(\text{OH})_2$  being regenerated from the carbonatation ppt. and from the soluble Ba salts in the filtrate from the saccharate ppt. About 90% of the sucrose in the molasses is recovered, and the by-products include alkali salts, nitrogenous matter, and C. J. H. LANE.

**Precipitation process [for recovery of sucrose from beet molasses].** R. W. SHAFOR, A. R. NEES, and R. J. BROWN (U.S.P. 1,769,469, 1.7.30. Appl. 19.7.24).—In the two-stage precipitation of Ca saccharate from diluted beet molasses, the hot precipitation is carried out after the filtrate from the cold process has

been diluted 20-fold with mother-liquor from a similar operation. The ppt. obtained is coarser and subsides more rapidly than that obtained without dilution. Suitable apparatus for continuous working is described.

J. H. LANE.

**Treatment of massecuites.** W. W. TRIGGS. FROM INTERNAT. PATENTS DEVELOPMENT CO. (B.P. 339,015, 16.9.29).—Sugar massecuites, in particular those of dextrose, are diluted with mother-liquor from a previous batch and passed to a trough in which is immersed the lower part of a rotary filter-drum. The layer of massecuite drawn against the immersed part of the drum is in the course of 1 rev. successively drained by increased suction as it leaves the trough, moistened by a mist of atomised washing liquor within a casing, again drained, and finally removed by a scraper. The interior of the drum is divided into radial compartments, groups of which are automatically put in communication with different receptacles and subjected to different degrees of vacuum according to their positions in the cycle, by means of ducts in the hub of the drum.

J. H. LANE.

**Saccharification of cellulosic materials.** H. R. FOUQUE, M. MOREAU, and SOC. DES PROC. FOUQUE (B.P. 344,342, 31.10.29. Fr., 6.11.28).—Sawdust, cereal husks, etc. are heated under pressure after removal of air, with "acidulated water" and  $\text{CO}_2$  or gaseous products of combustion or oxidation, and, if necessary, in presence of small quantities of reducing substances. The liquor may be used systematically for successive batches. Apparatus claimed comprises jacketed autoclaves connected with juice reheaters and juice boilers. No particulars of temp., pressures, or acid concentrations are given.

J. H. LANE.

**Sugar dryer.** D. K. RICHARDS (U.S.P. 1,782,177, 18.11.30. Appl. 19.7.29).—Within a fixed conical hopper a central rotary shaft carries slightly dished circular trays one above another. Moist sugar, fed continuously on to the top tray, is thrown centrifugally against the walls of the hopper, and directed on to the next tray by a frustoconical baffle surface fixed to the wall, and so on for successive trays. An upward current of hot air is supplied from a nozzle formed by two conical surfaces co-axial with the shaft near its lower end, from which the air is directed, as a conical sheet, against the circular edge of the lowest tray.

J. H. LANE.

**Wetting agents etc.**—See III. Fibrous material from sugar cane.—See V. Reviving kieselguhr.—See VII. Wall-board.—See IX. Sugar in urine. See XX.

#### XVIII.—FERMENTATION INDUSTRIES.

**Staining of yeast by methylene-blue and its relation to hydrogen-ion concentration and the problem of permeability.** III. H. FINK and F. WEINFURTER (Woch. Brau., 1931, 48, 159—162; cf. B., 1930, 526).—Yeast suspensions were diluted with (a) tap  $\text{H}_2\text{O}$ , (b) 6% dextrose in distilled  $\text{H}_2\text{O}$ , (c) 6% dextrose followed by methylene-blue after  $\frac{1}{2}$  hr., (d) as (c), but with a 0.1M addition of KCl before adding the stain. The stain was at a concentration of 1/15,000 in the diluted suspensions. Though 90% of the cells in dilution (b) were

stainable on addition of methylene-blue, against about 5% in dilutions (a) and (d), all contained the same number of cells capable of growth in plate cultures. On the other hand, the dilution with dextrose and methylene-blue, but with no electrolyte (c), showed 98% of stained cells of which only an occasional cell was capable of growth. The results, which were confirmed by incubation of the main quantity of the dilutions, support the authors' view that absence of electrolyte, especially in presence of sugar, affects the cell membrane, allowing the entry of methylene-blue, which, within the cell, acts as a poison. Such "sensitised" cells, unless treated with a poison such as methylene-blue, are capable of normal growth. F. E. DAY.

**Viscosity of malt extracts and its significance in the working of malt-extract evaporators.** J. WEICHERTZ (Chem.-Ztg., 1931, 55, 341—342; cf. B., 1927, 589).—Curves showing the variation of viscosity with concentration of the extract are plotted; they are smooth curves of the usual type, rising with increasing steepness. The slope, but not the form, of the curve alters with variation of the relative proportions of maltose and dextrin. The results are discussed in relation to heat transference and evaporator efficiency. E. LEWKOWITZCH.

**Carbon dioxide and its rôle in the saturation of beers.** G. CHABOT (Bull. Assoc. anc. Elèves l'Inst. de Gand, July, 1930; Woch. Brau, 1931, 48, 195—198, 206—208).—The adsorption of  $\text{CO}_2$  by the colloids of beer is favoured by prolonged contact under pressure. Head formation and retention is dependent on the presence of a sufficient amount of coagulable colloid of a moderate and uniform degree of dispersion. An excess of  $\text{CO}_2$  may result in too rapid loss when the pressure is released. F. E. DAY.

**Adulteration of sweet wine and its detection by determination of the lower fatty acids (butyric acid).** A. MIERMEISTER and F. BATTAY (Z. Unters. Lebensm., 1931, 61, 161—171).—Carob (St. John's bread) wine used to adulterate currant and raisin wine (cf. Kickton and Berg., B., 1929, 373) may be detected from the odour of isobutyric acid obtained when the distillate from the EtOH-free wine is freed from higher fatty acids and amino-acids by the action of alkaline  $\text{KMnO}_4$ , acidified, redistilled, and the second and third c.c. of distillate are collected. If the test is positive the butyric acid in the wine is determined by the method of Grossfeld and Battay (B., 1931, 652), the wine being distilled with  $\text{H}_3\text{PO}_4$ , and the neutralised distillate heated under reflux with 4 c.c. of 50% KOH before acidification and extraction. The mixed acetic and butyric acids in the final distillate may be titrated to the end-point, when the vol. of 0.1N-KOH  $\times$  0.013856 gives the  $\text{KClO}_4$  equivalent ( $x$ ), and  $k = 100 \times \text{wt. of K salts dried at } 140^\circ$ . Alternatively,  $k$  may be found gravimetrically (*loc. cit.*) and the factor 1.15 corrects for losses due to extraction and distillation etc. The mean mol. wt. of the middle fatty acids may be obtained in a similar way by extraction of the petroleum phase with alkali and distillation with  $\text{H}_3\text{PO}_4$  etc. No butyric nor middle fatty acids were obtained from currants, raisins, and wines known to be prepared from these fruits only, and from 2 out of 13 purchased sweet

dessert wines ("Samos" and "Grecian"); 5 of the latter, however, contained 30—60 mg./litre, and 6, in which carob was suspected, 8—250 mg./litre. Carob wine and Et butyrate give a similar intense luminescence in ultra-violet light, less than 35% of carob in raisin wine gives a doubtful colour, whilst pure raisin wine, butyric and isobutyric acid and its Et ester give feeble colours. The test, therefore, is useful only for sorting purposes (cf. Berg and Stockert, B., 1929, 953). J. GRANT.

**Continuous fermentation in the production of lactic acid.** E. O. WHITTIER and L. A. ROGERS (Ind. Eng. Chem., 1931, 23, 532—534).—Whey is fermented by *Lactobacillus* at  $43^\circ$  with gentle agitation. The reaction is maintained at  $p_{\text{H}}$  5.0—5.8 by the addition of slaked CaO. When the lactose content falls below 1% the continuous addition of whey is started. The stored whey is preserved until added to the fermentation tank by rendering it alkaline with CaO, and it is added at a rate about equal to the vol. of the fermentation tank each 24 hr. As sufficient overflow collects it is boiled for removal of protein, filtered, neutralised with CaO, and evaporated for recovery of Ca lactate. F. E. DAY.

**Acetic acid.**—See III. Dressings for vines.—See XVI.

#### PATENTS.

**[Heating of] brewing pans.** "MIAG" MÜHLENBAU U. IND. A.-G. (B.P. 346,075—6, 1.2.30. Ger., [A] 3.7.29, [B] 8.1.30).—(A) The pan has a central, rotatable steam element to which are attached tube loops curved about it so that the greatest heating surface lies in the neighbourhood of the central region of the pan. The steam inlet of each loop is attached to the central heating element at a higher point than the condensed-steam outlet of the loop, and the direction of rotation of the central element is the same as that of the circulation of the steam in the loops. (B) The series of loops described in (A) is replaced by a single heating tube of three loops or having the shape of a uniform trefoil. C. RANKEN.

**Manufacture of yeast.** H. CLAASSEN (B.P. 346,908, 20.2.30. Ger., 4.3.29).—Yeast prepared by the ordinary aeration method is enriched in zymase by a fermentation below  $27^\circ$  in a solution containing sugar and nutritives. The  $p_{\text{H}}$  of the solution is adjusted to 5.8—6.2, and the quantity of seeded yeast is 3 to 10 times the quantity of sugar, all of which is fermented in 1—2 hr.

C. RANKEN.

**Preparation of nutritive media for yeast.** H. CLAASSEN (B.P. 346,103, 18.2.30).—Comminuted feeding-stuff rich in protein is boiled with a somewhat greater quantity of dil.  $\text{H}_2\text{SO}_4$  than is required for the pre-treatment of molasses. When the protein content of the feeding-stuff has been rendered completely soluble, the mash is added to the molasses solution, and the whole filtered after keeping for several hr. at  $85^\circ$ .

C. RANKEN.

**Production of colouring malt or caramelised malt.** M. WEISENFELD (B.P. 345,951, 30.12.29).—Starting with a roasting temp. of  $40^\circ$ , the temp. of green malt from wheat or rye is raised within  $\frac{1}{2}$  hr. to  $60$ — $70^\circ$ , and, after being maintained at that temp. for 30—40 min., is gradually raised to  $140$ — $160^\circ$  during the next  $\frac{1}{2}$  hr.

C. RANKEN.

**Preparation of an organic substance having a high albumin content.** M. CARLSSON (B.P. 346,765, 16.10.29).—Fish or animal flesh is partly disintegrated mechanically and immersed for 2–4 hr. in  $H_2O$  at not above  $30^\circ$ , to which *Cryptococcus fungi* of the *saccharomyces* type (e.g., *Cryptococcus fermentum*) have been added. The material is then washed, pressed, and dried at approx.  $20^\circ$ . C. RANKEN.

**Denatured alcohol.** W. J. BANNISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,779,687, 28.10.30. Appl., 2.12.27).— $BuCl$  (1–5 vol.-%) and, if desired, brucine sulphate are added to  $EtOH$  for use, e.g., in preparing liniments, perfumes, etc. L. A. COLES.

### XIX.—FOODS.

**Determination of the ash content of bread.** H. KALNING (Chem.-Ztg., 1931, 55, 298).—The presence of  $NaCl$  in bread leads to erroneous ash determinations, the results varying with the conditions of incineration. By moistening the bread (5 g.) with 5 c.c. of 3%  $Mg(OAc)_2$  solution before incineration and determining the  $Cl$  content of the ash so obtained, the ash content of the flour may be found. The results are not entirely satisfactory. As the  $P_2O_5$  content of the ash of white flour is constantly 48%, the ash content of the flour may be readily calc. from the  $P$  content of the dried bread, provided no phosphate has been added in the baking process. W. J. BOYD.

**Determination of the sodium bicarbonate added to milk.** K. SARLÓ (Chem.-Ztg., 1931, 55, 374–375).—The solid residue obtained by evaporating 10 c.c. of milk is carefully ashed, taken up with  $H_2O$ , and treated in the presence of a few g. of  $Zn$  with a few c.c. of 20%  $HCl$  in a special apparatus. The mixture of  $CO_2$  and  $H$  is bubbled slowly through 22 c.c. of saturated  $CaO$  water of known concentration for 30–35 min. The residual  $Ca(OH)_2$  is then titrated with 0.04N- $H_2SO_4$  against methyl-orange. Normal milk ash contains 1.5–2.0%  $CO_2$  and a deduction of 1.8 c.c. from the titer is made in evaluating the amount of added bicarbonate, which is then given by the formula,  $NaHCO_3(mg./litre) = 336 \times (22 - 1.1v - 1.80)$ , where  $v$  is the vol. of  $H_2SO_4$  required. If the factor 1.6 is used instead of 336, the "neutralisation" of the milk in degrees acidity is given. The method is sensitive to within  $1^\circ$  acidity and is suitable for routine-testing. H. J. DOWDEN.

**Determination of lævulose in sweetened condensed milk.** C. L. HINTON and T. MACARA (Analyst, 1931, 56, 286–292).—Lactose (and dextrose) present in the milk serum are oxidised by alkaline  $I$ ; lævulose is then determined by Luff's  $Cu$  solution followed by iodometric titration. Direct reduction does not give accurate results, since most of the lævulose is converted into lævan. T. McLACHLAN.

**Cause of beet odour and taste in milk and butter.** P. POST (Z. Unters. Lebensm., 1931, 61, 171–174).—Addition of  $NMe_3.HCl$  to pure milk (10 mg./100 g.) produces a taste and odour similar to that of the so-called "beet milk" obtained in October and November from cows fed on beet heads and leaves. Distillation of 200 c.c. of such milk with  $CaO$ -water into  $HCl$ , evapora-

tion of the distillate, and extraction with abs.  $EtOH$  yielded a substance giving the reactions of  $NMe_3.HCl$  (e.g., odour with alkali, and ppts. with solutions of  $I$  in  $KI$  and of  $HgKI_3$ ), whilst that from pure milk gave negative results. Possible remedies are discussed, of which warming the milk under reduced pressure and washing the butter with dil. lactic or tartaric acid, followed by  $H_2O$ , yielded promising laboratory results. J. GRANT.

**Determination of water in cheese.** S. H. MEI-HUIZEN (Chem. Weekblad, 1931, 28, 288–290).—Addition of  $EtOH$  in the determination of water in cheese yields low results as a result of adsorption of the  $EtOH$  by the colloidal material present; even at  $135^\circ$  the last traces of  $EtOH$  are not evolved. Cheese cannot be dried to constant weight at  $107^\circ$ , but satisfactory results may be obtained by drying for about 40 min. at  $135^\circ$  and making a correction (about 0.2%) for the slight decomposition which occurs. H. F. GILLBE.

**Composition of hays of "l'Ancien Royaume."** J. VOICU (Bul. Soc. Chim. România, 1930, 12, 173–182).—Analyses are given of 32 samples of Rumanian hay. A. RENFREW.

**Yellowing of the fat in Australian frozen rabbits: its nature and cause.** J. R. VICKERY (J. Coun. Sci. Ind. Res., Australia, 1931, 4, 1–5).—The liver fat of rabbits kept in cold storage for several months undergoes a decomposition characterised by a deep yellow colour and a pungent odour which permeates the surrounding flesh, making it unpalatable. The deterioration is markedly affected by the temp. of storage, the periods taken to acquire a certain degree of yellowness being 2–3 months at  $-5^\circ$ , 4–5 months at  $-10.5^\circ$ , and 9 months at  $-17.8^\circ$ , in the case of animals frozen immediately after slaughter. When storage at room temp. had preceded freezing, the periods were shorter by about 1 month at each temp. The extracted fat is a highly unsaturated "semi-drying" oil (I value 119–179) and contains about 50% of linoleic acid. The concomitant rancidity suggested that oxidation is the cause of the yellowing, and this was confirmed by the observation that by storage in  $N$  at  $-5^\circ$  or  $-10.5^\circ$  the deterioration is completely prevented for more than 1 year. Oxidation of the fat is catalysed by an oxidase present in the tissues, by hemoglobin, and by  $H_2O$ . The preventive measures recommended are: exclusion of air by a new method of folding the carcass, shorter periods between death and freezing, removal of all blood, and storage at not above  $-16^\circ$ . H. J. DOWDEN.

**Apparent sucrose content of certain "honeydew" honeys.** F. E. NOTTBOHM and F. LUCIUS (Z. Unters. Lebensm., 1931, 61, 195–202).—Certain dew honeys (particularly larch honeys) having apparently high sucrose contents (12.7–21.4%) as determined by the official method were found to contain 3–25% of melezitose which, when allowed for, brought the sucrose within the range 1–4% (so-called normal range 5–10%). Such honeys are usually solid in consistency. Melezitose may be separated by precipitation with 4 pts. of 50%  $EtOH$ . The determination of melezitose and sucrose in the presence of one another is based on the fact that both are inverted by acid, but the latter

only is affected by yeast invertase (in 1 hr. at 47–50° for 1 g. of sample). J. GRANT.

**Judgment of foreign honeys according to the honey regulations.** F. E. NOTTBOHM and F. LUCIUS (Z. Unters. Lebensm., 1931, 61, 182–195).—The analytical data and tests of 94 honeys from N. and S. America, W. Indies, Hawaii, Russia, and Ukraine are tabulated. J. GRANT.

**Determination of trigonelline in raw and roasted coffees.** F. E. NOTTBOHM and F. MAYER (Z. Unters. Lebensm., 1931, 61, 202–210).—The ground coffee is extracted with  $\text{CHCl}_3$  (which is preferable to the usual  $\text{CCl}_4$ ), and an extract of the residue in 96% EtOH precipitated with  $\text{Pb}(\text{OAc})_2$ , excess of Pb being removed from the liquid by  $\text{H}_2\text{S}$ . The resulting solution is evaporated twice with HCl and charcoal, the residue extracted with  $\text{H}_2\text{O}$  and a further quantity of charcoal, and the ppt. obtained on addition of acid and 0.1N-I (steel-blue needles from raw, and an oily liquid forming leaflets from roasted, coffee) filtered, dissolved in warm EtOH, and titrated with 0.1N- $\text{Na}_2\text{S}_2\text{O}_3$  (1 c.c. of 0.1N-I = 4.90 mg. of trigonelline). The titrated solution is then shaken with  $\text{Ag}_2\text{O}$ , filtered, the filtrate evaporated with HCl and charcoal, and the residue extracted with hot 96% EtOH. The forms, sources, and properties of trigonelline gold chloride are recorded. J. GRANT.

**Chemical composition and cookery technique of potatoes.** B. von GÖZSY and G. MÉSZÁROS (Z. Unters. Lebensm., 1931, 61, 174–182).—An attempt has been made to correlate the flavour, cooking and keeping properties of 7 varieties of potato with their chemical composition ( $d$  1.0741–1.1161, solids 18.4–28.5%, starch 12.7–21.7%, N 1.7–2.6%). In general, potatoes with least starch (i.e., most N) have the best taste and keeping properties, whether in air or in pure or saline water. No definite relation exists between the starch and N contents, and it is considered that the type of nitrogenous material is more important than its amount. No general relationship exists between chemical composition and fat absorption or suitability for the preparation of various potato dishes, though varieties having the greatest fat absorption cooked the most rapidly and produced the best flavour. Indications are provided of the most suitable and economical uses to which the varieties studied may be put. J. GRANT.

**Ethylene oxide as a new fumigant for dried fruits.** J. E. THOMAS (J. Coun. Sci. Ind. Res., Australia, 1931, 4, 53–54; cf. B., 1929, 1029).—Ethylene oxide is a valuable fumigant against the dried-fruit moth, *Plodia interpunctella* (cf. Roark and Cotton, B., 1930, 634). With experimental packs exposure for 4 hr. at a mean temp. of 20° was sufficient to kill eggs and larvæ. With similar treatment at lower temp. (mean 10°) about 30% of the larvæ were alive after the fumigation, but died within 36 hr.

H. J. DOWDEN.

**Determination of calcium, magnesium, and phosphorus in feeding-stuffs and cattle excreta.** H. P. MORRIS, J. W. NELSON, and L. S. PALMER (Ind. Eng. Chem. [Anal.], 1931, 3, 164–167).—It is impossible to account for all the Ca, Mg, and P in metabolism experiments by extracting the ash with HCl on account

of the formation of complex silicates, which may be broken down by fusion with  $\text{Na}_2\text{CO}_3$ . This point is very noticeable with samples of hay and of cow faeces, the latter of which, on fusion, may give results for Ca, Mg, and P which are, respectively, 3, 9, and  $1\frac{1}{2}$  times as great as by acid extraction. T. McLACHLAN.

**Detection, determination, and occurrence of butyric acid in foodstuffs.** J. GROSSFELD and F. BATTAY (Z. Unters. Lebensm., 1931, 61, 129–161).—One in 12,500 pts. of butyric acid (or, in presence of 0.6% of AcOH, 1 in 10,800) is detectable from its odour by distillation of the aq. solution after removal of other substances by oxidation with alkaline  $\text{KMnO}_4$  (see below). The optimum conditions for the determination of butyric acid in foods have been obtained from the physico-chemical theories underlying the individual processes of the following method:—The sample is distilled with  $\text{H}_3\text{PO}_4$ , the neutralised distillate heated under reflux with 4 c.c. of 50% KOH, and, after acidification and dilution, the mixture is extracted with 25% of light petroleum to remove hexoic and higher fatty acids. The aqueous phase is evaporated with 1 c.c. in excess of N-NaOH, extracted with 10 c.c. of  $\text{H}_2\text{O}$ , and amino-acids and fatty acids other than butyric and acetic are oxidised by means of 20 c.c. of 1%  $\text{KMnO}_4$  for 24 hr., the excess being destroyed by 20 c.c. of a mixture containing 20% of  $\text{FeSO}_4$  and 5 vol.-% of  $\text{H}_2\text{SO}_4$ . Under these conditions distillation (into a weighed flask) gives most of the butyric acid and a little AcOH, which may be titrated to the exact end-point with 0.1N-KOH ( $a$  c.c.), and the  $\text{KClO}_4$  value ( $k$ ) found from the expression  $1.3856a/A$ , where  $A$  is the total weight of the two K salts in the evaporated distillate. Alternatively,  $k$  may be found (more accurately) by determination of the K by the perchlorate method. The percentage of butyric acid in the residue is given by  $2.223(141.23-k)$ , the factor 1.15 being used to correct for losses due to extraction, distillation, etc. Distillation curves for the acids concerned indicate that under the above conditions Walter's "birectifier" is unnecessary for the separation of the acids by distillation. The following values (% butyric acid) were obtained: milk (sweet 0.004, sour 0.008), cheese (Limburger 1.340, Harzer 0.142, rindless Emmenthaler 0.066, Swiss 0.356), meat (beef 0.036, pork 0.010, mutton 0.009), sauerkraut 0.071, pickled cucumber 0.023 (0.022 in the pickle), sultanas and currants 0; and (g. per litre): wine (Malaga 0.110, Muscat 0.117, good Samos 0, poor Samos 0.213, dry red wine 0), beer (light 0.048, dark 0.081), pressed yeast 0.019 (cf. B., 1931, 650). J. GRANT.

**Formaldehyde in foodstuffs.**—See III. **Hardening of margarine etc.**—See XII. **Pasture herbage.**—See XVI. **Protein scums from beet juice.** **Potato flour.**—See XVII.

PATENTS.

**Bleaching agent and process of utilising the same for bleaching flour.** J. R. SHORT MILLING Co. (B.P. 346,408, 1.10.29. U.S., 5.10.28).—Partly germinated soya beans, which have been swollen by soaking in  $\text{H}_2\text{O}$ , are ground and mixed with gelatinised wheat starch. The resulting mixture is dried *in vacuo*

at not above 60°, then finely ground, and added to the unbleached flour either before or during dough-mixing. The bleaching action of the ground soya bean depends on enzymes which react only on the carotene in the presence of warmth and moisture, *i.e.*, during dough-fermentation. Addition of the bleaching mixture (1–2% on the wt. of flour) is sufficient for almost complete decolorisation of the carotene. E. B. HUGHES.

**Obtaining white and purified pectin.** R. PAUL and R. H. GRANDSEIGNE (B.P. 347,961, 28.6.30).—Pectin is precipitated by  $\text{COMe}_2$ , with or without the addition of a mineral acid, in a special rotary jellifier, washed centrifugally, and dried on heated rollers.

E. B. HUGHES.

**Maintenance of the vitamins in the preservation of vegetable materials.** U. BRINCH, H. and C. E. SPEHR (BRINCH & SPEHR) (B.P. 346,574, 12.4.30. Ger., 15.6.29).—A salt and an acid are used simultaneously or successively in such a way that all of the vegetable or fruit comes into contact with them at  $\text{pH}$  2–6. *E.g.*, lucerne (25 lb.) is plasmolysed in 15 min. with  $\frac{1}{2}$  pint of 10%  $\text{H}_3\text{PO}_4$  and 20%  $\text{NaCl}$  and then dried in an air current at 80° for 2 hr., when it is ready for storage and retains to a full extent its fresh appearance, odour, and vitamin content. A method is also given for the treatment of tomatoes to give a dry powder rich in vitamin-C.

E. B. HUGHES.

**Chilling and freezing of fish and other foodstuffs.** D. ROBERTSON (B.P. 348,208, 14.2.30).

**Chilling of beef.** W. W. TRIGGS. From SWIFT & Co. (B.P. 347,668, 31.12.29).

**Preserving meat in ice.** M. ZIZLAVSKY (B.P. 348,443, 30.6.30).

**Roasting apparatus. Drying of cereal grains.**—See I. Sodium glutamate.—See VII. Used cooking fat.—See XII. Nutritive media for yeast. Albuminous substances.—See XVIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Determination of hexamethylenetetramine in certain [pharmaceutical] compounds.** M. J. SCHULTE (Pharm. Weekblad, 1931, 68, 485–488).—For the determination in Helmitol an aq. solution of the sample is exactly neutralised with 0.5*N*-alcoholic  $\text{NaOH}$  and evaporated to dryness; the residue is extracted several times with  $\text{CHCl}_3$  and the hexamethylenetetramine is weighed. About 94.6% is recovered, but a complete determination requires only 1 hr. With Hexal the recovery, after rendering slightly alkaline with the calc. quantity of alkali, is 92.9%, whilst with Saliformine, after neutralising the alcoholic solution, it reaches 98.5%.

H. F. GILLBE.

**Characterisation of drugs. IX. Sinistrin and mucilage in *Bulbus scillae*.** L. ROSENTHALER and R. KOHLI (Apoth. Ztg., 1930, 45, 1097–1099; Chem. Zentr., 1931, i, 1323).—The mucilage gives a red colour with thymol and  $\text{H}_2\text{SO}_4$  and reduces Fehling's solution. After hydrolysis with dil.  $\text{H}_2\text{SO}_4$  it gives a phenylosazone, m.p. 180°. Sinistrin is not attacked by enzymes. When heated with 20%  $\text{HCl}$  on the water-bath it gives a red colour which is deepened by the addition of resorcinol (phloroglucinol, brownish-yellow becoming dirty

red). Heating with orcinol and  $\text{HCl}$  gives a yellowish-red, or with vanillin and  $\text{HCl}$  a raspberry-red colour. Sinistrin is detected microscopically by boiling the section several times with  $\text{EtOH}$ , evaporating the alcohol, treating with  $\text{Sr}(\text{OH})_2$  solution, and immediately covering with a cover-glass. Sinistrin strontium is deposited in the parenchyma cells. A. A. ELDRIDGE.

**Raw tobacco. Nicotine content at various temperatures.** C. PYRIKI and H. DITTMAR (Z. Unters. Lebensm., 1931, 61, 210–217).—Loose, fermented, Grecian tobacco (19 samples) contained 0.75–3.41% of nicotine, Bulgarian (7) 0.63–3.17%, and Turkish (14) 0.90–2.17%, as determined by Pfyl and Schmitt's method (B., 1927, 955). In general, low nicotine contents are distinguished by a thin leaf and light colour, and less nicotine is present in the lower than in the upper leaves and in the portion nearest the stem compared with the tip. The high nicotine content of cigars is due to removal of stalk. Determinations of moisture by distillation with toluene (Gavrilov and Eyslina, A., 1929, 855) showed that tobacco loses nicotine on drying to an extent which increases with increase in original moisture content, and with temp. of drying (50–90°); after 3 hr. at 120° 0.4–1% of moisture remains. After drying for 3 hr. at temp. below 95° variations in nicotine content due to volatilisation are negligible, but low results are obtained above 95°. Other volatile bases were determined by titration of the steam distillate with 0.1*N*-acid, and were thus shown to be retained below 50°. Desiccation in a vacuum over  $\text{CaCl}_2$  produces smaller losses than for 3 hr. at 50°. Disagreements with Petri (B., 1931, 318) for tobaccos of low nicotine content may be explained by the high experimental error of the method in such cases.

J. GRANT.

**Determination of nicotine in tobacco and tobacco smoke.** K. BRAUN (Chem.-Ztg., 1931, 55, 354–355).—A discussion of the work of Pfyl and Schmitt (B., 1927, 955). The use of absorbents for removing nicotine from smoke is generally very unsatisfactory, tests having shown that the most efficient absorbent removes only about 25% of that present in the smoke. A. R. POWELL.

**Reaction of tobacco smoke as an index of the quality of tobacco.** J. O. GABEL and G. I. KIPRIANOV (Ukrain. Chem. J., 1930, 5, [Tech.], 167–184).—Since tobacco smoke is the more acid the higher is the quality, an admixture of low-quality tobacco improves the smoking properties of superior tobacco. The alkalinity of the smoke is unaffected by the moisture content of the tobacco, but increases with the diam. of the cigarette, and with the protein and alkaloid content. It can be reduced by 50% by the addition of glucose.

R. TRUSZKOWSKI.

**Pentosan content of [Russian] tobacco.** J. O. GABEL and L. G. SCHMUKLOVSKAYA (Ukrain. Chem. J., 1930, 5, [Tech.], 185–192).—The ratio of pentosans to soluble carbohydrates is greater for dark-coloured pipe and cigar than for cigarette tobacco. R. TRUSZKOWSKI.

**Volatile constituents of the leaves of *Chamaecyparis Formosensis*, Matsum.** K. KAFUKU and N. ICHIKAWA (Bull. Chem. Soc. Japan, 1931, 6, 94–102).—The leaves on steam-distillation gave 0.16% of oil having  $d_4^{25}$  0.8645,  $n_D^{25}$  1.4724,  $\alpha_D^{25}$  –24.67°, acid



value 1.06, ester value 5.78, and after acetylation 26.35. Constituents identified were *l*- $\alpha$ -pinene, *l*-camphene, dipentene,  $\alpha$ - and  $\gamma$ -terpinene, cincole, borneol (and its esters), cadinene, humulene, a sesquiterpene alcohol which gives cadinene dihydrochloride and cadalene and is possibly identical with cadinol (cf. A., 1915, i, 163), formic, acetic, and (?) citronellic acids, (?) homocatechol, and a ketone, m.p. 139–140°, b.p. 157–161°, isolated by means of its bisulphite compound (cf. B., 1931, 565). A. A. LEVI.

**Opium alkaloids as reagent.**—See III. **Iodine-iodide tinctures.**—See VII.

## PATENTS.

**Water-soluble carbonates from  $\beta\beta\beta$ -tribromoethyl alcohol [narcotics].** J. CALLSEN, ASST. to WINTHROP CHEM. CO., INC. (U.S.P. 1,775,587, 9.9.30. Appl., 15.4.29. Ger., 28.4.28).— $\beta\beta\beta$ -Tribromoethyl chloroformate, from the alcohol and phosgene, is treated with  $\beta$ -dimethylaminoethyl alcohol or similar aminoalcohol to give, e.g.,  $\beta\beta\beta$ -tribromoethyl  $\beta$ -dimethylaminoethyl carbonate (hydrochloride, m.p. 184°).

C. HOLLINS.

**Manufacture of [therapeutically valuable] amidines.** BOOTS PURE DRUG CO., LTD., F. L. PYMAN, and A. P. T. EASSON (B.P. 343,577, 22.11.29).—A hydroxylated aralkyl cyanide is converted by alcoholic HCl into the imino-ether, which by interaction with  $\text{NH}_3$  or an amine gives the amidine. *p*-Hydroxyphenylacetamide (hydrochloride, m.p. 253–254°; nitrate, m.p. 175–176°), obtained from *p*-hydroxybenzyl cyanide by way of the imino-ether (hydrochloride m.p. 153–154°), has a sustained pressor action. C. HOLLINS.

**Purification of sexual hormone-like substances.** SCHERING-KAHLBAUM A.-G. (B.P. 346,130, 14.3.30. Ger., 15.3.29).—The crude extract is dissolved in EtOH, mixed with 10–20 times the total vol. of Et<sub>2</sub>O, and shaken with Al amalgam for 10–20 hr.; the impurities are adsorbed in the ppt., and the purified hormone is recovered by evaporation of the Et<sub>2</sub>O.

E. H. SHARPLES.

**Manufacture of new [organo-]antimony compounds.** CHEM. FABR. VORM. SANDOZ (B.P. 343,898, 23.11.29. Ger., 30.11.28).—Sodium antimonyl gluconate, obtained by dissolving antimony oxhydrate (precipitated from tartar emetic by acid) in aq. Na gluconate and pptd. by EtOH, gives clear aq. solutions, which are stable to neutralisation with alkali in presence of excess Na gluconate. C. HOLLINS.

**Manufacture of organic arsenic and antimony compounds.** I. G. FARBENIND. A.-G. (B.P. 318,491 and Addn. B.P. 344,532, [A] 30.8.29, [B] 18.10.29. Ger., [A] 3.9.28, [B] 21.2.29).—(A) Compounds of the type OH·Alk·CO·NHAr·X, in which Alk is alkylene, Ar is arylene, and X is an arsenic or stibinic acid group, are synthesised. Examples are: 4-nitroanisole-2-arsinic acid, reduced to amine and heated with glycollic acid [product, m.p. 239° (decomp.)]; *m*-aminobenzenestibinic acid with glycollic acid; arsanilic acid with glycollic acid, product reduced with 3-amino-4-hydroxybenzene-1-arsinic acid to give the unsymmetrical arsenobenzene; 4-glycollamido-3-methoxybenzene-1-stibinic acid (from glycollic 5-nitro-*o*-anisidide, reduced) reduced with

3-amino-4-hydroxybenzene-1-arsinic acid. (B) The same products are obtained from aminoacyl or halogenoacyl derivatives by replacement of  $\text{NH}_2$  or halogen by OH. Chloroacetylarsanilic acid is converted directly by hydrolysis, or by way of the aminoacetyl compound by action of  $\text{HNO}_3$ , into glycollyarsanilic acid; 2-chloroacetamidotoluene-5-arsinic acid is aminated and treated with  $\text{HNO}_3$ ; 4-chloroacetamidophenylarsenoxide, 4-chloroacetamidobenzene-1-stibinic acid, 5-chloroacetamidotoluene-2-arsinic acid, m.p. 192–193°, and 5-*o*-bromopropionamidotoluene-2-arsinic acid, m.p. 197–198°, are hydrolysed with NaOH. C. HOLLINS.

**Manufacture of [complex] metal compounds of the thiazole series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,569, 21.11.29).—Carboxylic or phenolic derivatives of amino- or imino-thiazoles are treated with compounds of heavy metals, e.g., Au, Ag, Hg, or Cu, in presence of solvent if desired, to give therapeutically valuable complex compounds which form soluble alkali salts. The Ag, Hg, An, and Cu complexes of  $\omega$ -thiohydantoinacetic acid, Ag complexes of 2-amino-5-methyl-4-thiazolyacetic acid,  $\omega$ -thiohydantoin-*m*-benzoic acid (from *m*-thiocarbamidobenzoic and chloroacetic acids), and  $\alpha$ -( $\omega$ -thiolhydantoin)-propionic acid, and Cu complex of *p*-hydroxyphenyl- $\omega$ -thiolhydantoin, are described. C. HOLLINS.

**Sugar testing [in urine].** A. G. SHEFTEL (U.S.P. 1,769,862, 1.7.30. Appl., 15.8.29).—Prescribed vols. of urine and an alkaline Cu reagent are heated together and matched against a colour chart graded in terms of sugar contents. The  $\text{Cu}_2\text{O}$  is made to separate in a yellow form by addition of gum arabic and creatinine to the reagent. J. H. LANE.

**Dressing material for surgical purposes.** N. CAHN (B.P. 346,497, 31.1.30. Ger., 1.3.29).

**Derivatives of 3 : 4-dihydroisoquinoline.**—See III. **Heating elements.**—See XI. **Dentures.**—See XIII. **Denatured alcohol.**—See XVIII. **Tooth pastes.**—See XXIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Flame studies.**—See II.

## PATENTS.

**Production of printing images on printing plates.** O. STRECKER (U.S.P. 1,777,706, 7.10.30. Appl., 16.2.29. Ger., 8.2.28).—The Zn plate is coated with a film of colloid sensitised with dichromate, exposed to light, and washed with  $\text{H}_2\text{O}$ . After drying, the thin film of colloid adhering to the unexposed part of the plate is removed by treatment with a solution of an alkali salt of a higher fatty acid, with or without the addition of KBr,  $\text{NH}_4\text{NO}_3$ ,  $\text{FeSO}_4$ ,  $\text{Na}_2\text{SO}_4$ , or NaCl. The plate is then rinsed, dried, coated with asphalt, developed with a weak acid, and finished as usual.

A. R. POWELL.

**[Lens system for colour] photography and kinematography.** SOC. FRANÇ. DE CINEMAT. ET DE PHOT. FILMS EN COULEURS KELLER-DORIAN, and I. KITROSER (B.P. 348,465, 19.7.30. Fr., 19.7.29).

**Protection of materials.**—See III.

## XXII.—EXPLOSIVES; MATCHES.

**Recovery of ammonium nitrate from amatol.** Explosion at ammonium nitrate plant of Ammonite Co. R. N. SHREVE (Ind. Eng. Chem., 1931, 23, 566—573).—The  $\text{NH}_4\text{NO}_3$  liquor, obtained by treatment of amatol with steam, was filtered in a Kelly filter press, purified with activated C, filtered through sand, and evaporated under vacuum, the hot liquor, still under vacuum, being passed into vessels in which it crystallised and was granulated. The grains were finally coated in the grainer with a mixture of paraffin, vaseline, and rosin, followed by kieselguhr. An explosion (Mar., 1924), which originated in one of the grainers containing 1800—2000 lb. of  $\text{NH}_4\text{NO}_3$ , involved the further explosion of 16 tons of the finished product in barrels. Although it was presumed that organic matter, *e.g.*, nitrated products of toluene, might have accumulated in a grainer, there was no confirmatory evidence, and the cause of the explosion remains unknown. Certain precautions are recommended.

W. J. WRIGHT.

**Physical tests of trinitrotoluene.** H. ARUNDEL (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 115—117).—Under standard conditions there is little difference in lead-block results for trinitrotoluene (TNT) of setting point between  $75^\circ$  and  $80.5^\circ$ , but the grained material tends to give slightly greater expansion than the flaked. In the lead-plate test, failures occur if the detonator fits tightly in the charge, and are more frequent with trinitrotoluene pressed at 20 than at 10 tons/in.<sup>2</sup> Grained TNT is less sensitive than the flaked, and gives lower results than either flaked or alcohol-crystallised material. For the Hess brisance test, the flaked charges must be pressed at a lower pressure than 0.5 ton/in.<sup>2</sup> to give the required dimensions; the grained requires 1.2 tons/in.<sup>2</sup> Only the ground, flaked material complies with the test. Ground TNT is less sensitive under the fall-hammer (20 kg./200 cm.) than the unground; benzene-purified TNT does not always satisfy the requirements. The bullet test fails to produce detonation with either crude or purified TNT. Compliance with the petard test may be ensured if the distance between the charges is measured from centre to centre, and if the detonators face each other.

W. J. WRIGHT.

**Burning characteristics of smokeless powder.**  
**I. Burning temperature.** A. M. BALL (Ind. Eng. Chem., 1931, 23, 498—501).—A method of calculating the temp. of combustion of a smokeless powder from its heat of combustion and the sp. heats of the explosion gases is shown. The addition of 20% of nitroglycerin to a nitrocellulose powder raises the combustion temp.  $300^\circ$ ; 5.5% of starch lowers it  $200^\circ$ . The combustion temp. of a nitroglycerin-nitrocellulose powder (20:80) is only 10% above that of a nitrocellulose powder of the same N content. This difference diminishes if lower-nitrated cellulose is used in the former powder, and a nitrocellulose powder (13.28% N) has a higher combustion temp. than a 20% nitroglycerin powder (12.0% N). The combustion temp. of nitroglycerin powders is lowered by addition of materials low in O and high in C and H. Temp. for various compositions are tabulated.

W. J. WRIGHT.

**Determination of certain aromatic amino-compounds with special reference to the stabilising ingredients of propellant explosives.** O. C. ELLINGTON and H. G. BEARD (J.S.C.I., 1931, 50, 151—154 r).—The method of Levenson (B., 1930, 930) for the determination of centralite in propellant explosives has been modified and improved as to accuracy and simplicity by the substitution of carbon tetrachloride for alcohol as medium for bromination. The proposed method is applied also to diphenylamine and other aromatic amino-compounds, and it is shown that, in each case, the number of bromine atoms substituted per mol. closely approximates to a simple integer. When applied to an explosive containing diphenylamine the proposed method gives a measure of the content of unchanged diphenylamine, whereas other well-known methods give figures which include also the contents of diphenyl-nitrosoamine.

**Residual carbon from certain explosives.** A. ALBERTO (Ann. Acad. Brasil. Sci., 1930, 2, 187—188).—The resulting high pressure is a dominant factor in the reactions which occur during the explosion of, *e.g.*, nitro-explosives. The formation of free C from explosives such as trinitrophenol which contain an excess of O is ascribed to the reversal, at high pressure, of the reaction  $\text{C} + \text{CO}_2 \rightarrow 2\text{CO}$ .

H. F. GILLBE.

Sulphuric acid.—See VII.

PATENT.

**Production of non-solvent propellant powders.** IMPERIAL CHEM. INDUSTRIES, LTD., O. W. STICKLAND, and E. WHITWORTH (B.P. 347,274, 25.1.30).—The gelatinising agent in such powders consists of a mixture of centralite and phenylurethane, which may either be or not be liquid at room temp., such mixtures being miscible with nitroglycerin at that temp. without separation of crystals on keeping.

W. J. WRIGHT.

## XXIII.—SANITATION; WATER PURIFICATION.

**Utilisation of refuse and refuse fuels.** A. B. SCORER (J. Inst. Fuel, 1931, 4, 287—288).—With forced draught, household, factory, and colliery refuse may be burned to generate steam.

D. K. MOORE.

**Determination of carbon in sewage and industrial wastes.** F. W. MOHLMAN and G. P. EDWARDS (Ind. Eng. Chem. [Anal.], 1931, 3, 119—123).—A modified  $\text{CrO}_3$  digestion is employed for the determination of C, in which the products are passed through acid KI to absorb free Cl and through 0.1N-Ba(OH)<sub>2</sub> for the  $\text{CO}_2$ . The ratio organic C to 5-day O absorption is fairly constant at 0.6—1.0, but there is no relationship between the total C or the N and the O absorbed. The C:N ratio in different wastes varies according to the industry.

T. McLACHLAN.

**Use of sea-water for regeneration of base-exchange materials in water softening.** H. INGLESON and B. ADAMS (J.S.C.I., 1931, 50, 123—124 r).—Preliminary experiments have shown that sea-water may be used either alone or with added salt to regenerate beds of base-exchange materials. The suitability of sea-water for this purpose is compared with that of

solutions of sodium chloride when beds of Kenzelite and of Doucil were used.

**Decrease in mineral matter content of mineral waters containing alkaline earths after removal of iron.** K. WREDE and W. KLING (*Z. angew. Chem.*, 1931, 44, 302—304).—The Fe present in natural mineral waters as  $\text{FeCO}_3$  must be removed before bottling etc., as otherwise cloudiness develops later. This is effected by agitation with air, which causes rapid oxidation and precipitation.  $\text{CO}_2$  is generally added afterwards. Analyses show that this treatment also results in the partial precipitation of  $\text{CaCO}_3$ . The loss is less if  $\text{CO}_2$  treatment follows promptly after the removal of Fe. No precipitation of Mg occurs. If different springs are compared, unexplained differences are found in this loss of Ca, which does not vary directly with the Ca content. The water as marketed may therefore vary considerably in composition from that shown in analyses of the spring water. C. IRWIN.

**Methods attempted for the prevention of phenol and other tastes in the water supply at Rahway, N.J.** M. M. GIBBONS (*J. New England Water Works' Assoc.*, 1931, 45, 6—14).—Medicinal tastes in filtered water drawn from the Rahway river have not always been successfully prevented by any of the methods hitherto tried, which include superchlorination,  $\text{NH}_3\text{-Cl}$ ,  $\text{KMnO}_4$ , and activated C. A reduction in potential taste-producing properties is obtained in the sand-filtration plant, and activated C has proved the most useful agent for post-filtration treatment. Removal of the pollution at its source is indicated as the only way to deal effectively with the trouble in this instance. C. JEPSON.

**Gas production and  $p_{\text{H}}$  determination of *coli-aërogenes* cultures in sugar broths.** C. C. RUCHHOFT, G. H. KALLAS, and B. CHINN (*J. Amer. Water Works' Assoc.*, 1931, 23, 565—577).—Lactose broths buffered with  $\text{K}_2\text{HPO}_4$  have an advantage over unbuffered broths in maintaining a higher final non-lethal  $p_{\text{H}}$  value. An initial adjustment to  $p_{\text{H}}$  7.0 with 0.05—0.2%  $\text{K}_2\text{HPO}_4$  is recommended to give optimum results with brilliant-green as a confirmatory medium and considering the maintenance of a non-lethal  $p_{\text{H}}$  for 48 hr., the gas productivity, and the inhibition of spores. Evidence is produced that the methyl-red and Voges-Proskauer tests are interfered with by the presence of either *B. coli* or *B. aërogenes* as a contaminant of the other. C. JEPSON.

**Interpretation of water analysis.** D. S. MCKINNEY (*Ind. Eng. Chem. [Anal.]*, 1931, 3, 192—197).—The present method of determining the alkaline ions in  $\text{H}_2\text{O}$  cannot account for corrosion. The whole subject must be studied from the physico-chemical consideration of equilibrium and concentration of ions, and for this purpose a complete analysis of the water must be carried out. Tables are presented which reduce the calculations to simple arithmetic, but owing to the present difficulty of determining  $[\text{H}^+]$  at high temp., these cannot be applied fully to boiler work. T. MCLACHLAN.

**MeOH poisoning.**—See I. **Process water in pulp and paper manufacture.**—See V.

# PATENTS.

**Purification of air.** P. O. ROCKWELL (U.S.P. 1,781,664, 11.11.30. Appl., 2.7.26).—Apparatus suitable for respirators is described in which air vitiated with  $\text{CNCl}$ ,  $\text{HCN}$ , etc. is dried and then purified by means of a dry, granular, inorganic, alkaline material, e.g., pumice or activated C impregnated with a caustic alkali and subsequently dried. C. JEPSON.

**Production of acid tooth pastes.** A./S. SI-KO (B.P. 347,377, 25.2.30. Austr., 27.2.29).—To ensure uniformity of the paste, the colloidal grinding of the ingredients is effected at a temp. up to  $70^\circ$ , so that the gel assumes a liquid state and air is prevented from entering. Grinding may be carried out in an inert atm., e.g.,  $\text{CO}_2$ . Any substances added to the  $\text{SiO}_2\text{-H}_2\text{O}$ -glycerin gel must be soluble in  $\text{H}_2\text{O}$  and glycerin so as to maintain the liquid state when heated, but they must not prevent coagulation on cooling. W. J. WRIGHT.

**Water purification.** M. BRANDT, Assr. to DARCO CORP. (U.S.P. 1,781,314, 11.11.30. Appl., 29.3.26).—Water is passed through a bed of activated C in firm coarse grains which is retained between layers of sand in a metal container lined with electrical insulating material to prevent corrosion. Forms of apparatus suitable for small- or large-scale operation are described. C. JEPSON.

**Purification of water.** R. L. HILL, Assr. to DARCO CORP. (U.S.P. 1,782,850, 25.11.30. Appl., 8.5.26).—Apparatus is described in which tastes, odours, etc. may be removed from water by gently agitating it with 0.05—0.2% of powdered activated C and then allowing it to settle in a vessel in which separation of finest particles of C is facilitated by maintaining a rotational movement of 1—2 turns per hr. during the settlement period. The C sludge removed may be re-used, after revivification if necessary. The plant is suitable for either batch or continuous treatment. C. JEPSON.

**Water softening.** I. KRÜGER A./S. (B.P. 347,590, 28.12.29. Denm., 23.5.29).—Precipitation and settling of impurities are accelerated by causing the water, immediately after the addition of precipitating agents ( $\text{CaO}$  and/or  $\text{NaOH}$  or  $\text{Na}_2\text{CO}_3$ ), to flow over contact material (marble, limestone, dolomite, or gravel or granite coated with  $\text{CaCO}_3$ ) before it passes into the settling tanks. L. A. COLES.

**Water-softening apparatus.** J. B. COPLAND (B.P. 346,468, 16.1.30).—The length of the path traversed by water in a zeolite softening plant of the trickling-filter type is increased, with consequent improved efficiency, by introducing baffles, which may consist of a number of overlapping transverse plates or be in the form of a screw fitting closely to the sides of the outer casing. C. JEPSON.

**Respiratory helmets or the like.** H. E. POTTS. From CHEM. FABR. DR. H. STOLTZENBERG (B.P. 348,261, 3.3.30).

**Manufacture of incenses for repelling noxious insects, sterilisation, or medical treatment.** K. ITO (B.P. 347,783, 12.2.30).

**Apparatus for softening water.** C. P. EISENHAUER (B.P. 348,130, 4.1.30).

**Boiler feed-water.**—See I. **Zeolite.**—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 17 and 24, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Measurement of thermal conductivity.** L. JOLY (Compt. rend., 1931, 192, 797—799).—The arrangement consists of three adjoining chambers, 1, 2, and 3, each 2 m. cube. The wall between 1 and 2 is of cement 75 mm. thick, that between 2 and 3 of the material to be tested. Chamber 2 is maintained at a const. higher temp., say 60°, and the temp. in 1 and 3 give approx. the comparative conductivity of the material. To determine the conductivity more accurately, in chamber 2 is placed, with axis horizontal, a cylindrical box, 40 cm. in diam., with double walls and glass wool between and a thermometer each side, open at one end. This open end is placed against the wall of the material under examination. Inside the box is an electric heater. The current necessary to maintain the temp. inside and outside the box the same is a measure of the conductivity across the section of material enclosed by the open end of the box.

C. A. SILBERRAD.

**Diatomaceous earth. I. Structure and properties of diatoms in heat-insulating materials.** W. HUGILL (Trans. Ceram. Soc., 1931, 30, 123—137).—The life history and structure of the various types of diatoms are described in some detail and forthcoming papers on the effect of structure on insulating properties etc. are indicated.

J. A. SUGDEN.

**Construction of acid pumps.** H. WINKELMANN (Chem. Fabr., 1931, 4, 195—196).—A detailed description of two types of pump for corrosive liquids: one type depends on the use of non-corrodible alloys, *e.g.*, Si-Fe or bronze; the other on the use of an inert immiscible liquid, such as oil.

H. F. GILLBE.

**Mechanism of the atomisation of liquids.** R. A. CASTLEMAN, JUN. (Bur. Stand. J. Res., 1931, 6, 369—376).—Theoretical. The mechanism of the air-stream atomisation of liquids is discussed at length, especially in its relation to the internal-combustion engine. Correlation of previous measurements of the size of the droplets with geometrical and physical considerations and with Rayleigh's work on the rate of collapse of liquid columns shows: (i) that the higher the air speed the smaller is the diam. of the ligaments and the shorter their life, and hence the smaller are the droplets formed; and (ii) that, in accordance with observation, at sufficiently high air speeds the droplets will appear to be formed directly from the bulk of the liquid.

H. F. GILLBE.

**Laws of movement of bodies in a fluid.** R. H. KIRKUP (Fuel, 1931, 10, 196—205).—The theory of the motion of spherical and irregularly shaped bodies in a fluid is summarised and applied to the fall of coal

and dirt in air and water. The size classifications of coal suitable for treatment by wet- and pneumatic-separation processes, respectively, are discussed. Whereas a coal sized 1—3 mm. can be treated successfully in a pneumatic separator, the classification 0.5—3 mm. gives unsatisfactory results; the fraction 0.5—1 mm. should therefore be removed and treated separately.

A. B. MANNING.

**Complete combustion devices for furnace tube boilers.** Y. SEKIGUCHI (J. Fuel Soc. Japan, 1931, 10, 29—30).

**Recent advances in the production and measurement of high vacua.** S. DUSHMAN (J. Franklin Inst., 1931, 211, 689—750).

**Portable crusher.**—See II. **Cooling on evaporation of solvents.**—See III. **Sulphuric acid [and gas washing].**—See VII. **Continuous kilns.**—See VIII. **Surface pyrometers.** **Heat-resistant alloys.**—See X. **[Turboviscosimeter for] paints.** **Machinery for lacquers.**—See XIII.

See also A., June, 675, **Dichlorodifluoromethane as refrigerant.** 703, **Colorimeter.** **Thermometer for precision calorimetry.** 704, **Fractional-distillation apparatus for gas analysis.**

### PATENTS.

**Furnaces.** J. SCOGNIO (B.P. 346,872, 25.1.30).—In a furnace the main part of the gases pass over the firebridge and down a passage behind it, then pass on rearwardly through an opening in the bottom of the wall bounding the passage. Two sheets of O-containing gas, *e.g.*, air, impelled by steam jets are blown from the front of the furnace and from the firebridge, respectively, and converge at a point at the top of the above-mentioned passage.

B. M. VENABLES.

**Destructor furnaces.** C. C. ILVING (B.P. 347,573, 24.10.29).—The furnace comprises a shaft in and from which the clinker is supported and removed by a rotating perforated pyramid; the clinker drops to a lower chamber and is there cooled by the air for combustion, being finally discharged by rotating arms.

B. M. VENABLES.

**Heat interchangers for fluids.** V. MICKELSEN, W. M. W. BRUNYATE, and F. E. REBBECK (B.P. 347,904, 17.5.30).—Twisted strips are placed within the tubes of a heat-exchanger, and are rotated either by the flow of fluid along them, or indirectly by means of a propeller in the main pipe and gearing, or by a combination of the two influences.

B. M. VENABLES.

**Improving the thermal economy of adsorption processes performed by the aid of bodies of large**

\* The remainder of this set of Abstracts will appear in next week's issue.

**superficial area and with expulsion of the adsorbed substances by means of scavenging media.** METALLGES. A.-G. (B.P. 348,006, 29.10.30. Ger., 11.12.29).—The heat of the products of the regeneration period is saved in a heat accumulator and used for other purposes. The condensed vapours from a previous regeneration may be used as the heat-accumulating substance. B. M. VENABLES.

**Drying of moist substances.** "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND. (B.P. 347,911, 21.5.30. Ger., 6.12.29).—A rotary drum dryer is provided with both indirect heating by hot gases outside the drum flowing concurrent with the material and with direct heating countercurrent; the latter process is effected in several stages, the hottest gases being introduced some distance up the dryer, *i.e.*, at a point where the material is still very wet. To effect this the lower end of the drum is preferably divided into a number of smaller cylinders grouped round the axis, the axial passage at that end being used to lead the hottest gases up the kiln and extending beyond the small tubes for the material. B. M. VENABLES.

**Liquid for producing low temperatures for cooling purposes.** A. SCHMIDT, ASSR. to I. G. FARBEN-IND. A.-G. (U.S.P. 1,777,183, 30.9.29. Appl., 6.6.29. Ger., 6.7.28).—Mixtures of chlorosulphonic acid (100 pts.) and solid CO<sub>2</sub> (25—100 pts.) are claimed to produce temp. down to -70°. H. ROYAL-DAWSON.

**[Mercury]-vapour generators.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of A. J. NERAD (B.P. 348,004, 15.10.30. U.S., 16.10.29).—In a vapour generator where the amount of liquid must be reduced to the barest minimum, the Field tubes, which are the heat-receiving elements, are connected to as many drums as convenient. Each drum has three concentric walls and two small annular spaces for the vapour and liquid, respectively, which are conveyed by pipes to or from the upper and lower parts of a single drum of usual construction. B. M. VENABLES.

**Construction of workshops such as dye-works etc. for the purpose of dispersing mist caused by the generation of steam.** B. BAUER (B.P. 347,411, 17.3.30. Austr., 20.7.29).—Distribution conduits for preheated humidified air are formed in the roof of the factory between upper and lower glass walls forming skylights; preferably also there is a third, uppermost, glass wall protecting the inner upper one from the atm. Rows of distribution conduits alternate with rows of chimney-like outlet ventilators. B. M. VENABLES.

**Mechanical preparation of pulverised solid materials.** M. MADORE (B.P. 347,914, 23.5.30).—The apparatus comprises a drying circuit, a pulverising circuit, a classifying circuit, and a storage vessel for finely-ground material, *e.g.*, coal. The material is air-borne in the pulveriser and classifier and is transferred elsewhere by worm conveyors. The conveyor which feeds the pulveriser is arranged to run full and to collect, in order: fine coal from storage in excess of that required for consumption, oversize coal from the classifier, and, lastly, new dried coal from another worm-conveyor which also runs full and discharges what it can at this

point, sending the excess back to the dryer and re-filling itself with new undried coal on the way. Thus, once the apparatus is full, the new coal introduced in any period is exactly equal to the consumption of fine coal and the pulveriser and classifier are at all times working with a constant mass of material in circuit.

B. M. VENABLES.

**Feeding solid material to pulverising or like machines.** E. W. GREEN and G. R. UNTHANK (B.P. 347,372, 24.2.30).—The material is fed by a worm attached to a ratchet wheel; the operatingawl oscillates through an arc of constant length, but is held out of engagement during more or less of its stroke by a detent adjusted by any suitable governor. The feeder is driven from the pulveriser through a slipping or frangible safety clutch. B. M. VENABLES.

**Ball or tube mills.** E. G. STONE (B.P. 347,413, 18.3.30).—The mill comprises a number of cylinders parallel to and grouped round the axis of rotation, each one being provided, at one end, with a trap door and spoon-like feeding device operated by tappets when the cylinder is in a suitable position to receive an increment of its charge, and, at the other end, with outlet screens for delivering two sizes of product. The framing of the whole mill may be reciprocated during operation. B. M. VENABLES.

**Multi-stage apparatus for mixing, stirring, emulsifying, etc.** I. G. FARBENIND. A.-G. (B.P. 347,966, 8.7.30. Ger., 13.7.29. Addn. to B.P. 301,701; B., 1929, 268).—In an apparatus on the principle of the prior patent, a method of introducing the gas in a finely-divided state is described. B. M. VENABLES.

**Mixing of materials under vacuum.** MODERN CONCRETE DEVELOPMENT CO., LTD., and A. C. KNIFE (B.P. 347,839, 27.3.30).—In a concrete mixer the discharge door remains latched until a vac. has been produced. B. M. VENABLES.

**Storing and mixing of [semi-fluid] materials.** C. B. THORNE (B.P. 347,628, 23.1.30).—A cylindrical silo is divided into a number of compartments by radial partitions, and materials (*e.g.*, paper pulp) are removed through the bottom of each compartment in succession by a rotating rake which delivers in turn to another rake, a conveyor, an elevator, and a splitting device which cuts out part of the mixed material for delivery and returns the remainder to a distributor and thence to the several compartments of the silo. B. M. VENABLES.

**Separation of intermixed divided materials.** R. PEALE, W. S. DAVIES, and W. S. WALLACE, ASSEES. of R. PEALE (B.P. 347,162, 15.10.29. U.S., 31.8.29).—Material, *e.g.*, coal and shale, which varies greatly in size, but not much in sp. gr., is treated on a reciprocating pneumatic table the upward air currents of which are at first sufficiently intense to drive the smaller particles to the top and are later suddenly reduced, stratification in the second zone taking place according to sp. gr. [Stat. ref.] B. M. VENABLES.

**Filters.** H. S. HELE-SHAW and T. E. BEACHAM (B.P. 347,627, 23.1.30).—The prefilter is contained in a vessel the upper part of which is filled with a gas under pressure; the filter vessel is adjacent and contains a pack filter

with axial outlet for filtrate. When the filter needs cleaning, the pack is removed from its vessel and the pressure gas from the other vessel allowed to enter the axial passage, thus blowing off the cake.

B. M. VENABLES.

**Leaf filters.** E. A. ALLIOTT, and MANLOVE, ALLIOTT & Co., LTD. (B.P. 347,194, 23.1.30).—In a filter comprising a number of leaves in a pressure-tight casing, which when closed forms a reservoir for the prefilter and when opened serves for the discharge of the cakes, the fluid prefilter remaining in the lower part of the casing is drained by filtering through a separate small leaf or by an extension of one or more of the main leaves. Methods of forming the necessary channels and of attaching the leaves are described. B. M. VENABLES.

**Removal of solid particles from liquids.** DORR Co. (B.P. 347,618, 23.12.29. U.S., 24.12.28).—A method of removing sand, in cleaned condition, from, e.g., sewage is described. The total sewage flows through a thickener at such a rate that the organic matter is carried off; the settled sand is raked to the periphery and passes under one wall of the thickener to the lower end of a rake-classifier, by which it is lifted above the liquid level and freed from offensive matter by a water-wash. The classifier and thickener are also in communication at an intermediate level, so that organic matter removed from the sand flows back to the thickener.

B. M. VENABLES.

**Removal of solid particles from liquids.** DORR Co. (B.P. 349,473, 23.12.29. U.S., 24.12.28).—Square thickeners may be used in the process described in the preceding abstract.

B. M. VENABLES.

**Centrifugal separators.** AKTIEBOLAGET SEPARATOR (B.P. 347,993, 26.8.30. Swed., 31.8.29).—A separator in which the supply pipe and receiving vessels are formed in a hinged lid and the delivery spouts in the main body is described.

B. M. VENABLES.

**Continuous-discharge centrifugal machines.** T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 347,985, 11.8.30).—A centrifugal separator of the type having a helical rake to discharge solid matter has the relative motion produced by differential sun-and-planet gearing, the fixed element of which is a sun-pinion attached to a central shaft within the driving shaft.

B. M. VENABLES.

**Washing of gases and vapours.** M. AURIG (B.P. 347,303, 30.1.30).—The gas is passed together with a washing-liquid through the spaces between a number of nested corrugated plates which are spaced far enough apart to prevent the spaces becoming filled with veils of spray, the separation taking place by impingement of the particles of higher sp. gr. upon wetted surfaces. The plates may be assembled in such a way that the passages alternately contract and diverge, producing corresponding compressions and expansions in the gas.

B. M. VENABLES.

**Gas filters for use in gas analysis.** AKTIEBOLAGET CARBA, Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 347,170, 14.12.29. Swed., 17.12.28).—Prior to analysis by diffusion or other method a sample of the gas is filtered through crystals of NaCl or similar material; the saturated solution which forms

on the surface of the crystals causes the gas to acquire a const. moisture content and catches the solid impurities, which are allowed to drain away with it. The NaCl filter may be followed by one composed of wadding or other fibrous material. B. M. VENABLES.

**Apparatus for measuring and testing the physical properties of materials.** [Leather-stretching test.] H. BRADLEY, and BRITISH BOOT, SHOE, AND ALLIED TRADES' RESEARCH ASSOC. (B.P. 347,715, 1.2.30).—A disc of patent leather or other sheet material which is to be stretched until it fails by cracking or otherwise is clamped between annular flanges and a load applied to the centre by means of a ball-headed plunger. The plunger is hollow and telescopic and contains a spring; the end opposite the ball is advanced by a screw, and verniers are provided to measure the advance of the ball end, i.e., the stretch, and the contraction of the plunger itself, i.e., the load applied.

B. M. VENABLES.

**Crystallisers.** W. G. HALL (B.P. 348,950, 8.7.30. U.S., 31.7.29).—See U.S.P. 1,769,779; B., 1931, 183.

**[Suspended] furnace roofs.** H. RÖMER and G. KARRENBURG (B.P. 349,266, 2.5.30).

**Refrigerating apparatus.** METERS, LTD., and C. C. APPLETON (B.P. 349,179, 12.3.30).

**Burning of fuel in furnaces.**—See II. Cement kiln.—See IX. Gas purification.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Classification and development of carbonaceous minerals.** H. BRIGGS (Proc. Roy. Soc. Edin., 1931, 51, 54—63).—The points obtained by plotting the O content against the C content (on the ash-, moisture-, S-, and N-free basis) for a series of coals from lignites to anthracites lie within a narrow continuous band, the median line of which represents the gradual change in rank of the mineral with age. A similar procedure with other types of carbonaceous minerals, e.g., cannel, paraffin shales, torbanites, and petroleums, shows that each type is characterised by a distinct "development line," which may be utilised in classifying such minerals. Each line is directed towards an end-product of definite C:H ratio, e.g.,  $C_{5n}H_{6n}$  for the paraffin shales. The further from the coal line in the direction of the origin of the chart an analysis is situated, i.e., the higher the H proportion, the more suitable is the mineral as a source of oil.

A. B. MANNING.

**Behaviour of solid fuels during oxidation.** IV. **Combustion of the banded ingredients—vitrain, clarain, durain, and fusain.** B. MOORE (Fuel 1931, 10, 205—211; cf. B., 1927, 130).—The four banded ingredients of a coal seam differed considerably in "spontaneous heating tendency," "relative ignition temp.," and "combustible capacity," as determined by the method described previously (B., 1925, 486). No correlation could be observed between these results and the composition of the ingredients as shown by their proximate and ultimate analyses. Coal dusts rich in fusain may be regarded as less liable to undergo spontaneous heating during storage than those resulting from the pulverisation of the whole coal, and should be

suitable, from the ignition temp. and combustible capacity aspects, for use as pulverised fuel.

A. B. MANNING.

**Gray-King high-temperature coal assay.** J. BROWN (J.S.C.I., 1931, 50, 190 T).—The plugging of the tar tube is greatly facilitated by cutting off 95 mm. of the limb containing the plug. This is rejoined to the main tube, after filling, by a short rubber tube.

**Caking, swelling, and expansive pressure of coking coals.** G. LAMBRIS (Brennstoff-Chem., 1931, 12, 181—187).—Determinations of coke yield, caking index (Meurice), swelling power (B., 1929, 270), expansive pressure (Korten-Damm and Koppers; cf. B., 1931, 373), softening point, bitumen extractable by  $C_6H_6$  under pressure, gas evolution (B., 1928, 802; 1929, 666), plasticity (J.S.C.I., 1921, 40, 193 T), and yields of low-temp. tar (Fischer's Al assay apparatus), have been made on 12 coking coals. Apart from a rough parallelism between the swelling powers and expansive pressures of 8 of the coals, no definite correlation could be observed between any of these properties. Of the strongly caking coals some neither swelled nor exerted any expansive pressure on coking; all the swelling coals caked. Although all the coals exerting pressure on coking were both swelling and caking coals, the converse was not necessarily true. On heating the coals in N at 200° the expansive pressures fell to zero within 4—8 hr.; within the same period the swelling powers fell 10—40%, whilst the caking indices either rose or fell slightly and with continued heating the caking indices gradually fell. The bearing of these results on the behaviour of coal in the coke oven is discussed. In general, coals which have been preheated until they no longer exert any expansive pressure also exhibit no "plasticity" when examined by Foxwell's method. Storage of the powdered coals in vessels containing air produced after three months a marked rise (75—90°) in the softening point, but had little effect on the caking index, swelling power, or expansive pressure.

A. B. MANNING.

**Briquetting of brown-coal coke.** F. SEIDENSCHNUR (Brennstoff-Chem., 1931, 12, 191—193).—By the carbonisation of brown coal in a current of inert heating gas at about 380° a coke was obtained containing 1—2% of residual tar and capable of forming satisfactory briquettes. The strength of the briquettes varied both with the tar and the  $H_2O$  content of the coke, the optimum  $H_2O$  content being 12%. A rotary briquetting press working at a pressure of 1800 kg./sq. cm. was used.

A. B. MANNING.

**Determination of the after-coking heat of coke.** G. AGDE and F. SCHIMMEL (Stahl u. Eisen, 1931, 51, 460—462).—The heat required to expel the volatile matter from coke can be calc. by deducting from the total heat of combustion of the coke the heat of combustion of the residue obtained by again coking the material at a higher temp. than that at which it was originally produced and the heat of combustion of the volatile material produced in the second coking. The last-named is calc. from the quantity and composition of the coke before and after re-coking; in these analyses N is determined by combustion of the coke with  $PbCrO_4$  in a current of  $CO_2$ , removal of any O with red-hot Cu

gauze and  $CO_2$  with KOH, and measurement of the N in a nitrometer.

A. R. POWELL.

**Apparatus for the determination of the reactivity of coke.** W. J. MÜLLER and E. JANDL (Brennstoff-Chem., 1931, 12, 187—191; cf. B., 1930, 647).—The following modifications have been made in the apparatus of Agde and Schmitt: a constant-pressure device has been attached to the  $CO_2$  reservoir; a specially designed sensitive stopcock has been provided to regulate the flow of gas; and the two nitrometers have been replaced by a single one, comprising a storage vessel and a measuring tube, and so designed that the flow of gas can be maintained const. throughout the whole experiment. Complete removal of the air from the apparatus is accelerated by evacuating and filling with  $CO_2$  several times before starting the determination. The results obtained by the modified apparatus show considerably less fluctuation than the results of previous determinations.

A. B. MANNING.

**Portable equipment for crushing and quartering samples of coal, coke, or other lumpy materials.** E. S. PERRYJOHN (Ind. Eng. Chem. [Anal.], 1931, 3, 163—164).—Details of construction of a portable steel quartering hopper and a portable steel hand crusher are given. The instruments are designed to facilitate reduction in the wt. and size of lumps of material when working away from the laboratory.

E. S. HEDGES.

**Light creosote from beech wood.** J. GADAMER and G. WESTERBURG (Arch. Pharm., 1931, 269, 307—326).—PhOH, *o*-cresol, guaiacol, creosol, homoveratrole, and ethylguaiacol (*benzoate*, m.p. 65°, and its *nitro*-derivative, m.p. 124°) were identified. Two substances (*semicarbazone*,  $C_8H_{13}ON_3$ , m.p. 230°, and *oxime*,  $C_{16}H_{15}O_2N$ , m.p. 160°, respectively) were also isolated.

A. A. LEVI.

**Oils and schists of S. Romedio.** G. ROBERTI (Annali Chim. Appl., 1931, 21, 116—127).—Investigation of these schists and of the oil they contain shows that the ordinary methods of desulphuration used in petroleum refineries are incapable of removing the S compounds present in the oil. The conversion of the oil into ichthyobenzine, ichthyol, etc. is recommended.

T. H. POPE.

**Separation of *n*-octane from petroleum by distillation and crystallisation.** R. T. LESLIE and S. T. SCHICKTANZ (Bur. Stand. J. Res., 1931, 6, 377—386). A 100—130° fraction from an Oklahoma crude oil contains over 1% of *n*-octane, which is separated by groups of fractional distillations alternating with fractional crystallisations. The *n*-octane isolated is of over 94% purity. Apparatus for the distillations and for the crystallisations at low temp. are described.

G. DISCOMBE.

**Determination of the toluene content of a mid-continent petroleum.** J. H. BRUUN, R. T. LESLIE, and S. T. SCHICKTANZ (Bur. Stand. J. Res., 1931, 6, 363—367).—The distillates from an Oklahoma crude oil of boiling range 70—130° contain 0.3% PhMe. By fractional distillation the PhMe is concentrated in the fraction 98—99°, but the highest PhMe percentage (32%) is found in that of b.p. 107—108°. On nitration of the distillates with a mixture of 10 vols. of  $H_2SO_4$



(*d* 1.84), 2 vols. of oleum (23% SO<sub>3</sub>), and 15 vols. of HNO<sub>3</sub> (*d* 1.50) the PhMe is converted quantitatively into dinitrotoluene (cf. A., 1896, i, 269; 1901, i, 441; B., 1931, 187).

G. DISCOMBE.

**Components of petroleum distillates. II. Aniline-point determination of low-boiling paraffin-naphthene mixtures.** A. SCHAAERSCHMIDT, W. HOFMEIER, and H. LEIST (Z. angew. Chem., 1930, 43, 954—956).—The aniline-point method for determining the naphthene content of binary mixtures of methylcyclohexane, cyclohexane, *n*-hexane, and *n*-heptane, may be employed, using the formula  $N = 100(A - A_m)/(A - a)$ , where *N* is vol.-% naphthene, *A* and *a* are the aniline points of the components (*A* being < *a*), and *A<sub>m</sub>* is the aniline point of the mixture. The accuracy of the method is about ± 1%, but falls rapidly with falling b.p. of the components. The method may also be applied to ternary and quaternary mixtures of these hydrocarbons.

E. S. HEDGES.

**Detonation characteristics of some aliphatic olefine hydrocarbons.** W. G. LOVELL, J. M. CAMPBELL, and T. A. BOYD (Ind. Eng. Chem., 1931, 23, 555—558).—The knocking tendencies of 25 unsaturated hydrocarbons in admixture with petrol have been compared with that of PhNH<sub>2</sub>, employing a bouncing-pin method. The compounds chosen were of the straight- and branched-chain types with one and with two double linkings. Very great differences in knocking tendency were observed even among isomerides, the position of the double linking having considerable influence. With straight-chain olefines the tendency to knock rose in an homologous series with the length of the saturated chain. In an isomeric series the tendency decreased progressively with centralisation of the double linking. The tendency to knock is roughly determined by the length of the longest saturated C chain. With branched-chain olefines the tendency to knock decreases with the introduction of a double linking, and it is related both to the position of this linking and to the branched structure of the mol.

H. INGLESON.

**Chemical nature of Ural benzine and its catalytic aromatisation.** N. D. ZELINSKI and J. K. JURIEV (Bull. Acad. Sci. U.S.S.R., 1930, 851—861).—Ural benzine, a works' distillate which had been treated with H<sub>2</sub>SO<sub>4</sub>, contained no thiophen, but a high proportion of compounds of mercaptan and sulphide type. These were removed by treatment with M'OH, prolonged shaking with HgCl<sub>2</sub>, filtration, and fractional distillation over molecular Cu recently reduced by means of H. The aromatic hydrocarbons were determined by measuring the diminution in vol. when shaken with oleum (10% SO<sub>3</sub> for the lower fractions, 7% SO<sub>3</sub> for the higher ones); the proportion of total aromatic hydrocarbons was 33%. Similar determinations were made of the aromatic hydrocarbons in the various fractions after these had been subjected to catalytic dehydrogenation by passing their vapours over platinised C at 300—310°. Calc. on the original benzine, the total proportion of aromatic hydrocarbons thus formed amounts to 16.75%. Hence this benzine is capable of yielding 50% of aromatic hydrocarbons, the proportions—

for some of its fractions being considerably higher. Analysis of the residue indicates the absence of any further cyclic compounds, such as pentamethylene hydrocarbons. Surachani benzine contains no aromatic hydrocarbons, but yields higher proportions than the Ural product after catalytic dehydrogenation.

T. H. POPE.

**Production of petroleum hydrocarbons from carbon monoxide and hydrogen at the ordinary pressure.** E. R. MÜLLER [with S. KALLENBERG] (Ingen. Vetensk. Akad. Handl., 1930, No. 106, 39 pp.; Chem. Zentr., 1931, i, 1205).—A description of the application of Fischer and Tropsch's process. 245 g. of pine charcoal (88.17% C) gave 20 g. of gas oil (not including C<sub>2</sub>H<sub>6</sub> and C<sub>2</sub>H<sub>4</sub>), 26 g. of benzine and 14 g. of petroleum hydrocarbons, and 15 g. of vaseline.

A. A. ELDRIDGE.

**Determination of sulphur in liquid fuels. I.** F. GARELLI and B. SALADINI (Annali Chim. Appl., 1931, 21, 136—140).—The procedure recommended is as follows: 10 c.c. of H<sub>2</sub>O are pipetted into the calorimetric bomb, the Pt or quartz crucible containing the igniting Fe wire coil being charged with a few crystals of NH<sub>4</sub>NO<sub>3</sub> and with a definite wt. of the liquid fuel from a Sprengel pyknometer. The bomb is at once closed, the compressed O<sub>2</sub> introduced, and, after the bomb has been placed in the calorimeter, the fuel ignited. After 1 hr. the gas is allowed to escape from the bomb and the H<sub>2</sub>SO<sub>4</sub> formed determined as usual.

T. H. POPE.

**Oxidation of paraffin oil by air.** G. ARDITTI (Compt. rend., 1931, 192, 944—946).—Air was bubbled for varying periods and at varying temp. through a paraffin oil free from unsaturated compounds and from S. Oxidation did not occur in 1500 hr. at temp. up to 85°, but was apparent after 12, 3, and ½ hr. at 110°, 123°, and 150°, respectively, and markedly at room temp. under ultra-violet light. The free acidity, total acidity, and viscosity for various periods up to 82 hr. at 110°, 123°, and 150° vary substantially similarly.

C. A. SILBERRAD.

**Use of lead tetraethyl in internal-combustion motors.** P. DIDIER (Chim. et Ind., 1931, 25, 803—810).—EtOH and aromatic hydrocarbons act as diluents in increasing the possible compression ratio. The efficacy of EtOH is due to its high latent heat of vaporisation. The importance of PbEt<sub>4</sub> to France in case of war is stressed. A French Government committee, on the strength of the British committee's interim report, has advised that "ethyl" containing 0.08% PbEt<sub>4</sub> is unsuitable for use in towns.

C. IRWIN.

**Stabilisation of [lubricating] oils for chronometers.** P. WOOG, (MLLE.) E. GANSTER, and J. GIVAUDON (Compt. rend., 1931, 192, 923—925; cf. B., 1926, 37).—The possible stabilising effect of various "antioxygens" on sheep's-foot oil (as a typical triglyceride lubricating oil) has been examined. Addition of, e.g., 0.5% of β-naphthol causes a marked decrease in the rate at which acidity is produced in the air, provided the oil is in darkness, but in light an increase. The effect of darkness can, however, be obtained by adding a red dye, e.g., Soudan I, II, or III, Scarlet 2R, etc. Brass on which a drop of oil thus stabilised has been

placed shows no sign of attack by the time it has been blackened by the untreated oil. C. A. SILBERRAD.

**Movement of bodies in a fluid.**—See I.  $\text{H}_2\text{SO}_4$  [and gas washing]. **Absorption of  $\text{H}_2\text{S}$ .**—See VII. **Resins from low-temp. tar.**—See XIII.

See also A., June, 677, Adsorption of H on charcoal. 678, Sorption of H by Pt metals. 689, Explosions of  $\text{C}_2\text{H}_2$ —electrolytic gas mixtures. Flames and their propagation. 690, Behaviour of antiknocks. 704, Gas analysis by fractional distillation. 705, Slow-combustion pipette. 708, Japanese black shale. Origin of Japanese petroleum. 751, Organic Pb compounds.

## PATENTS.

**Pneumatic coal-cleaning plants.** A. F. BURGESS. From ROBERTS & SCHAEFER Co. (B.P. 347,578, 26.11.29).—A number of pneumatic shaking tables are arranged in series in such a way that dusty air from one table or set is roughly cleaned in a cyclone and supplied to another table or set. B. M. VENABLES.

**Burning of fuel in furnaces.** STOCKHOLMS AKTIEBOLAGET PRIVAT (B.P. 347,174, 20.12.29. Ger., 17.1.29).—Combustion is facilitated by blowing incandescent particles of fuel, together with hot gases and a part of the air for combustion, at high velocity from below into the layer of fuel on the front portion of the grate. The incandescent fuel particles may be obtained by sucking off gases laden therewith from a suitable part of the fire space or smoke channels; in addition, particles falling through the grate or other coke dust may be mixed with these gases. Furnaces for carrying out the process are described; they embody also means for preheating the air for combustion and for predrying the fuel. A. B. MANNING.

**Heating of coal or the like.** F. PUENING, Assr. to KOPFER Co. (U.S.P. 1,781,079, 11.11.30. Appl., 3.9.27).—Coal for coking purposes is dried and preheated at 100—300° in externally heated vertical conduits. The elevator conveying the raw coal to the top of the preheater chamber also conveys the heated coal to the top of the hot coal bin, the arrangement being such that one end of each elevator bucket is charged with raw coal and the other end with heated coal, whereby part of the sensible heat of the latter is utilised in evaporating the moisture from the raw coal. A. B. MANNING.

**Combustion and gasification of carbonaceous materials.** F. C. GREENE (B.P. 347,575, 25.10.29).—The fuel is progressively advanced through zones wherein its temp. is progressively raised until it reaches the hot exposed surface of the mass, over which air is passed. The air current is so adjusted that the  $\text{CO}_2$  first produced by combustion of the fuel is reduced to CO in passing over the incandescent material. In one form of apparatus for carrying out the process the fuel is supported on a frusto-conical surface carrying laterally extending spiral platforms for effecting the required movement of the fuel, over the surface of which the air passes in a downward direction. Provision may be made for separately withdrawing the light distillates evolved in the zones of intermediate temp. A. B. MANNING.

**Distillation of solid carbonaceous material.** H. L. DOHERTY (U.S.P. 1,781,871, 18.11.30. Appl., 8.5.25).—The material is distilled in an upward current of hot inert gases, which, together with the volatile products of distillation, are withdrawn through a number of gas-offtakes distributed over the cross-section of the retort in such a manner as to ensure a uniform distribution of the heating gases in the charge of fuel, and so avoid "channeling." The fuel is charged into the retort through a corresponding number of separately controlled charging conduits. A. B. MANNING.

**Cooling of coke and obtaining a nitrogen-hydrogen mixture.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,601, 28.1.30).—The hot coke is charged into a large cooling chamber into which water is sprayed at a depth where the coke has cooled to about 550°, while air is injected at a higher level, where the temp. is about 900°. The air supply is so regulated that after the catalytic conversion of the CO produced into its equivalent of  $\text{H}_2$ , and after purification, the gas mixture formed consists of  $\text{N}_2$  and  $\text{H}_2$  in the stoichiometric ratio of 1:3. A. B. MANNING.

**Production of water-gas.** S. J. McAULIFFE, Assr. to FULLER LEHIGH Co. (U.S.P. 1,780,759, 4.11.30. Appl., 21.9.21).—Pulverised coal and steam are injected horizontally into a narrow vertical retort having side walls of relatively large area. These walls are heated to 1500—1600° by flues communicating with a combustion chamber disposed below the retort and designed to burn pulverised fuel. A. B. MANNING.

**Water-gas generators.** A. BREISIG (B.P. 347,520, 4.6.30. Austr., 5.6.29).—In the distillation and gasification of bituminous fuel by means of alternately working water-gas generators, the hydrocarbon vapours generated in the distillation zone during the blast period in the gasification zone are condensed in a zone of cold fuel surmounting the distillation zone. The mixed water-gas and distillation gases are preferably withdrawn through a duct which extends to the centre of the fuel column at the top of the distillation zone. A. B. MANNING.

**Desulphurisation of gas.** F. W. WERNER and E. T. JOHNSTON (U.S.P. 1,779,024, 21.10.30. Appl., 16.9.26).—The gas is treated with CaO at 550°, preferably in a rotating drum heated externally by flue gases. The S compounds are decomposed with the formation of CaS and the liberation of  $\text{H}_2$ , which, in the presence of the CaO, interacts with the  $\text{N}_2$  in the gas to produce  $\text{NH}_3$ . A. B. MANNING.

**Production of acetylene.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,493, 8.5.30).—Gaseous hydrocarbons are submitted to incomplete combustion, preferably at 700—1100°, under pressures of 0.5—5 atm., in the presence of a catalyst containing free Si. A. B. MANNING.

**Apparatus for generating acetylene.** I. G. FARBENIND. A.-G. (B.P. 347,537, 28.6.30. Ger., 1.8.29).—The carbide receptacle is flared downwardly and its lower part, which projects into the generating chamber, is designed as a cage the carbide in which is sprayed with  $\text{H}_2\text{O}$  in considerable excess. The spraying  $\text{H}_2\text{O}$  is circulated by a pump which draws it, roughly freed

from CaO sludge, from the generating chamber, and delivers it to the spraying device. A. B. MANNING.

**Distillation of (A, B) tar and (B) like hydrocarbons.** BARRETT Co., Asscs. of S. P. MILLER (B.P. 347,107 and 347,240, [A] 16.1.30, [B] 18.12.29. U.S., [A] 16.1.29, [B] 18.12.28).—(A) Tar is brought into intimate contact with hot distillation gases, *e.g.*, coke-oven gases, in a non-externally heated still, under such conditions that a high-m.p. pitch is produced. The enriched gases leaving the still are passed through a condenser wherein they are brought into indirect contact with the incoming tar, which is thereby preheated sufficiently to distil off part of the light oils therein. (B) Intimate contact between the hot gases and the tar is brought about by providing horizontal rollers which dip into a body of tar maintained at the bottom of the still, and which are rotated at a relatively high speed, and thereby maintain a fine spray within the still. A. B. MANNING.

**Rotary retorts for treatment of oil shale and the like.** T. M. DAVIDSON (B.P. 347,121 and 347,142, 17.10.29).—A rotary retort is provided with a longitudinal vapour-outlet pipe situated in that part of the cross-section of the retort which is remote from the part where the material piles up during the rotation; the outlet pipe is non-circular in cross-section, and serves also as a support for reciprocating scrapers operating upon the interior of the pipe and of the rotating retort. Forms of gastight joints between the rotating and fixed parts are described. B. M. VENABLES.

**Treatment of shale or the like.** W. H. HAMPTON (U.S.P. 1,778,515, 14.10.30. Appl., 16.12.20).—Ground shale is digested with heavy oil at 260–370°, and the solid matter is then separated by means of a centrifuge. Vapours evolved during the digestion are separately condensed. The solid is finally destructively distilled to recover additional bituminous matter. A higher yield of better-quality oil is obtained by this treatment than by direct destructive distillation. T. A. SMITH.

**Cracking of [hydrocarbon] oils.** E. C. HERTHEL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,781,390, 11.11.30. Appl., 7.1.25).—Cracking stock is pumped from a bulk-supply tank upwardly through a vertically arranged tubular heater and back to the tank, the oil being maintained at cracking temp. throughout the cycle. The vapours escaping from the tank are refluxed in direct contact with fresh charging stock, and the reflux and admixed fresh oil are together forced upwardly into the oil circulating through the lower part of the heater to assist circulation.

H. S. GARLICK.

**Crude oil and natural gas separator.** M. H. KOTZEBUE, Assr. to TRUMBLE GAS TRAP Co. (U.S.P. 1,782,783, 25.11.30. Appl., 22.9.24).—A vertical vessel is fitted with a float-controlled valve by means of which a supply of oil is retained in the lower portion of the separator. A gas offtake extends down the separator and is surrounded by a spiral pathway on to which the oil and gas from the well are delivered. Good separation of oil from gas takes place as a result of the oil flowing in thin films down the pathway. An inclined screen is interposed in the gas offtake to assist in the removal of entrained oil. T. A. SMITH.

**Conversion of hydrocarbons into those of lower b.p.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,727, 2.1.30).—The hydrocarbons are treated with free halogen, preferably Cl, and the products obtained, admixed, if desired, with a further quantity of the initial material and/or with at least part of the H halide formed during the first stage of the process, are subjected to the action of activated Al at 150–250°.

A. B. MANNING.

**Conversion of high-boiling hydrocarbon oils.** E. E. BARTELS, Assr. to STANDARD OIL Co. (U.S.P. 1,782,160, 18.11.30. Appl., 12.7.26).—Oil heated to the inversion temp. under a pressure of 4–5 atm. is passed through a confined passage at a rate sufficient to prevent the deposition of coke. The vapour passes through a dephlegmator countercurrent to a stream of condensate and is then condensed. Part of the condensate is returned to the dephlegmator and the vapours leaving the condenser serve to preheat the fresh oil.

J. A. SUGDEN.

**Cracking of hydrocarbon oils.** R. J. DEARBORN, Assr. to TEXAS Co. (U.S.P. 1,783,010, 25.11.30. Appl., 19.1.28).—Oil is passed through a heating coil of restricted cross-section, where it is raised to substantially cracking temp., and delivered into an enlarged cracking drum. Further oil is simultaneously cracked in the vapour phase at a relatively high temp. and the cracked products are passed in direct contact with the oil in the cracking drum, thereby maintaining its temp. and effecting liquid-phase cracking. H. S. GARLICK.

**Cracking of hydrocarbon oil.** J. G. HAWTHORNE, Assr. to W. M. CROSS (U.S.P. 1,782,686, 25.11.30. Appl., 9.7.27).—Oil is raised to a conversion temp. in a primary heating stage and the vaporised products are reheated and superheated by passage through conduits in a reheating stage, and then cooled to a predetermined temp. selected to effect separation of the heavier and lighter fractions. The heavier fractions are discharged from the system as liquid, whilst the lighter are refluxed and condensed, the reflux condensate being recycled to the primary heating stage. A balanced pressure is maintained between the material undergoing superheating and the combustion gases, the heat of which is used to heat in turn the primary heating stage.

H. S. GARLICK.

**Cracking and processing of petroleum hydrocarbons.** J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,782,676, 25.11.30. Appl., 17.5.24. Renewed 17.4.30).—A primary supply of oil is heated in a furnace to cracking temp. and discharged, under sufficient pressure to prevent material vaporisation, into a C-precipitating and reaction chamber wherein is introduced a secondary supply of oil heated in a tubular heating element in the same furnace to incipient cracking. The temp. of the mixed hydrocarbons is sufficient to crack the secondary supply of oil and any carbonaceous matter is deposited from the liquid mixture in a soft granular form. The heated hydrocarbons are then fractionated and condensed, those fractions suitable for re-cracking being returned to the system. Alternatively, the vapours from the secondary supply of oil may be led direct to the fractionator, and only the

unvaporised portion discharged into the reaction chamber. H. S. GARLICK.

**Cracking of hydrocarbons.** J. E. BELL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,782,056, 18.11.30. Appl., 12.11.24).—Cracking takes place in a series of stages of a pressure still in which the temp. is progressively lowered and the heat-exchanging surface is increased in proportion. The vapours are refluxed with fresh oil in such a way that the lightest oils pass off, but the heavier oils mix with the fresh oil passing to the first stage. Thus the concentration of tarry matter in the first (highest-temp.) stage of the cracking is reduced and tar deposition on the heating surface is prevented. The concentration of tar progressively increases in the succeeding cooler stages and is drawn off. The heavier cracked oils carried along with the fresh oil to the first stage thus undergo repeated cracking and only the lightest oils pass from the dephlegmator to the condenser. J. A. SUGDEN.

**Cracking of hydrocarbons.** H. K. ROGERS, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,781,884, 18.11.30. Appl., 11.10.28).—The cracked vapours from the pressure still are passed into a fractionating tower at an intermediate point so that stripping of the oil may take place in the lower part. The vapours passing from the condenser proper are further condensed by undergoing indirect heat exchange with a scrubbing medium in which the remainder of the uncondensed vapours have been absorbed later. This heated medium, containing absorbed vapours, passes to the stripping tower while the remainder of the uncondensed cracked vapours leaving the heat-exchanging condenser are recovered in the scrubber. J. A. SUGDEN.

**Splitting of hydrocarbons such as acetylene or the like.** J. MACHTOLF, Assr. to C. HOSTMANN-STEINBERG'SCHE FARBENFABR. G.M.B.H. (U.S.P. 1,782,540, 25.11.30. Appl., 9.12.26. Ger., 11.12.25).— $C_2H_2$  alone or mixed with other hydrocarbons is subjected to thermal dissociation in a suitable container. The product, including  $H_2$  and soot, is passed through a separator and a portion of the gas still containing a certain quantity of soot in suspension is repeatedly circulated through the splitting chamber and separating device until the chamber is entirely free from soot and only pure gas is being circulated. H. S. GARLICK.

**Manufacture of unsaturated hydrocarbons.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,620, 17.1.30).—Liquid or solid saturated hydrocarbons boiling above  $180^\circ$  are oxidised, preferably in the liquid phase, e.g., by blowing with air at  $140$ – $160^\circ$ , and the products are subjected to a sufficiently high temp. to cause them to split off  $H_2O$  and/or  $CO_2$ . Both stages of the process may be carried out catalytically. The oxidation products may be freed from carboxylic acids, e.g., by treatment with alkali, or may be separated into fractions sol. and insol. in light petroleum, before being heat-treated. A. B. MANNING.

**Apparatus for distilling hydrocarbon oil.** H. G. SCHNETZLER, Assr. to STANDARD OIL Co. (U.S.P. 1,779,918, 28.10.30. Appl., 10.3.27).—Condensed vapours from a fractionating column are received into a vessel fitted with a weir. The liquid flowing over the weir is distributed

in several compartments, from any one of which it may be returned to the fractionating column for refluxing purposes or passed to storage. The compartments are so arranged that the flow over the weir is divided in the ratio 2:8:4:3, and one or more of these portions may be returned as reflux. T. A. SMITH.

**Treatment of (A) [hydrocarbon] oil, (B) hydrocarbons.** J. DELATTRE-SEGUY, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,782,809 and 1,782,811, 25.11.30. Appl., 28.6.26).—(A) In the distillation of hydrocarbon oils, these are caused to flow continuously, by gravity in a thin film, over an unheated inclined surface. The heat necessary for distillation is supplied by radiation to the upper surface of the oil film by means of a heating surface placed above and spaced away from the upper surface of this film. The inclined surface and the heating surface are situated in a closed chamber provided with means for removing and condensing the evolved vapours. (B) The oil is passed in a continuous film (1 in. thick or less) by gravity along an inclined shallow channel in a cracking zone (or shell) heated to at least  $455^\circ$  at a pressure greater than 100 lb./in.<sup>2</sup>. Heat is applied to the upper surface of the oil film in order to avoid deposition of C, either by heating the upper portion of the shell externally with furnace gases, or by passing a molten material along a trough within the shell placed immediately above the oil channel.

R. W. L. CLARKE.

**Distillation of petroleum oils.** BARRETT Co., Assees. of A. A. MACCUBBIN and J. ZAVERTNIK (B.P. 346,407, 30.9.29. U.S., 15.12.28).—Petroleum is continuously distilled by passing it through a pipe coil where it is heated to a temp. sufficient to vaporise a large proportion of the lighter fractions, and is then discharged into a vapour-separating chamber at approx. atm. pressure, with the resultant separation of oil vapour and the production of a residual oil. The residual oil, which may be a lubricating oil stock, is drawn off while still hot and subjected to a high vac. in a vac. still so that approx. all the oil distillable at that temp. is vaporised without additional heat. H. S. GARLICK.

**Distillation of material and cracking of oil.** F. T. SNYDER (U.S.P. 1,781,934, 18.11.30. Appl., 8.6.25).—Solid material (coal, garbage, sawdust, oil shale, etc.) is distilled in a producer-like retort where the necessary heat is provided by combustion of the solid matter remaining after the material has passed the distillation zone. The vapours leaving the retort are cooled and the heavier oils, which are condensed, are passed back to the middle zone of the retort, where they undergo cracking. Free C which is deposited on the fuel passes down into the combustion zone. The light oils from the cooler are condensed and the uncondensed vapours undergo heat exchange in the cooler with the hot vapours from the retort. These reheated vapours are passed into the top of the retort, where they preheat the fresh fuel. This precaution prevents the condensation of oils upon the cold fuel and the consequent "sticking" of the descending charge. J. A. SUGDEN.

**Making motor fuel.** B. FIXMAN (U.S.P. 1,781,872, 18.11.30. Appl., 18.11.26).—Raw heavy oil passes through a pressure still and is sprayed in the liquid

phase to meet a stream of recycled oil in the vapour phase. The mixture of oils is passed, at a pressure sufficiently reduced to form a completely gaseous phase, into an electrostatic precipitator, where solid particles of C are removed. The vapours are then cooled and the light oils pass to a condenser, while the unconverted fraction is collected. According to the rate at which this fraction is collected, it is automatically pumped at a higher pressure through a second pressure still and superheater and becomes the recycle oil supply. A heat balance is maintained between the once- and twice-treated oils so that there is no fall in temp. in the condenser as the light oils distil off. J. A. SUGDEN.

**Treatment of hydrocarbons.** R. CROSS, Assr. to CROSS DEVELOPMENT CORP. (U.S.P. 1,782,808, 25.11.30. Appl., 20.1.26).—A process for refining cracked and other hydrocarbon vapours consists in fractionating the vapours and passing those of the lighter fractions first through towers containing Cu or oxides and salts of metals having an affinity for S, whereby desulphurisation is effected, and then through towers containing highly adsorbent material such as fuller's earth or bentonite, which effect purification and polymerisation. The purified vapours then pass to rectifying and condensing stages. R. W. L. CLARKE.

**Treatment of [removal of sulphide impurities from] hydrocarbons.** D. L. THOMAS (U.S.P. 1,781,826, 18.11.30. Appl., 23.7.27).—Sulphide impurities etc. are removed from oils, preferably in the vapour phase, by exposure to the combined action of atomic H (produced by electric arc) and a catalyst such as Sn or Pb. The gases after treatment pass over an Fe<sub>2</sub>O<sub>3</sub> purifier to remove the H<sub>2</sub>S. J. A. SUGDEN.

**Dewaxing of hydrocarbon oils.** A. R. GREIG, Assr. to TEXAS CO. (U.S.P. 1,782,467, 25.11.30. Appl., 20.12.27).—The economy of operation of centrifugal apparatus engaged in the separation of wax from chilled hydrocarbon oil is markedly improved by interrupting the flow of wax-bearing oil periodically to the centrifuge, when the capacity of the apparatus becomes reduced by the accumulation of solid wax and ice. A heating medium or steam is then introduced into the bowl of the machine by the usual oil inlet. The normal process is resumed when the accumulated solids have been removed from the working surfaces of the apparatus. R. W. L. CLARKE.

**Dewaxing of oils.** E. F. BURCH, Assr. to CREW LEVICK CO. (U.S.P. 1,782,028, 18.11.30. Appl., 14.3.27).—The oil containing precipitated wax is fed into a centrifuge so arranged that the wax is collected in an outer chamber and is separated from the incoming mixture by a chamber through which the separated oil is passed. Thus the separated wax may be melted and allowed to run out by suitable steam-heating, without any appreciable amount of heat reaching the incoming mixture and so redissolving some of the wax. J. A. SUGDEN.

**Recovery of waste clays [from petroleum refining].** W. D. RIAL and W. R. BARRATT, Assrs. to RICHFIELD OIL CO. OF CALIFORNIA (U.S.P. 1,782,744, 25.11.30. Appl., 5.1.27).—Clay from petroleum purification is washed with naphtha to remove oil, and then

with an "acid oil," obtained from acid sludge by separating its H<sub>2</sub>O-sol. constituents, to remove oil-insol. compounds, such as gums and resinoids. After a further washing with naphtha, the clay is heated to 82–204° and treated with superheated steam to eliminate odour and hydrate the clay. W. J. WRIGHT.

**Lubricant.** G. BROWN and F. LEVY (B.P. 346,379, 9.1.30).—Hg (1 pt.) is intimately mixed with petroleum jelly (2 pts.) to form a bearing lubricant, which may be subsequently rendered fluid by the addition of up to 10 times the wt. of a fluid mineral oil. [Stat. ref.]

H. S. GARLICK.

**Engine lubricant.** R. E. CLOUGH, Assr. to CASTOR LUBE REFINING CO. (U.S.P. 1,782,501, 25.11.30. Appl., 13.4.27).—Castor oil is gradually heated up to, and maintained at, not above 311° (at ordinary pressure) until not more than 4.2 wt.-% has been distilled off, and is mixed with mineral oil (20–98%).

E. LEWKOWITSCH.

**Lubricating compositions.** A. E. WHITE. From PANOLEUM CO. (B.P. 347,574, 25.10.29).—Compositions of desired consistency and m.p. are produced by adding to a lubricating oil 5–15% of an amide or anilide of a higher fatty acid, e.g., stearanilide, and 3–5% of a metal soap. A. B. MANNING.

**Mechanically-clinkered gas generators or producers.** HUMPHREYS & GLASGOW, LTD., and A. R. GRIGGS (B.P. 349,383, 12.8.30).

**Pulverised solid material.**—See I. Oxygenated compounds. Mineral oil amino-sulphonates. C<sub>6</sub>H<sub>6</sub> from butane etc.—See III. Fibrous products. —See V. Rubber-coated fabrics.—See VI. Fuel for firing bricks.—See VIII. Cement mixture [for oil wells]. Asphalt emulsions and compositions. Tar for roads. Road-building material.—See IX. Sulphonation products. Waxes.—See XII. Coating compound. Colour lakes from petroleum. Synthetic resin varnishes.—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Cooling on evaporation of solvents.** H. DABISCH (Farben-Ztg., 1931, 36, 1300).—By means of an apparatus based on the wet- and dry-bulb hygrometer, the cooling on evaporation of a range of solvents was determined under two sets of conditions (temp. 27° and 10°, and R.H. 63% and 93%, respectively). Five high-boiling solvents behaved anomalously, showing a max. instead of a min. temp. on the "wet thermometer." This is explained by their slight hygroscopicity.

S. S. WOOLF.

**Determination of hexamethylenetetramine methylenecitrate and hexamethylenetetramine when mixed.** D. MAROTTA and F. DI STEFANO (Annali Chim. Appl., 1931, 21, 150–154).—The mixture is first rendered alkaline and distilled, the amount of CH<sub>2</sub>O distilling over corresponding with the hexamethylenetetramine methylenecitrate present. The residue is then made acid and again distilled, the CH<sub>2</sub>O thus obtained being derived from the free hexamethylenetetramine. T. H. POPE.

**Hydrolysis of acetylindoxyllic acid and the acetylindoxyls.** G. SPENCER (J.S.C.I., 1931, 50,

63—64 r).—Alkaline hydrolysis of *N*-acetylindoxyl acid gave about 20% of indigo and 50% of *N*-acetyl-anthranilic acid;  $\text{HCl}$  and  $\text{FeCl}_3$  afforded mainly anthranilic acid and no indigo. Hydrolysis of *N*-acetylindoxyl with dil.  $\text{Na}_2\text{CO}_3$  in presence of air gave 55% of indigo, 8% of diacetylindigo, and 12% of isatin; dil.  $\text{NaOH}$  yielded mainly indigo (74%) and isatin, whilst acid  $\text{FeCl}_3$  gave only isatin. *O*-Acetylindoxyl gave indigo (95%) and isatin (3—4%) with dil.  $\text{Na}_2\text{CO}_3$ ; with  $\text{H}_2\text{O}_2$  and  $\text{NaOH}$  mainly isatin together with 18% of indigo and a little anthranilic acid were obtained. With acid  $\text{FeCl}_3$  indirubin (18%) and isatin were produced. E. H. SHARPLES.

Acetate from soda-pulp liquors.—See V. Ti compounds.—See VII. Diacetyl in butter.—See XIX. Trichloroethylene as disinfectant.—See XXIII.

See also A., June, 692, Catalyst for decomposition of  $\text{MeOH}$ . 693, Oxidation of  $\text{MeOH}$  with air. 718—9, Preparation of organo-Mg halides. 722, Fluorescence reaction for  $\beta$ -naphthol. 740, 2-Chloropyridines. 3-Aminopyridine. 748, Thioindigotins of the naphthalene and anthraquinone series.

## PATENTS.

Production of acetone from acetylene. J. MARTIN and I. J. KRCHMA, Asss. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,779,676, 28.10.30. Appl., 18.5.29).—A mixture of irreducible metal oxides and a metal halide is used as catalyst, *e.g.*,  $\text{Zn}$ ,  $\text{Cr}$ , and  $\text{Fe}^{+++}$  oxides with  $\text{ZnCl}_2$ , or  $\text{Cr}$  and  $\text{Zn}$  (or  $\text{Ni}$ ) oxides with  $\text{ZnCl}_2$ . C. HOLLINS.

Hydration of acetylene [to acetaldehyde and crotonaldehyde]. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,288, 6.1.30).—The use of a  $\text{Cd}$  phosphate catalyst leads to 91% conversion in one passage of  $\text{C}_2\text{H}_2$  with steam at 100 reciprocal hr. into acetaldehyde and crotonaldehyde. C. HOLLINS.

Manufacture of methylamine. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 344,971, 24.12.29).—Phthalimide, or other suitable imide, is methylated with  $\text{CH}_3\text{O}$  and hydrolysed to give pure  $\text{NH}_2\text{Me}$ . Examples are: phthalimide or *N*-hydroxymethylphthalimide with aq.  $\text{CH}_3\text{O}$  in  $\text{MeOH}$  at  $220^\circ/70$ — $90^\circ$  atm.; *o*-sulphobenzoic imide with trioxymethylene,  $\text{H}_2\text{O}$ , and  $\text{MeOH}$  at  $220^\circ$ . Diacetamide, succinimide, or cyanuric acid may also be used. C. HOLLINS.

Manufacture of polymerisation products [of alkylene oxides]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 346,550, 20.3.30).—Ethylene oxide, led over lumps of  $\text{KOH}$  at  $120$ — $130^\circ$ , evolves much heat and gives a wax, but over  $\text{NaHSO}_4$  at  $120^\circ$  it gives dioxan and a little glycol ethylidene ether, b.p.  $82^\circ$ . Propylene oxide with alkaline catalysts gives an oil; with acid catalysts 2:5-dimethyldioxan, b.p.  $117^\circ$ , or  $50^\circ/2$  mm. C. HOLLINS.

Polymerisation of vinyl derivatives. E. I. DU PONT DE NEMOURS & Co. (B.P. 319,587—8, 23.9.29. U.S., 22.9.28).—(A) Vinyl compounds (vinyl chloride or acetate, styrene) are polymerised, especially in a solvent ( $\text{MeOH}$ , ethylbenzene,  $\text{BuOH}$ , ethylene dichloride), by treatment with  $\text{O}_3$ , with the assistance of

heat ( $90$ — $120^\circ$ ) or light. The solvent may be pre-treated with  $\text{O}_3$ . (B) A solution of a vinyl compound and a polymerisation catalyst are passed through a hot tube, and preferably are thereafter rapidly cooled; *e.g.*, vinyl chloride and benzoyl peroxide in  $\text{PhCl}$ , forced through a Sn-lined tube at  $118^\circ$ , give a new polymeride sol. in  $\text{PhMe}$ . C. HOLLINS.

Production of the  $\alpha$ -polymeride of vinyl chloride. E. I. DU PONT DE NEMOURS & Co. (B.P. 319,591, 23.9.29. U.S., 22.9.28).—Vinyl chloride, preferably in a solvent (ethylene dichloride,  $\text{MeOH}$ ,  $\text{PhCl}$ ,  $\text{AcOEt}$ ), is heated with a catalyst (benzoyl peroxide, ozonised solvent) above  $80^\circ$ , *e.g.*, at  $120^\circ$ . The  $\alpha$ -polymeride is obtained substantially free from the  $\beta$ -compound. C. HOLLINS.

Purification of synthetic alcohols. J. R. PARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 344,796, 10.12.29).—Alcohols from hydrogenation of C oxides are treated with caustic alkali for several days, preferably with stirring with a current of air followed by keeping for some days before distillation. C. HOLLINS.

Manufacture of oxygenated organic compounds. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 345,391, 16.1.30. Addn. to B.P. 312,388; B., 1929, 746).—Alcohols are the main product if in the process of the prior patent a petroleum of b.p. range  $40$ — $130^\circ$  is oxidised in presence of 2% or more of a saturated aliphatic acid below  $\text{C}_5$ ; *e.g.*, hexane oxidised in presence of 10% of  $\text{AcOH}$  and 0.05% of  $\text{Mn}$  acetylacetone with air at  $145^\circ/20$  atm. gives mainly acetates of lower alcohols. C. HOLLINS.

Manufacture of ketones [acetone from ethyl alcohol]. H. DREYFUS (B.P. 345,271, 18.12.29).— $\text{EtOH}$  vapour passed over  $\text{Ba(OH)}_2$  in a ( $\text{Cu}$ ) tube at  $250$ — $350^\circ$  gives  $\text{Ba(OAc)}_2$ , or at  $400$ — $500^\circ$  it gives  $\text{COMe}_2$  directly. C. HOLLINS.

Preparation of ethyl esters. A. R. CADE, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,781,050, 11.11.30. Appl., 7.7.23).—An org. Na salt ( $\text{BzONa}$ ) is treated with  $\text{Et}_2\text{SO}_4$  in presence of the Et ester ( $\text{BzOEt}$ ) as solvent and in substantial absence of  $\text{H}_2\text{O}$ . C. HOLLINS.

Production of lactic esters. IMPERIAL CHEM. INDUSTRIES, LTD., W. R. H. HURTLEY, and T. S. WHEELER (B.P. 346,486, 25.1.30).—The lactic ester of one alcohol is heated with another alcohol, *e.g.*, ethyl lactate with butyl alcohol, and the displaced alcohol is removed continuously or at intervals. C. HOLLINS.

Preparation of butadiene. E. K. BOLTON and F. B. DOWNING, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,777,600, 7.10.30. Appl., 8.1.27).—Diacetylene and  $\text{H}_2$  are passed over a reduced  $\text{Ni}$  catalyst at  $40$ — $80^\circ$ . C. HOLLINS.

Manufacture of butadiene or its homologues. I. G. FARBERIND. A.-G. (B.P. 345,270, 18.12.29. Ger., 18.12.28).—Olefinic alcohols above  $\text{C}_3$  give diolefines when passed as vapour, preferably with steam, over a dehydrating catalyst (dried alums, acid phosphates) at  $200$ — $300^\circ$ . Allylcarbinol yields 85—90% of butadiene;  $\beta$ -methyl- $\Delta^2$ -penten- $\delta$ -ol, b.p.  $127$ — $130^\circ$ , gives 80% of dimethylbutadiene, b.p.  $75.5$ — $77^\circ$ . C. HOLLINS.

**Mineral oil amino-sulphonates [detergents].** W. T. REDDISH, Assr. to TWITCHELL PROCESS CO. OF CINCINNATI (U.S.P. 1,780,144, 28.10.30. Appl., 28.5.28).—An oil-soluble mineral oil sulphonic acid is combined with "triethanolamine" or other hydroxylated alkylamine. C. HOLLINS.

**Manufacture of basic products [dialkylamino-alkyl derivatives of higher fatty acids].** I. G. FARBENIND. A.-G. (B.P. 346,387, 2.1.30. Ger., 2.1.29).—A higher fatty acid, ester, chloride, or amide is heated at 200—230° with a di- or poly-amino-alcohol, one  $\text{NH}_2$  group of which is tertiary, to give thickening agents for cosmetics, emulsifying agents, etc. Examples are:  $\beta$ -amino- $\beta'$ -diethylaminoisopropyl alcohol with oleic acid, ethyl oleate, stearamide;  $\beta$ -anilino- $\beta'$ -diethylaminoisopropyl alcohol with oleyl or stearyl chloride. C. HOLLINS.

**Manufacture of benzene and by-products from butane, propane, ethane, or mixtures thereof.** F. PORTER, Assr. to CONTINENTAL OIL CO. (U.S.P. 1,777,894, 7.10.30. Appl., 27.4.27).—The initial gases are passed downward through a brick-packed tower at 800—950°, the speed being adjusted as the tower cools. C. HOLLINS.

**Indophenol intermediate and blue sulphur dye derived therefrom.** W. A. MANSS, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,777,757, 7.10.30. Appl., 27.10.25).— $\text{NHPh}_2$  in 93%  $\text{H}_2\text{SO}_4$  is mixed with *p*-nitrosophenol in 64%  $\text{H}_2\text{SO}_4$ , the mixture being kept below 0° (e.g., —20°). The product is sulphurised in the usual manner. C. HOLLINS.

**Manufacture of multivalent metal salts of half-esters of phthalic acid.** W. J. BANNISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,779,654 and 1,779,688, 28.10.30. Appl., [A] 8.7.29, [B] 13.6.28).—Phthalic anhydride and  $\text{BuOH}$  are heated in  $\text{PhMe}$  at 100°, aq.  $\text{NaOH}$  is added below 50° until alkaline to phenolphthalein, then aq.  $\text{ZnSO}_4$ . The resin is freed from solvent and  $\text{H}_2\text{O}$  by distillation below 80°. Alternatively, the alkyl hydrogen phthalate may be treated directly with  $\text{ZnO}$  in presence of the org. solvent. C. HOLLINS.

**Production of imino-ethers.** K. F. SCHMIDT and P. ZUTAVERN (B.P. 345,397, 18.1.30. Addn. to B.P. 331,947; B., 1930, 939).—Acid amides or lactams are acylated (e.g., with benzenesulphonyl chloride) and treated with an alcohol, whereby the *O*-acyl group is replaced by alkyl. Cyclic imino-ethers are thus obtained from piperidone (b.p. 161—165° in vac.) and leucine-lactam. C. HOLLINS.

**Manufacture of unsaturated ethers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,253, 12.12.29).—An acetal vapour is led at 200—350° over a Pd, Ag, or other noble-metal catalyst, preferably on a carrier (asbestos, clay,  $\text{SiO}_2$  gel, Si). Other metals of groups II—IV, V—VIII, or compounds of these or of group I metals, may be added. Vinyl ethyl ether is obtained from diethyl acetal; vinyl *n*-butyl ether, b.p. 92—93°, from di-*n*-butyl acetal; isopropenyl ethyl ether, b.p. 61—62°/735 mm., from  $\beta\beta$ -diethoxypropane; *n*-propenyl ethyl ether, b.p. 69°, from  $\alpha\alpha$ -diethoxypropane; *n*-butenyl methyl ether, b.p. 73—74°/749 mm.,

from  $\alpha\alpha$ -dimethoxybutane;  $\alpha$ -styryl ethyl ether, b.p. 93—96°/12.5 mm., from  $\alpha\alpha$ -diethoxyethylbenzene; and vinyl  $\beta$ -ethoxyethyl ether [glycol vinyl ethyl ether], b.p. 128°, from ethylidene di- $\beta$ -ethoxyethyl ether, b.p. 106—116°/14 mm. C. HOLLINS.

**[Production of] dye intermediates [nitro- and amino-derivatives of 2-(3 : 4-dichlorobenzoyl)benzoic acid].** R. J. LOVELUCK, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 345,277, 13.9.29).—2-(3 : 4-Dichlorobenzoyl)benzoic acid is nitrated with 97%  $\text{HNO}_3$  at 15° or mixed acids at 80°, and the product is reduced. C. HOLLINS.

**Production of condensation products [o-benzoylbenzoic acids, their nitro- and amino-compounds, and anthraquinones] from o-chlorophenol.** R. TONKIN, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 345,204, 4.12.29).—*o*-Chlorophenol is condensed with phthalic anhydride, the product is nitrated, reduced, and cyclised to give a mixture of chloroaminohydroxy-anthraquinones. C. HOLLINS.

**Manufacture of halogenated 3 : 4 : 8 : 9 [2 : 3 : 7 : 8]-dibenzpyrene-5 : 10[1 : 6]-quinones.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,650, 23.9.29).—Uniform products are obtained by mono- or di-chlorinating or -brominating 2 : 3 : 7 : 8-dibenzpyrene-1 : 6-quinone (a) in an organic medium (nitrobenzene) in presence of a carrier, (b) in aq. suspension, (c) in absence of diluent, (b) and (c) preferably in presence of a carrier (S). Bromination gives only a monobromo-compound. The products are yellow to orange vat dyes. C. HOLLINS.

**Liquid supersaturated solution of thiocarbamilide.** P. SIEDLER and A. MOELLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,780,634, 4.11.30. Appl., 16.5.29. Ger., 28.1.28).—Premature crystallisation of supersaturated solutions of thiocarbamilide, e.g., in *o*-toluidine, is prevented by addition of 0.3—20% of colophony, Venetian turpentine, resins from  $\text{CH}_2\text{O}$  and  $\text{NH}_3$  or amines, low-temperature carbonisation tar, etc. The use of mixed toluidines in place of pure *o*-toluidine is made possible. C. HOLLINS.

**Manufacture of aminoarylthiazole compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,735, 22.1.30).—2-Amino-benz- and -naphtha-thiazoles are obtained by action of  $\text{Cl}$ ,  $\text{SO}_2\text{Cl}_2$ , etc. on arylthiocarbamides,  $\text{Ar} \cdot \text{NX} \cdot \text{CS} \cdot \text{NH}_2$ , where X is H, alkyl, or aralkyl. 2-Amino- (m.p. 126—128°), 2-amino-4-methyl- (m.p. 138°), 2-amino-6-ethoxy- (m.p. 160—163°), 6-chloro-2-amino-4-methyl- (m.p. 200—205°), and 2-amino-6-methyl- (m.p. 142°)-benzthiazoles, 2-imino-3-methyl- (m.p. 123°) and 2-imino-3-phenyl-benzthiazolines, and 2-amino- $\alpha$ -naphthathiazole, m.p. 259—261°, are thus prepared. Amongst the starting materials are: *o*-tolyl- (m.p. 156—158°), 5-chloro-*o*-tolyl- (m.p. 175—177°), and *p*-tolyl- (m.p. 188°)-thiocarbamides. C. HOLLINS.

**Aldehyde-amine condensation products [ulcanisation accelerators].** I. WILLIAMS and W. B. BURNETT, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,780,326 and 1,780,334, 4.11.30. Appl., [A] 13.3.25, [B] 12.2.26).—An aldehyde is caused to react with an amine in mol. ratio above 2 : 1,  $\alpha$ -unsaturated



aldehydes being excluded, (B) in presence of an acid. Examples are: (A) heptaldehyde with *n*-butylamine; *n*-butaldehyde with aniline, with *o*-tolylidiguanidine, or with *n*-butylamine; propaldehyde with aniline; the mol. proportions being 2, 3, 5, 7, 10, or 15 : 1; (B) *n*-butaldehyde with aniline or *o*-toluidine, and heptaldehyde with *n*-butylamine, in presence of *n*-butyric, stearic, or *n*-valeric acids, AcOH, or H<sub>2</sub>SO<sub>4</sub>, the mol. ratio of aldehyde to amine being 3 or 5 : 1.

C. HOLLINS.

**Manufacture of substituted thiuram polysulphides [vulcanisation accelerators].** G. S. WHITBY, ASSR. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,545, 4.11.30. Appl., 28.4.28).—A disubstituted dithiocarbamate is treated with a S chloride, which may contain dissolved S; e.g., K dimethyldithiocarbamate and S<sub>2</sub>Cl<sub>2</sub> in Et<sub>2</sub>O give tetramethylthiuram disulphide, m.p. 108°.

C. HOLLINS.

**Manufacture of albumose-like degradation products.** A. CARPMAEL, FROM I. G. FARBENIND. A.-G. (B.P. 345,630, 24.12.29).—Wool, horn, hair, or other keratin-containing substance is converted into H<sub>2</sub>O-sol. degradation products containing thiol groups by hydrolysis (e.g., with dil. HCl) and reduction (e.g., with Sn and HCl), which may be simultaneous.

C. HOLLINS.

**Manufacture of thymol and its isomerides and homologues.** SCHERING-KAHLBAUM A.-G. (B.P. 344,970, 24.12.29. Ger., 31.12.28. Addn. to B.P. 273,685 and 279,855; B., 1930, 136; 1929, 237).—The condensation product obtained according to B.P. 273,684 (B., 1929, 236) from *m*- or *p*-cresol and an aliphatic ketone (COMe<sub>2</sub>) is heated in H<sub>2</sub> at 300°, and the vapours and H<sub>2</sub> are passed over reduced Ni on pumice at 200°.

C. HOLLINS.

**Purification of chloronaphthalenes.** HALOWAX CORP., ASSEES. of S. BROWN and E. R. HANSON (B.P. 343,878, 18.10.29. U.S., 19.10.28).—Crude chlorinated C<sub>10</sub>H<sub>8</sub>, prepared by chlorinating molten C<sub>10</sub>H<sub>8</sub> until the desired sp. gr. is reached (*d* 1.41—1.43 at 150° for higher chlorination products, 1.20 for mono- and di-chloronaphthalenes), is fractionally distilled at reduced pressure (e.g., 100—150 mm.), preferably after neutralising with CaO, NaOH, etc. Mono- and di-chloro-derivatives are collected at 140—180°, higher derivatives at 200—225° and 225—250°, the last fraction being the most valuable. Suitable plant is figured.

C. HOLLINS.

**Manufacture of [derivatives of] hydrocarbons containing halogenomethyl [substituents].** I. G. FARBENIND. A.-G. (B.P. 345,146, 7.5.30. Ger., 8.5.29).—A polynuclear hydrocarbon is treated with aq. CH<sub>2</sub>O and H halide (HCl), without condensing agent or org. solvent, so as to introduce 2 or more halogenomethyl groups. The chloromethyl derivatives of naphthalene (m.p. 130—140°), anthracene, and tetrahydronaphthalene (m.p. 117.5°) are described.

C. HOLLINS.

**Production of acetyl-Cleve [acetyl 1:6(7)-naphthylaminesulphonic] acid.** W. M. RALPH, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,778,914, 21.10.30. Appl., 9.7.25. See U.S.P. 1,566,425; B., 1926, 185).—Cleve acid is heated with AcOH and AcONa in substantial absence of H<sub>2</sub>O. C. HOLLINS.

**Purification of anthracene.** C. J. THATCHER (U.S.P. 1,782,488, 25.11.30. Appl., 16.6.23).—The crude anthracene (30% pure) is ground in a ball mill with 1—2 times its wt. of a solvent, e.g., C<sub>6</sub>H<sub>6</sub>, CCl<sub>4</sub>, in which anthracene (and carbazole) is relatively insol., but which dissolves phenanthrene, oils, etc.; carbazole can be removed from the finely-divided product (45% anthracene) by solvents, e.g., COMe<sub>2</sub> or pyridine, or these may be used as the grinding solvent.

E. LEWKOWITSCH.

**Manufacture of [hydroxylated] anthraquinone derivatives.** BRIT. CELANESE, LTD., E. W. KIRK, H. C. OLFIN, and G. H. ELLIS (B.P. 346,355, 8.1.30).—In the nitrosylsulphuric acid method for introducing OH groups into anthraquinones, the amount of Hg catalyst should be 0.05—0.40%; P<sub>2</sub>O<sub>5</sub> may be added. Anthraquinone is oxidised to quinizarin, 1:8-dihydroxyanthraquinone to 1:5:8-trihydroxyanthraquinone.

C. HOLLINS.

**Manufacture of halogenated products of anthraquinone derivatives [halogenated anthraquinoneacridones from anthraquinonylisatins].** J. Y. JOHNSON, FROM I. G. FARBENIND. A.-G. (B.P. 346,359, 30.10.29).—*N*-Anthraquinonylisatins are halogenated and converted into halogenated anthraquinoneacridones. Examples are: *N*-α-anthraquinonylisatin, dichlorinated, converted into the acridone (red vat dye) by vating; or chlorinated and brominated in presence of NaCl-AlCl<sub>3</sub> to give directly the chlorobromoacridone (blue-red); or brominated in presence of I and Fe in nitrobenzene at 160—170° for the bromoacridone (blue-red); 4(or 6)-chloro-1-α-anthraquinonylisatin, brominated for a chlorobromoacridone (blue-red), or chlorinated and vatted for the chloroacridone; 1-α-anthraquinonyl-5-methylisatin chlorinated for the chloroacridone (red); *N*-α-anthraquinonyl-β-naphthisatin brominated in presence of S and I in nitrobenzene at 140—160° for the bromoacridone (yellow-red).

C. HOLLINS.

**Splitting of acetylene.**—See II. Alginic acid products.—See VII. EtOH.—See XVIII. Cineoles.—See XX.

#### IV.—DYESTUFFS.

**Hydrolysis of acetylindoxyllic acid etc.**—See III.

See also A., June, Absorption of dye granules by hydrosols. 721, Azo chromophores. 722, Light-sensitive diazo compounds. 725, Nitro-dye from aminosulphosalicylic acid. 732, Insol. disazo dyes from 1:5-diaminoanthraquinone. 733, Plant colouring matters. 738, Dye of the corn poppy. Dye of acacia wood. Anthochlor of *Linarea vulgaris*. 741, ψ-Cyanine dyes. 748, Thioindigotins of the naphthalene and anthraquinone series. 777, Colouring matter of "Awobana."

#### PATENTS.

**Dye preparation.** M. P. PARRISH (U.S.P. 1,780,981, 11.11.30. Appl., 19.12.28).—An aniline dye is pre-digested with pancreatin, ox gall, NaCl, citric acid, AcOH, and COMe<sub>2</sub>, boiled with soap and tinctures of camomile flowers, hyssop, ground cedar wood, and myrrh, then fermented for 2 weeks with pancreatin, gelatin, COMe<sub>2</sub>, NaCl, ox gall, and AcOH, and finally

baked with dextrin until crisp. The product dyes cotton and wool directly. C. HOLLINS.

**[Manufacture of] indigoid dyes.** W. BAUER, W. NEELMEIER, and T. NOCKEN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,782,706, 25.11.30. Appl., 9.5.27. Ger., 11.6.26).— $\beta\beta$ -Naphthisatin  $\alpha$ -chlorides (etc.), having halogen in the 4-position, are condensed with a 4-alkoxy- $\alpha$ -naphthol, having the 2-position free, to give green vat dyes; e.g., brominated 4-chloro- $\beta\beta$ -naphthisatin, m.p. 313°, with 4-methoxy- $\alpha$ -naphthol. C. HOLLINS.

**Manufacture of indigoid vat dyes.** I. G. FARBENIND. A.-G. (B.P. 345,130, 16.4.30. Ger., 22.4.29).—6-Methylthioindoxyl is condensed with 5-chloro- or 5-bromo-isatin  $\alpha$ -chloride (etc.) to give fast grey vat dyes. C. HOLLINS.

**Manufacture of indigoid dyes.** SOC. CHEM. IND. IN BASLE (B.P. 345,743, 28.1.30. Switz., 2.2.29).—4 : 5-Dichloro-7-methylthioindoxyl is condensed, e.g., with 5 : 7-dichloroisatin  $\alpha$ -chloride (violet vat dye), acenaphthoquinone (red), isatin  $\alpha$ -anil (red-violet; brominated, violet), 9-chloro- $\beta\beta$ -naphthathioisatin  $\alpha$ -*p*-dimethylaminoanil (violet). C. HOLLINS.

**Manufacture of [thio]indigoid dyes.** I. G. FARBENIND. A.-G. (B.P. 345,349, 23.12.29. Ger., 22.12.28).—A pyrazolothioindoxyl is oxidised or is condensed with an isatin or thioisatin  $\alpha$ -chloride (etc.) to give vat dyes. 6-Aminoindazole, m.p. 210°, is converted by way of its 5- or 7-thiocyano-derivative, m.p. above 300°, into 6-aminoindazolyl-5- or -7-thioglycollic anhydride, m.p. above 300°, the  $\text{NH}_2$  group is exchanged for the CN group, and the product is heated with  $\text{Na}_2\text{S}$  solution to give, after treatment with  $\text{H}_2\text{SO}_4$ , 5 : 6- or 7 : 6-pyrazolothioindoxyl, m.p. 235°, which is oxidised to a grey vat dye. The *N*-Me compound from 6-amino-2-methylindazole (thiocyano-derivative, m.p. 280—285°; thioglycollic anhydride, m.p. 278—280°; thioindoxyl, m.p. 200°) dyes olive shades. 6-Amino-5-methylindazole, m.p. 240—242° (7-thiocyano-derivative and thioglycollic anhydride, m.p. above 300°; thioindoxyl, m.p. 285°) gives a grey vat dye. The pyrazolothioindoxyl first mentioned is condensed with  $\beta\beta$ -naphthathioisatin  $\alpha$ -anil (bluish-grey), 5-chloroisatin  $\alpha$ -chloride (bluish-grey),  $\beta$ -naphthisatin  $\alpha$ -chloride (olive-green), and  $\alpha$ -naphthathioisatin (grey). C. HOLLINS.

**Manufacture of vat dyes [from naphthalene-tetracarboxylic dianhydride and *o*-diamines].** I. G. FARBENIND. A.-G. (B.P. 344,779, 29.10.29. Ger., 29.10.28).—Vat dyes from naphthalenetetracarboxylic dianhydride and halogenated *o*-arylenediamines are condensed with suitable  $\text{NH}_2$  compounds. The product from 4-chloro- or 4-bromo-*o*-phenylenediamine is condensed with  $\alpha$ -aminoanthraquinone (corinth), 1-amino-2-methylanthraquinone (dull brown), 1-amino-5-benzamidoanthraquinone (dull brown), 1-amino-4-benzamidoanthraquinone (greenish-grey); that from 3 : 5-dibromo-*o*-phenylenediamine with  $\alpha$ -aminoanthraquinone (grey-brown). Similar products are obtained by condensing halogeno-compounds with diaminobisimidazoles made, e.g., from nitro-*o*-phenylenediamine. C. HOLLINS.

**Manufacture of vat dyes [of the thiazolanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,373, 30.9.29).—Thiazolanthrone-3-carboxylic

chloride etc. is condensed with an *o*-amino-phenol or -thiophenol or an *o*-diamine, the product, if desired, being halogenated, nitrated, etc. With 2-amino-3-hydroxyanthraquinone and with *o*-phenylenediamine yellow vat dyes are obtained. C. HOLLINS.

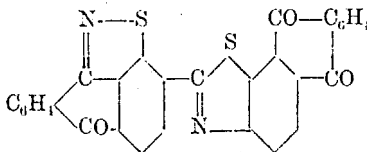
**Manufacture of vat dyes of the *N*-dihydro-1 : 2 : 2' : 1'-anthraquinoneazine [indanthrone] series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,328 and 344,777, [A, B] 2.10.29, [A] 8.1.30).—(A) The condensation product from impure indanthrone and  $\text{CH}_2\text{O}$  is halogenated, or is first treated with P pentahalide (etc.) and then purified and/or halogenated by means of halogen. Fast greenish-blues are obtained. (B) The vat dyes obtained by oxidising the condensation products from indanthrones and  $\text{CH}_2\text{O}$  (B.P. 322,281; B., 1930, 316) are halogenated to give redder shades of increased fastness to bleach. C. HOLLINS.

**Vat colour of the 1 : 9-thiazolanthrone series.** R. N. LULEK, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,777,756, 7.10.30. Appl., 18.3.27).—A yellow vat dye (annexed formula) is obtained by condensing 2-amino-1-thiolanthraquinone in  $\text{PhNO}_2$  at 180° with thiazolanthrone-*o*-carboxylic chloride or the corresponding aldehyde. C. HOLLINS.

**Manufacture of [vat] dyes [chlorinated dibenzanthrone].** BRIT. ALIZARINE Co., LTD., C. W. SOUTAR, and J. ANDERSON (B.P. 345,623, 24.12.29).—Indanthrone is heated with large excess (1.6—3.4 pts.) of  $\text{SO}_2\text{Cl}_2$  below 80° (65—75°) for about 24 hr. or for about 5—6 hr. in presence of a carrier, preferably in a large bulk of solvent ( $\text{PhNO}_2$ ), in which the dibenzanthrone may be preheated and cooled before chlorination. Blue, not violet, vat dyes result. C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone-pyrazolanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,651, 14.10.29. Addn. to B.P. 341,884; B., 1931, 476).—All the Br in the brominated benzanthronepyrazolanthrone of the prior patent is exchanged for one or more of the groups NHR, OR, and SR; the products may be alkylated and/or further condensed, e.g., by alkaline fusion. The dibromocompounds are condensed with 1-aminoanthraquinone (grey vat dye), 1-amino-4-benzamidoanthraquinone (green-grey), 2-aminoanthraquinone (blue-grey), benzamide (green-blue), and 2 : 4-dichloro-7-amino-8 : 9-phthaloylacridone (green-grey); the monobromocompound is condensed with 1-aminoanthraquinone (blue-grey to blue-black), and  $\text{PhOK}$  (navy-blue). C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone-pyrazolanthrone series].** I. G. FARBENIND. A.-G., and A. G. BLOXAM (B.P. 345,728, 20.1.30. Addn. to B.P. 298,284; B., 1928, 923).—A substituted (especially anthraquinonyl) amino-derivative of an *N*-3-benzanthronylpyrazolanthrone is heated with (alcoholic) alkali to give a vat dye, usually bluish-grey. Examples of starting materials are the condensation products from *N*-(9-bromo-3-benzanthronyl)pyrazolanthrone with



1-amino-, 2-amino-, 1:4-diamino-, 1-amino-4-benzamido-, 1-amino-5-benzamido-, and 1-amino-2-aldehydo-anthraquinones (grey to black), 3-aminobenzanthrone, aminobenzanthronylpyrazolanthrone, carbazole, or 1:2-diaminoanthraquinone; from 5-, 7-, or 10-chloro-*N*-3-benzanthronylpyrazolanthrone with 1-aminoanthraquinone, etc. C. HOLLINS.

**Disazo dye containing a diphenylcarbamide nucleus.** L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., Inc. (U.S.P. 1,782,682, 25.11.30. Appl., 28.4.24).—The dye *p*-nitroaniline  $\rightarrow$  salicylic acid is reduced and phosgenated together with the dye *o*-toluidine-4-sulphonic acid  $\rightarrow$  cresidine. The product is a direct yellow dye for cotton. C. HOLLINS.

**Dye-works.**—See I. Blue sulphur dye. Halogenated dibenzpyrenequinones. Halogenated anthraquinoneacridones.—See III. Lakes etc.—See XIII. Colouring butter etc.—See XIX.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Structure of textile fibres.** (MILE.) V. BOSSUYT and G. CHAUDRON (Compt. rend., 1931, 192, 1050—1053).—The transverse discontinuities visible, best in polarised light, in many textile fibres, and especially in flax, are not due to fracture caused by scutching (cf. B., 1922, 853 A), being equally visible in fibres separated without scutching. Comparison of a stretched and an unstretched fibre after treatment with 25% NaOH solution shows that they are merely folds of the external fibrils, and disappear on stretching. C. A. SILBERRAD.

**Mounting of textile fibre sections.** J. M. PRESTON (J. Soc. Dyers and Col., 1930, 46, 295—297).—When searching for foreign matter in a section it is preferred to use a mountant having the same *n* value as the section. Aq. mountants are not suitable for textile fibres and particularly for regenerated cellulose silks because of the consequent swelling. The most suitable mountants for fibre sections (other than cellulose acetate) are collodion of *n* 1.44 (for contrast) and Canada balsam of *n* 1.53 (for rendering invisible); for cellulose acetate fibre sections no mountant is available which does not cause swelling, but the most suitable are glycerin jelly of *n* 1.37 (for contrast) and Euparal of *n* 1.50 (for rendering invisible). A. J. HALL.

**Measurement of the colour of textile fabrics.**

**IV. The Ostwald colour system.** P. W. CUNLIFFE and P. N. LAMBERT (J. Soc. Dyers and Col., 1930, 46, 297—307).—The instruments used and the principles on which the Ostwald colour system is based are discussed critically in relation to actual colour measurements of the standards by the Guild colorimeter. It is concluded that the grey standards are satisfactory as regards their neutral colour and their brightness, but with "full" or "pure" colour standards the steps are too close together in the blue and blue-green regions and too widely separated in three other regions. Colours which are diametrically opposite in the colour standard circle deviate slightly from the requirement that they should be complementary. A. J. HALL.

**Determination of the quantity of silk in silk fabric.** D. ONGARO (Giorn. Chim. Ind. Appl., 1931, 13, 159—162).—Determination of silk by the Kjeldahl

method requires the preliminary removal of nitrogenous dyes and, like other suggested methods, occupies much time. The use of Van Slyke's method leads to variable results, owing to the influence of extraneous substances on the hydrolysis of the silk proteins and to the formation of artificial melanins or humin substances. The amino-N in fibroin may be determined by heating with alkaline  $\text{KMnO}_4$  for  $\frac{1}{2}$  hr. and titrating the  $\text{NH}_3$  distilled. The % N in dry de-gummed fibroin, thus determined, is 15.89, the ratio between the weights of fibroin and N varying, for cocoons of different races, from 6.26 to 6.32. With various silk tissues this method gives results agreeing closely with those of the determination of total N by Kjeldahl's method.

T. H. POPE.

**Decomposition of maize stalks by nitric acid.** E. HORVÁTH and G. EBER (Cellulosechem., 1931, 12, 85—95; cf. B., 1929, 937, 974).—The cell substance remaining after treatment of maize stalks with 2, 2.5, and 3%  $\text{HNO}_3$  at 70°, 90°, and 98.3° (bath temp.) during various periods has been determined. Most of the decomp. appears to take place while the temp. of the reaction mixture is rising to that of the bath, which temp. is never exceeded. It is concluded that the more conc. is the acid and the higher the bath temp., the more speedily the reaction is ended, little reaction taking place after the temp. of the reaction mixture is approx. that of the bath. For a given yield, the higher is the bath temp. and the more conc. the acid, the greater is the quantity of acid used up in the reaction. J. D. A. JOHNSON.

**Action of caustic soda on cellulose.** S. M. NEALE (J.S.C.I., 1931, 50, 177—182 r).—Recent theoretical developments are summarised and special attention is devoted to the characteristic properties of "mercerised cellulose." This material merely exhibits in greater degree the chemical properties of the original cellulose, and must be clearly distinguished from cellulose modified by oxidation or acid attack, which exhibits new chemical properties. Various methods may be used for estimating the "activation" of cellulose which persists after swelling; the results they yield are compared.

**Affinity of nitrocellulose for gelatinising agents.** J. DESMAROUX (J. Chim. phys., 1931, 28, 163—173).—A discussion of published work on the dissolution, swelling, and gelation of nitrocellulose in a number of solvents and solvent mixtures. E. S. HEDGES.

**A reaction of sulphite-cellulose.** C. KULLGREN (Svensk Kem. Tidskr., 1931, 43, 99—105).—Sulphite-cellulose removes Cu from a dil. solution in distilled  $\text{H}_2\text{O}$  filtered through it, the action being due to a base exchange similar to that operating in permutit filters; in one experiment 95% of the Cu present in solution was taken up by the cellulose. The rate of absorption of the Cu is practically instantaneous, but the total amount which can be taken up is small, sulphite-cellulose with 0.5% S taking up only 0.5% Cu. The presence of other cations, especially Ca and Na, in concentrations of more than 100 times that of the Cu, entirely prevents the absorption of the latter. The Cu taken up by sulphite-cellulose can be detected by treatment with KI and starch in the usual way. H. F. HARWOOD.

**Effect of swelling agents on the creasing properties of viscose silk.** A. J. HALL (J. Soc. Dyers and Col., 1930, 46, 293—294).—After swelling treatments with cold 5% NaOH,  $H_3PO_4$  ( $d$  1.55), and  $H_2SO_4$  ( $d$  1.44), viscose yarn has an increased affinity for direct dyes, but a slightly decreased elasticity and resistance to creasing as measured by the methods described previously (B., 1930, 1023), except in the case of  $H_3PO_4$ , when the swelled silk is somewhat less creasable than the untreated silk.

A. J. HALL.

**Determination of the degree of decomposition of pulps.** K. BERNDT (Zellstoff u. Papier, 1931, 11, 257—258).—A summary (with references) of the methods in use for indicating or determining the extent of purification of pulps is given. The methods comprise staining, e.g., with malachite-green or a mixture of this with Congo-red, measurement of fluorescence, determination of lignin and of halogen consumption, and the use of oxidation methods, e.g., with saturated aq. quinone or with  $KMnO_4$ . There is an approx. linear relation between lignin content and Cl consumption.

B. P. RIDGE.

**Recovery of acetate from the black liquors of soda-pulp manufacture.** H. SCHMIDT (Zellstoff u. Papier, 1931, 11, 259—260).—The liquor is used in continuous process and submitted to pressure and heat (about 350° and 150—200 atm.), whereby it is largely freed from organic impurities. Its alkalinity is also raised by addition of NaOH or by treatment with CaO and the process is continued until an acetate content of 10—15% is reached, when part of the liquor is drawn off for recovery of the acetate as such, or as MeOH or  $COMe_2$ . High yield of  $COMe_2$  is obtained by evaporating the solution and dry-distilling the residue.  $AcONa$  is obtained by evaporation and subsequent crystallisation; dissolved NaOH does not interfere with this process. A method is also described whereby soda-free acetate may be obtained. The yield of  $AcONa$  depends on the nature of the wood or other raw material; with pine wood about 13% on the wt. of the dry wood is recovered.

B. P. RIDGE.

**Paper-making properties of New Zealand flax (*Phormium tenax*).** M. B. SHAW, G. W. BICKING, and M. J. O'LEARY (Bur. Stand. J. Res., 1931, 6, 411—420).—*P. tenax* should be a satisfactory material for the manufacture of wrapping and writing papers, the scutched material being the most promising. Prior to chemical treatment thorough mechanical cleaning by loosening the fibre, hand-sorting, and dusting is essential. The NaOH and two-stage sulphite-alkali processes of digestion are satisfactory as regards both the quality of the product and the yield. In semi-commercial-scale tests the yield of air-dried unbleached pulp from the fibre was 51—78%, the bleaching powder requirement was 12—20%, and the ratio of bursting strength to wt. for paper averaging 57 lb. per 500 sheets (25 in.  $\times$  40 in.) was 66.4—77.3% (91.3—104.5%) for bleached (unbleached) pulp, the sheet thickness being 0.004 in.

H. F. GILLBE.

See also A., June, 683,  $p_H$  stability region of insol. proteins. 716, Hydrolysis of cellulose. 717, Cellulose nitric esters.

## PATENTS.

**Felting [of fur and the like].** C. D. PARKS (U.S.P. 1,782,764—5, 25.11.30. Appl., 9.4.25. Renewed 14.2.30).—The material is treated with NaOH ( $d$  1.007—1.074) alone (B) or in admixture with  $H_2O_2$  (A). After drying, the fur fibres are removed from the skins, formed into hat shapes, and washed with dil. acid, e.g., 0.033% formic acid.

D. J. NORMAN.

**Manufacture of felted fibrous products.** H. L. LEVIN, Assr. to FLINTKOTE Co. (U.S.P. 1,782,855, 25.11.30. Appl., 11.2.28).—An aq. dispersion of a bituminous waterproofing substance is added to an aq. fibrous stock and the whole treated with a fixing agent (Na resinate or silicate and alum), the  $p_H$  of the mixture being adjusted to approx. that of the fixing agent at the isoelectric point; the mixture is then forced against a foraminous mould, and the formed article is removed and dried.

F. R. ENNOS.

**Cellulosic product.** E. A. VAZQUEZ, Assr. to VAZCANE PROCESS, INC. (U.S.P. 1,782,751, 25.11.30. Appl., 3.4.28. Cuba, 2.6.27. Cf. B., 1928, 940).—Sugar cane is ground or abraded to separate the fibres and disrupt the sugar cells, and then repeatedly extracted with  $H_2O$  in countercurrent until all the sugar is removed. The fibrous pulp remaining is suitable for paper or fibre-board manufacture.

F. R. ENNOS.

**Manufacture of plastic articles from artificial wood pulp.** K. KÜRSCHNER (B.P. 347,807, 4.3.30. Ger., 4.3.29).—A mixture of pulverised wood or like material with a dissolved compound of cellulose (cellulose xanthate, cuprammonium cellulose, nitrocellulose) is moulded under pressure and treated (e.g., with dil. alkali or alkali sulphide) to regenerate the cellulose.

F. R. ENNOS.

**Manufacture of soluble cellulose esters.** I. G. FARBENIND. A.-G. (B.P. 347,451, 4.4.30. Ger., 4.4.29. Addn. to B.P. 283,181; B., 1928, 637).—The process of the prior patent is extended to include esters prepared from halides of saturated or unsaturated heterocyclic carboxylic acids or heterocyclic substituted fatty acids, e.g., the carboxylic acids of furan, coumarone, indole, pyridine, pyrazine, etc. and their derivatives. Esters containing N in the ring form salts with acids and additive products with, e.g., alkyl halides. By suitable selection of the heterocyclic radical, pharmaceutical or bactericidal properties may be imparted to the resulting ester. The preparation of cellulose isonicotinic acid is described.

D. J. NORMAN.

**Manufacture and treatment of cellulose esters.** U.S. INDUSTRIAL ALCOHOL Co. (B.P. 346,430, 8.1.30. U.S., 30.4.29. Addn. to B.P. 306,531; B., 1930, 609).—Organic esters of cellulose are dissolved or dispersed in liquid  $SO_2$ , or esters of acids other than  $AcOH$  are produced in the liquid  $SO_2$  as medium; the liquid is then discharged into a precipitating medium so that the  $SO_2$  is suddenly gasified, causing disruption of the precipitated esters, to a light, fluffy, product.

F. R. ENNOS.

**Manufacture of alkyl and aralkyl derivatives of cellulose.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,426, 4.1.30).—Cellulose, cellulose

hydrate, or hydrocellulose is treated in one stage with caustic alkali solution and an alkylating or aralkylating agent in the presence of solid NaOH, an indifferent salt (NaCl), and an indifferent solvent or diluent ( $C_6H_6$ ), the wt. of  $H_2O$  present in the reaction mixture not exceeding that of the cellulose. F. R. ENNOS.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 347,926, 29.5.30. Switz., 27.7.29. Addn. to B.P. 342,167; B., 1931, 343).—The treated material described in the prior patent is further treated with a compound containing an NH, OH, or SH group, or with a salt of such a compound. The product, while still immunised to direct cotton dyes, has its affinity for basic dyes more or less replaced by an affinity for acid dyes. F. R. ENNOS.

**Esterification of cellulosic materials.** BRIT. CELANESE, LTD. (B.P. 346,292, 6.1.30. U.S., 4.1.29).—Cellulose is agitated in a pretreating chamber, *e.g.*, with AcOH, and is then discharged directly and preferably gradually into the esterification vessel. F. R. ENNOS.

**Manufacture of cellulose nitro-acetates.** I. G. FARBERIND. A.-G. (B.P. 344,761, 11.7.30. Ger., 11.7.29. Addn. to B.P. 319,285; B., 1931, 343).—By adding urea in suitable amount to the acetylating mixture, nitro-acetates containing < 0.1% N may be produced. H. ROYAL-DAWSON.

**Manufacture of cellulose ether nitrates.** DEUTS. CELLULOID-FABR. (B.P. 347,423, 25.3.30. Ger., 25.3.29).—An alkyl ether of cellulose containing at least 2 ether groups per mol. of  $C_6H_{10}O_5$  and free OH groups, *e.g.*, diethylcellulose (100 pts.), is treated with diacetyl-orthonitric acid (400 pts.) and a dehydrating agent, *e.g.*, a mixture of  $Ac_2O$  (25 pts.) and glacial AcOH (25 pts.) at  $10^\circ$ . After 10 min. the product is precipitated with  $H_2O$  and washed. The resulting ether ester is stable, sol. in many org. solvents and solvent mixtures, and insol. in  $H_2O$ . D. J. NORMAN.

**Production of nitrocellulose.** H. C. HEIDE. From E. TSCHUDIN (B.P. 347,169, 14.12.29).—The nitrating acid used in the process of B.P. 323,019 (B., 1930, 184) may contain considerably less  $HNO_3$ , *e.g.*,  $HNO_3$  37%,  $H_2SO_4$  57%,  $H_2O$  6%, if the partly gelatinised cellulose is allowed to retain 10–20%  $H_2O$ . In this case the final washing is preferably effected at  $60^\circ$ . Alternatively, the material may be washed with  $HNO_3$  before nitration. D. J. NORMAN.

**Nitrocellulose compositions.** G. H. PADGHAM (B.P. 347,597, 23.1.30).—A stable film of plasticised nitrocellulose is produced by mixing a solution of collodion and stearate with one consisting of "Neutroleum" (cyclohexyl acetate and 5% of a 1:1 mixture of  $AcOBu$  and butylcellulose) and pale mineral oil, and allowing to dry. F. R. ENNOS.

**Manufacture of mixed ethers of cellulose.** I. G. FARBERIND. A.-G. (B.P. 345,028, 30.1.30. Ger., 25.2.29).—Ethylene oxides accelerate the alkylation or benzylation of alkali-cellulose, and a mixed alkyl (or benzyl) hydroxyethyl ether of cellulose is produced. Examples are: 6 mols. of  $CH_3PhCl$  with 1 mol. of ethylene oxide, or 1.5 mols. of butylene oxide, heated with alkali-

cellulose at  $105$ – $110^\circ$ ; 20 mols. of  $PrCl$  and 3 mols. of ethylene oxide similarly at  $120^\circ$ . The products are sol. in  $C_6H_6$ . C. HOLLINS.

**Treatment of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 347,257, 22.1.30. U.S., 22.1.29. Addn. to B.P. 341,890; B., 1931, 395).—Cellulose esters purified by the process of the prior patent may be used in the manufacture of films etc. D. J. NORMAN.

**Manufacture of films containing cellulose acetate.** H. J. HANDS (B.P. 346,339, 30.12.29).—A mixed aryl phosphate containing a single naphthyl group, *e.g.*, diphenyl  $\beta$ -naphthyl phosphate, phenyl *m*-tolyl  $\beta$ -naphthyl phosphate, is used as plasticiser. F. R. ENNOS.

**Apparatus for manufacture of artificial silk.** H. F. NIOGRET, Assr. to DU PONT RAYON Co. (U.S.P. 1,782,581, 25.11.30. Appl., 14.4.28. Fr., 14.4.27).—Solutions of cellulose derivatives in a volatile solvent are dry-spun in a closed heated cell provided with one or more cooled condensers, each of which is directly connected to the cell by a single large aperture. F. R. ENNOS.

**Manufacture of artificial fibres.** HEBERLEIN & Co. A.-G. (B.P. 347,810, 7.3.30. Ger., 7.3.29).—Cuprammonium solutions of cellulose are spun, with or without preliminary coagulation in a known manner, in a precipitating bath of caustic alkali of mercerising concentration below  $0^\circ$ . F. R. ENNOS.

**Manufacture of artificial threads.** NOVASETA A.-G. ARBON (B.P. 347,878, 28.4.30. Switz., 11.7.29).—In the wet-stretch-spinning process the thread is passed substantially vertically first through a preliminary coagulating liquid, and then through one or more coagulating liquids of different composition from the first, each vessel containing coagulating liquid being so arranged with respect to the outlet of the preceding one that liquid flowing through this outlet does not enter the further vessels. F. R. ENNOS.

**Production of artificial filaments, yarns, or threads.** BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and W. I. TAYLOR (B.P. 346,354, 8.1.30. Addn. to B.P. 321,762; B., 1930, 98).—A systematic variation in denier is imparted to the threads by varying the degree of stretching, various mechanical means for which are described. F. R. ENNOS.

**Manufacture of diazotisable fibres.** C. GRÄNACHER (B.P. 346,385, 30.12.29. Switz., 28.12.28).—Fibres of cotton, regenerated cellulose, or partly acetylated cellulose, after being brought into a reactive condition if necessary, *e.g.*, by swelling with alkali and washing with water, are treated at about  $100^\circ$  with a nitrated benzyl halide, which may be undiluted, in an indifferent solvent or in aq. suspension, in the presence of an acid-binding agent other than caustic alkali, *e.g.*,  $CaCO_3$ ,  $Na_2CO_3$ ; the nitrobenzylated fibres are afterwards reduced to the  $NH_2$  derivative. F. R. ENNOS.

**Manufacture of paper.** MEAD RES. ENG. Co., Assees. of J. TRAQUAIR (B.P. 347,096, 7.12.29. U.S., 29.12.28).—Straw, bamboo, or similar cellulosic material is steeped for about  $\frac{1}{2}$  hr. in a large excess of a 1–6% solution of NaOH at about  $90^\circ$ , squeezed until it contains

3–4 pts. of liquor per 1 pt. of raw material, and digested, without further addition of treating liquor, for  $\frac{1}{2}$  hr. at 170–190°. The digester is heated by direct steam and the time taken to reach the cooking temp. should be about  $\frac{1}{2}$  hr. The resulting pulp is washed and chlorinated by the processes of B.P. 339,333–4 and 339,599 (B., 1931, 344). The process is continuous. Suitable apparatus is described.

D. J. NORMAN.

**Wood-pulp material.** J. H. WALLACE, ASSR. to PINE WASTE PRODUCTS, INC. (U.S.P. 1,781,712, 18.11.30. Appl., 5.2.25).—A more uniform pulp is obtained if the chips are first treated with steam in the presence of hot waste furnace gases, to bring them to a substantially uniform moisture content. Suitable apparatus is described.

D. J. NORMAN.

**Production of paper pulp.** R. B. WOLF (U.S.P. 1,780,638, 4.11.30. Appl., 23.12.29).—For the recovery of acid in the sulphite-pulp process, the gases and liquids from the digesters are cooled by passing through a relief line and an accumulator in which a constant pressure is maintained, whence they are discharged into an acid absorbent bath which is cooled to a predetermined temp. and maintained under pressure.

F. R. ENNOS.

**Manufacture of fibrous sheet material.** J. C. PEABODY and A. BROWN, ASSRS. to F. B. HOPEWELL (U.S.P. 1,781,668, 11.11.30. Appl., 23.7.29).—Cellulosic material, particularly wood pulp, is partly xanthated so that it retains its fibrous structure and is then beaten either alone or in admixture with, e.g., an equal quantity of untreated pulp and made into sheets. The resulting material is strong, does not disintegrate when immersed in  $H_2O$ , and is suitable as a base for artificial leather. Softening agents such as glycerin may be incorporated. Xanthation is conveniently effected by impregnating the pulp (100 pts.) with 10% NaOH solution at 15–32° for 1 hr., pressing the mass to 300 pts., and treating it for about 1 hr. with 25 pts. of  $CS_2$ . (Cf. B.P. 126,174; B., 1919, 458 A.)

D. J. NORMAN.

**Manufacture of fibre board.** E. S. EDWARDS, ASSR. to PANELYTE CORP. (U.S.P. 1,778,147, 14.10.30. Appl., 16.5.29).—Kraft pulp, fusible asphaltic material (gilsonite), red gum or resin, calcium chloride, and borax are beaten together with water, and the mixture is pressed and dried.

F. R. ENNOS.

**Manufacture of articles with surfaces having a pearl effect.** F. K. WICKEL (B.P. 347,888, 3.5.30).—Paper, leather, wood, etc. which has been coated with a suspension of powdered mica in an adhesive material and, if desired, with multicoloured patterns, is further coated with a transparent optically inactive layer (resin, albuminous substances, cellulose derivatives) and rubber with  $BiOCl$  or  $SbOCl$  to produce a "metallic mirror," through which the base material shows, thus producing the desired effect.

F. R. ENNOS.

**Machines for decorticating fibre-containing foliate plants.** F. KRUPP GRUSONWERK A.-G. (B.P. 349,343, 19.6.30. Ger., 13.5.30).

**Manufacture of crêpe-like fabrics.** BRIT. CELANESE, LTD. (B.P. 349,121, 17.2.30. U.S., 15.2.29).

**[Wet-stretching] treatment of artificial silk threads.** F. J. GAHLERT (B.P. 349,232, 10.4.30. Ger., 10.4.29).

**Manufacture of [linen-surfaced] waxed papers.** WAXED-PAPERS, LTD., and W. T. CLOUT (B.P. 349,104, 10.1.30).

**Manufacture of [toilet] paper or paper-like material.** ROBINSON & SONS, LTD., and V. O. ROBINSON (B.P. 349,139, 28.2.30).

**Storing and mixing materials [paper pulp].—**See I. Glass. **Transparent sheets.**—See VIII. **Treatment of surfaces. Fibrous compositions. Plastic masses. Adhesive.**—See XIII. **Absorbent for liquid O.**—See XXII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Influence of electrolytes of different valencies on dyeing with substantive dyes.** P. P. VIKTOROV (Kolloid-Z., 1931, 55, 72–81).—The influence of electrolytes on the adsorption of the substantive dye "Benzoreinblau" has been studied. The electrolytes used can be arranged in the following order of effectiveness of cations, the sulphate being used in each case:  $NH_4^+ < Na^+ < K^+ < Mg^{2+} < Ni^{2+} < Mn^{2+} < Zn^{2+} < Cd^{2+} < Al^{3+}$ . Greater amounts are adsorbed the higher is the valency of the cation. Little adsorption occurred with the dialysed dye, the addition of  $Na_2SO_4$  producing a tenfold increase, whilst  $Al_2(SO_4)_3$  causes an increase of a further 30%. Anomalies were noted in the effects of  $Cu^{2+}$ ,  $Cr^{3+}$ , and  $Fe^{3+}$  sulphates, where a different colour was given. The addition of gelatin increases the amount of dye taken up.

E. S. HEDGES.

**Action of certain [organic] acids on cellulose.** E. HIBBERT (J. Soc. Dyers and Col., 1930, 46, 294–295).—The tendering effect of drying a solution of oxalic acid in cotton fabric is very much accelerated by simultaneous exposure of the fabric to sunlight; the tendering commences when the fabric becomes dry. If fabric be dried with oxalic acid under conditions such that inappreciable tendering occurs and the fabric be then exposed to sunlight or light from a Fadeometer, marked tendering occurs so that the fabric has a decreased affinity for direct dyes, an increased affinity for methylene-blue, and strong reducing power which can be detected by its high Cu number and reactivity towards *p*-nitrophenylhydrazine and Ermen's solution. A similar but less marked effect is obtained when other org. acids such as phthalic, tannic, and gallic acids are used; phthalic acid which is formed during the fading of cotton dyed with Purpurin (B., 1929, 715) produces definite tendering although less than that of oxalic acid. Tendering is also produced when an org. acid is dried into fabric which is then quickly ironed at 170°; the tendering is greatest with oxalic acid and less with tartaric, malic, citric, and phthalic acids, and viscose is much more readily tendered than cotton.

A. J. HALL.

**Action of caustic soda on cellulose.**—See V. **Laundry materials.**—See VII.

See also A., June, 717, Action of  $CH_2O$  on cellulose.

## PATENTS.

**Bleaching of [wood] pulp.** D. C. ANDREWS, Assr. to NORTHWEST PAPER Co. (U.S.P. 1,782,800, 25.11.30. Appl., 23.6.26).—Wood pulp is bleached, in the form of a 10–20% slurry, in a wooden chamber provided with a false bottom. An alkaline solution of  $\text{CaOCl}_2$  is distributed over the slurry so that it percolates downwards and thereby displaces the  $\text{H}_2\text{O}$  present; the bleach liquor is then recirculated through the slurry until it is fully decolorised.

A. J. HALL.

**Immunisation of cotton and other cellulosic fibres.** CHEM. FABR. VORM. SANDOZ (B.P. 345,052, 19.2.30. Ger., 20.2.29).—In the alkalisation of cellulose etc. for immunising by esterification, the use of boiling dil. (12–14%) caustic alkali results in a more even product, especially in presence of phenols or cyclic alcohols, *e.g.*, cresol, cyclohexanol, which assist wetting.

C. HOLLINS.

**Dyeing or colouring of cellulose ethers and esters and materials containing them.** SOC. CHEM. IND. IN BASLE (B.P. 344,884, 9.12.29. Switz., 8.12.28).—Greenish-yellow shades are obtained on cellulose ethers or esters in textiles or varnishes by use of the dyes *p*-alkoxyaniline  $\rightarrow$  *p*-cresol. [Stat. ref.]

C. HOLLINS.

**Dyeing and printing with [soluble esters of leuco]-vat dyes.** DURAND & HUGENIN SOC. ANON. (B.P. 344,964, 23.12.29. Ger., 22.12.28).—Over-oxidation, even with excess of oxidant, in development of sol. esters of leuco-vat dyes, is prevented by addition of carboxylic or sulphonic acids of arylamines, *e.g.*, sulphanilic or diethylmetanilic acid, preferably as salts.

C. HOLLINS.

**Decorative treatment of textile fabrics.** CALICO PRINTERS' ASSOC., LTD., J. D. WEBSTER, and J. S. HEATON (B.P. 346,544, 13.3.30. Addn. to B.P. 326,823; B., 1930, 554).—Materials producing two or more different colours are used in the process of the prior patent. *E.g.*, fabric is prepared with Naphthol-AS, then printed "bang-through" with a mixture of Caledon Jade Green and Fast Scarlet R salt on one roller and a mixture of Indanthrene Brilliant Violet 4R and Fast Orange R salt on the other, and finally printed on one side with alkaline reducing paste. After steaming etc., the pattern appears in green and violet on one side of the fabric and in red and orange on the other.

C. HOLLINS.

**Manufacture of crimped artificial fibres and threads.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,799, 6.12.29).—Hardened threads or strips of the polymerisation products of butadiene hydrocarbons are crimped by immersion in organic swelling agents such as solutions or emulsions of cyclohexane,  $\text{C}_6\text{H}_6$ , ethers, esters, ketones, etc.

A. J. HALL.

**Degreasing of textile materials.** W. PFAFFENDORF, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,885, 4.11.30. Appl., 26.5.27. Ger., 14.6.26).—The materials are washed with ethylene chloride at 0–80°.

H. ROYAL-DAWSON.

**Sizing of textile materials.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 346,267, 29.11.29).—Textiles, especially cellulose acetate materials, are impregnated with polymerised or partly polymerised

vinyl acetate (etc.) preferably dissolved in MeOH or  $\text{C}_6\text{H}_6$ . Polymerisation may also be effected on the fibre.

C. HOLLINS.

**Rubber-coated fabrics.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 347,826, 17.3.30).—Rubber-coated fabric suitable for automobile tops or upholstery is provided with an external protective layer by applying, before vulcanisation, a coat of asphalt varnish, after which vulcanisation is effected at a temp. which causes the asphalt to fuse to a smooth, chemically inert film. An improved result is obtained if a coat of oil varnish is applied before the asphalt varnish. The asphalt varnish itself may contain  $2\frac{1}{2}$ –20 gal. of drying oil per 100 lb. of asphalt.

D. F. TWISS.

**Cloth fulling and washing machine.** D. GESSNER (B.P. 349,223, 7.4.30).

**Dye-works.**—See I. Detergents.—See III. Diazo-tisable fibres. Cellulose derivatives.—See V. Treatment of surfaces.—See XIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Intensive [chamber] process for the manufacture of sulphuric acid.** E. OVSIANY (Chim. et Ind., 1931, 25, 832).—The author's apparatus, which is not described, effects an intimate contact of atomised liquid and gas under alternate pressure and expansion. Two forms of apparatus have been tested as an auxiliary in front of the two leading chambers of a set and gave a yield per unit space of 270–400 times the yield of an ordinary chamber. Applications to other gas-washing processes are suggested.

C. IRWIN.

**Production of phosphoric acid from phosphorites by volatilisation in an atmosphere of gaseous hydrocarbons.** C. PADOVANI and A. NARDELLA (Annali Chim. Appl., 1931, 21, 109–115).—A Land Pebble phosphorite containing  $\text{P}_2\text{O}_5$  30.59,  $\text{SiO}_2$  6.60,  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  2.36% was mixed with sufficient  $\text{SiO}_2$  to give the  $\text{P}_2\text{O}_5 : \text{SiO}_2$  ratio the value about 1 : 2 and heated at 1150–1200° in a current of natural gas composed largely of  $\text{CH}_4$ . More than 90% of the total P present was volatilised, all the H produced by the scission of the  $\text{CII}_4$  and about one half of the C (as CO) being recovered.

T. H. POPE.

**Ammoniation of superphosphate with anhydrous ammonia.** F. G. KEENEN (Amer. Fert., 1931, 74, [10], 20–22).—A description of the commercial process and of the material produced.

A. G. POLLARD.

**Limitation in capacity of a platinum catalyst for ammonia oxidation.** H. W. WEBB (J.S.C.I., 1931, 50, 128–131 r).—It is shown that the max. point in the curve connecting the yield of NO with the gas velocity in  $\text{NH}_3$  oxidation is mainly due to the reaction between  $\text{NH}_3$  and NO. Mixtures of these gases in equimol. proportions are found to explode in contact with activated Pt at 800°. The spacing apart of Pt gauze catalysts in  $\text{NH}_3$  oxidation is shown to be inadvisable.

**Value of volumetric methods for determining the basic constituents in quicklime and slaked**



lime. F. MACH and R. HERRMANN (Z. anal. Chem., 1931, 84, 1—14). The Mg and Ca silicates which are found in CaO and slaked lime are of very variable composition and dissolve to varying extents in all the solutions used in the analysis; in weak organic acids and solutions of sugars or of glycerin the solubility is relatively small but even conc. HCl does not decompose them completely. All attempts to effect a separation of the CaO and MgO from the silicates and carbonates by differential dissolution failed to give good results; decomp. by HCl followed by complete analysis gave results of sufficient accuracy for technical purposes.

A. R. POWELL.

**Commercial calcium nitrate [alleged fire risks in handling].** C. MATIGNON (Chim. et Ind., 1931, 25, 799—802, and Compt. rend., 1931, 192, 777—780).—Norwegian Ca nitrate, containing 13% N and 22—23% H<sub>2</sub>O, and the "ammoniacal" product of the Société Badoise, containing 5% NH<sub>4</sub>NO<sub>3</sub> and 14% H<sub>2</sub>O, have been examined. Both products are hygroscopic and their decomposition is endothermic, requiring a temp. above 500°. Intimate mixtures of Ca(NO<sub>3</sub>)<sub>2</sub> with powdered charcoal, sawdust, and jute from the bags in which it is packed failed to ignite below 300°. No record (since 1903) can be traced of a case of spontaneous ignition due to the commercial product. C. IRWIN.

**Detection of sodium silicate in laundry materials.** J. J. J. DINGEMANS (Chem. Weekblad, 1931, 28, 229, 277—278).—A sample (1 g.), in aq. solution, after acidification with HCl is filtered and evaporated and after cooling, 10 c.c. of a 10% (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> solution are added; the appearance of a yellow coloration indicates the presence of silicate, which is confirmed by the production of a blue coloration on addition of a strongly alkaline stannate solution. The presence of bleaching agents interferes, and in their presence SiO<sub>2</sub> must be sought in the ash. In laundry materials containing phosphate and bleaching agents, the phosphate is removed by treating the solution with magnesia mixture, and bleaching agents by boiling in alcoholic solution. SiO<sub>2</sub> may then be detected as described above.

H. F. GILLBE.

**Gravimetric determination of lead in solution of lead subacetate [U.S.P.].** J. L. MAYER (J. Amer. Pharm. Assoc., 1931, 20, 363—364).—10 c.c. of a solution of 5 g. of the subacetate in 50 c.c. of H<sub>2</sub>O and 5 drops of HNO<sub>3</sub> are diluted to 300 c.c. and heated to 90—95°; aq. NH<sub>3</sub> is added to incipient precipitation and then a slight excess of 10% HNO<sub>3</sub>. To the hot solution 50 c.c. of hot 10% K<sub>2</sub>CrO<sub>4</sub> are added with stirring over a period of 40 sec. The ppt. is filtered off and dried at 140—150°; wt.  $\times 0.6410 = \% \text{ Pb}$ .

E. H. SHARPLES.

**Lead in basic bismuth nitrate.** N. SCHOORL (Pharm. Weekblad, 1931, 68, 277—279).—The usual pharmacopœia test for absence of Pb—a clear solution with 0.5 g. in 2 c.c. H<sub>2</sub>SO<sub>4</sub>—gives a positive result if carried out at room temp. if the Pb content exceeds 0.1%. At higher temp. a ppt. of basic Bi sulphate is formed at once; even in the cold, this ppt. forms after 24 hr.

S. I. LEVY.

**Alkali bismuth tartrates from the chemical and biological standpoint.** A. E. JURIST and W. G.

CHRISTIANSEN (J. Amer. Pharm. Assoc., 1931, 20, 349—355).—The behaviour of these salts in aq. solution containing stabilising substances, and experiments on the toxicity, adsorption, and local action of the compounds when injected intramuscularly are described. The most stable solutions are prepared using glycerin or, preferably, for biological use, 25% sucrose solution suitably buffered. The alkali Bi tartrates when injected cause severe local reactions, including pain, swelling, and lump formation, and though they have low toxicity and are readily absorbed they are not satisfactory therapeutic agents for syphilis. The local reactions could not be materially reduced by modifying the aq. solvent. E. H. SHARPLES.

**Preparation of water-soluble sodium bismuth citrate.** W. F. VON OETTINGEN (J. Amer. Pharm. Assoc., 1931, 20, 426—429).—Freshly prepared, moist Bi(OH)<sub>3</sub> when shaken with 0.5M-citric acid forms dibismuthyl-citric acid, which is dissolved in NaOH and the pure monosodium dibismuthylcitrate precipitated by addition of EtOH. Details are given (cf. A., 1927, 1109).

E. H. SHARPLES.

**Compounds of titanium with glycol and phthalic acid, and ethyl ortho-silicate and -titanate as film-forming materials.** H. A. GARDNER and E. BIELOUSS (Amer. Paint and Varnish Manufs.' Assoc., 1930, Circ. 366, 327—337).—Indefinite products, apparently of orthosilicate type, obtained by heating TiCl<sub>4</sub> with "pentasol," ethylene glycol, and glycerol until HCl ceases to be evolved, and by the action of phthalic anhydride in 50% EtOH on Ti(OEt)<sub>4</sub> followed by glycerol at the b.p., are described. The glycerol and glycol esters still contain Cl and are sol. in H<sub>2</sub>O, with hydrolysis in the latter case to a colloidal Ti(OH)<sub>4</sub> solution stable at 130—140°; the former forms films sol. in H<sub>2</sub>O, but the latter produces good films on a variety of surfaces and is compatible with cellulose esters. The glycol ester in contact with nitrocellulose in AcOBu produces a white Ti pigment in a finely-dispersed state, and gives a white ppt. with rubber latex, as also does TiCl<sub>4</sub>. Films of good adhesive strength were not obtained with Ti(OEt)<sub>4</sub> or Si(OEt)<sub>4</sub>. Ti(OEt)<sub>4</sub> is useless as an anti-knock agent.

H. A. PIGGOTT.

**Absorption and regeneration of hydrogen sulphide by alkaline liquids.** H. A. J. PIETERS and G. SMEETS (Chem. Weekblad, 1931, 28, 246—249).—The efficacy of Na<sub>2</sub>CO<sub>3</sub> solution for the removal of H<sub>2</sub>S from coal gas increases with increase of concentration up to about 3%; 1 vol. of the solution is sufficient to reduce the sulphide content of 3 vols. of gas containing 0.5% H<sub>2</sub>S to 90% of its initial value. The resulting solution does not evolve H<sub>2</sub>S if CO<sub>2</sub>-free air is passed through it, but by treatment with a rapid current of air it is partly regenerated, most of the S being evolved as H<sub>2</sub>S and the remainder oxidised to thiosulphate. Bicarbonate solutions are not satisfactory for the washing process, saturated NaHCO<sub>3</sub> solution being only about one half as effective as 3% Na<sub>2</sub>CO<sub>3</sub> solution. There is no advantage in using K<sub>2</sub>CO<sub>3</sub>. HCN is absorbed, and is converted into M'CNO and M'CNS during regeneration.

H. F. GILLBE.

**Acid pumps.**—See I. Acetate from soda-pulp liquors.—See V. Anglesite.—See VIII. S-resistant

alloys.—See X. Photosensitised oxidation of  $\text{NH}_3$  and  $\text{NH}_4$  salts.—See XVI.

See also A., June, 677, Adsorption of H on charcoal. 678, Sorption of H by Pt metals. 695, Ca oxalate from Ca cyanamide. 696, Phosphorescent boric acid. 697, Iodine residues. 698, Specifications for reagents. 699, Determination of  $\text{H}_2\text{O}_2$ . Test for halogens. Bromo-iodometry. 701, Separation of Ca and Mg by molybdate. 718—9, Preparation of organo-Mg halides. 775, I in algæ.

#### PATENTS.

**Manufacture of strontium oxide.** J. B. PIERCE, JUN., ASSR. to BARIUM REDUCTION CORP. (U.S.P. 1,782,830, 25.11.30. Appl., 29.1.27).—Amorphous  $\text{SrCO}_3$ , prepared by precipitating a solution of an alkaline Sr compound with  $\text{CO}_2$  or a carbonate, is mixed with ashless C black, and the mixture calcined in a closed muffle at  $1200^\circ$ . The easily oxidisable SrO produced may be used for preparing  $\text{SrO}_2$ . W. J. WRIGHT.

**Production of anhydrous chlorides.** SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 344,601, 18.1.30. Fr., 18.1.29).—Chlorides of Mg, Ca, Th, etc. which have been partly dehydrated and thereby rendered porous are dehydrated by treatment in an externally-heated vertical retort, in a countercurrent of HCl at a temp. below the softening point, e.g.,  $400$ – $450^\circ$  at the top of the tower and  $580$ – $620^\circ$  at the hottest point. A product is claimed containing  $\text{CaCl}_2$  0.05–0.5% and insol. material  $< 0.5\%$ . L. A. COLES.

**Cleansing agents [for metals etc.].** CHEM. FABR. J. A. BENCKISER G.M.B.H., and F. DRAISBACH (B.P. 347,072, 25.7.30. Ger., 23.4.30).—Up to 20% of  $\text{Al}_2(\text{SO}_4)_3$  is added to  $\text{Na}_3\text{PO}_4$  or to a mixture of that compound with soda. H. ROYAL-DAWSON.

**Production of compositions containing compounds of alginic acid and product thereof.** L. L. BURGESS, ASSR. to PLASTIC, INC. (U.S.P. 1,782,887, 25.11.30. Appl., 14.7.28).—Alginic acid or a composition containing it, dissolved in an alkaline reagent, is treated with a solution of a hydrocarbon, e.g., paraffin wax in  $\text{CCl}_4$ , and the product is precipitated by addition of a mineral acid. Finely-divided Cu or Zn may be added to the reaction mixture, precipitation then occurring after several hr. without addition of acid. The washed and dried ppt. is extracted with  $\text{CCl}_4$ , and the product plasticised with aq.  $\text{NH}_3$  and rendered insol. by addition of a suitable metal oxide. W. J. WRIGHT.

**Treatment of dolomite and other natural carbonates of magnesium.** SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 348,758, 10.3.30. Fr., 11.3.29).—See U.S.P. 1,778,659; B., 1931, 539.

**Manufacture of cakes of carbonic snow.** H. FIEVET (B.P. 348,581, 13.2.30).

**N-H mixture.**—See II. Lute.—See IX. Mixed fertilisers.—See XVI. Absorbent [for liquid O]. Liquid-O explosives.—See XXII.

#### VIII.—GLASS; CERAMICS.

**Continuous kilns as applied to the pottery industry.** R. WHITFIELD (Trans. Ceram. Soc., 1931, 30, 150–168).—The gas-fired “Robertson” tunnel

kiln and an electrically heated decorating kiln are described in detail. The former has been applied to all types of ceramic ware, including that produced by salt-glazing. Close control of atm. heating and cooling rates etc. has been provided. The thermal efficiency is very high. J. A. SUGDEN.

**Continuous kilns for the burning of clay products.** J. WILLIAMSON (Trans. Ceram. Soc., 1931, 30, 21–32).—Descriptions are given of the different types of continuous kilns, including the “Manchester,” “Staffordshire,” “Ideal,” “Simplex,” and the various tunnel kilns, and their adaptability to the firing of clay wares is indicated. F. SALT.

**Alkali-lime-silica glasses.** G. KEPPELER and K. BÖHMERT (Keram. Rund., 1930, 38, 663–666, 697–700, 711–713, 738–742, 787–788; Chem. Zentr., 1931, i, 1341).—Devitrification limits, m.p., sp. gr., and extraction values of K glasses were determined. For similar mol. composition Na glasses are better; except with glasses high in K or Ca, for similar wt. composition K glasses are the better. Na-K-Ca-SiO<sub>2</sub> glasses showed no extraction min. contrary to Peddle's result, which is attributed to devitrification. A. A. ELDRIDGE.

**Mercury glasses.** F. H. ZSCHACKE (Glastech. Ber., 1930, 8, 519–525; Chem. Zentr., 1931, i, 1341).—When  $\text{HgCO}_3$  is added to the batch only a vanishingly small trace remains in the glass. The colour is golden-yellow to bluish-green, depending on the temp. and atm., and is attributed to colloidal Hg. Spectroscopic observations are recorded. A. A. ELDRIDGE.

**Sulphur trioxide content of glass. Clarification.** H. JEBSEN-MARWEDEL and A. BECKER (Glastech. Ber., 1930, 8, 525–529; Chem. Zentr., 1931, i, 1340–1341).—The effect of various furnace atm. on the removal of  $\text{SO}_3$  from window glass was examined. Vigorous reduction at  $1350^\circ$  removes the  $\text{SO}_3$ , whilst in an oxidising atm. a temp. of  $1500^\circ$  is necessary. The gradient of the removal is not const. for a particular temp., but appears to depend on the rate of heating. A. A. ELDRIDGE.

**Permeability of glasses to ultra-violet radiation.** II. P. GILARD, P. SWINGS, and A. HAUTOT (Bull. Acad. roy. Belg., 1931, [v], 17, 362–368; cf. B., 1930, 557).—The transparency of glasses to ultra-violet light is diminished by addition of As, Sb, Cr, Co, Mn, and Fe oxides. The effect of NiO is generally in the same sense, but depends on the composition of the glass. C. W. GIBBY.

**Plastic flow measurements [in clay] and their bearing on the plasticity problem.** G. W. S. BLAIR (Trans. Ceram. Soc., 1931, 30, 138–149).—Measurements of the rate of flow of slips through a capillary-tube type of plastometer were made. The curve showing the relationship between the rate of flow and pressure may be divided into four regions: (1) no flow; (2) linear flow—the paste flows through the tube as a solid plug; (3) mixed flow—the paste has started to flow telescopically, but there is still a plug in the centre; (4) telescopic or stream-line flow as a true fluid. The intercept of region (4) on the pressure axis,  $a$ , is Bingham's “yield value,” and the point where region (3) begins,  $b$ , is the “critical shearing stress.” Unsuccessful attempts were made in a series of clays

to correlate the values of  $a$  and  $b$  with the plasticity as measured by Bischof's "wire method" (plasticity is inversely proportional to diam. of the thinnest wire that can be rolled without breaking). All these attempts were made at the same arbitrarily chosen  $H_2O$  content for all the clays. The conception of "flow plasticity" is developed by the author. The underlying idea is to compare the values of  $b$  at the same arbitrarily chosen value of  $a$  for all the clays, thus making the comparison at corresponding (but not identical) moisture contents when the hydration phenomena mask the variations in plasticity. This new conception enabled many clays to be placed in correct order of plasticity and the observation that the combined action of  $NaOH$  and  $NaCl$  on a clay has a much greater plasticising effect than the action of either alone was confirmed. Certain plastic-flow constants are shown to be systematically affected by the addition of  $CaCO_3$  and  $CaO$ . It is pointed out, however, that the flow-plasticity method must be used with caution, since the constants are somewhat sensitive to the previous moisture-history of the material. J. A. SUGDEN.

**Grog.** C. R. F. THRELFALL (Trans. Ceram. Soc., 1931, 30, 1—18).—Grog is defined as "any material present in a wet batch in a non-plastic or comparatively non-plastic form," and therefore includes both fired and unfired material. The term "green grog" is given to the nodules found in fired firebricks and caused by the coarser fractions in the green clay. The functions of grog, as thus defined, in refractory bodies are discussed in detail in relation to porosity, shrinkage, etc., and to the various processes of manufacture. F. SALT.

**Effects of various kiln gases on the burning of bright gold for gilding ceramic ware.** A. NAKATSUCHI (J. Soc. Chem. Ind., Japan, 1931, 34, 67—69B).—The results of burning "bright gold" in atm. of  $CO$ ,  $H_2$ ,  $SO_2$ ,  $H_2S$ ,  $CO_2$ ,  $O_2$ ,  $N_2$ , and  $H_2O$  vapour are reported. The first five gases are injurious; the best atm. is one of  $N_2$  with a little  $O_2$ , but this is impracticable. The suggested burning process for bright gold is to remove the injurious gases from the kiln at about  $300^\circ$  before raising the temp. to  $750^\circ$ . F. SALT.

**Moisture expansion of glazes and other ceramic finishes.** H. G. SCHURECHT and G. R. POLE (Bur. Stand. J. Res., 1931, 6, 457—463).—When subjected to artificial weathering by the action of steam at  $150\text{ lb./in.}^2$  for 1 hr., the average expansion of slip finishes was  $0.033\%$ , of matt glazes  $0.011\%$ , of vitreous slip  $0.005\%$ , and of lustrous glazes  $0.004\%$ . The necessity of considering this type of expansion when adapting a glaze or finish to a ceramic body is emphasised, since it is often of greater importance as regards the crazing of the glaze than is the elasticity or tensile strength. The ring test may be employed for comparative moisture expansion tests of ceramic finishes if the rings are all made from the same body and are treated in the same manner. H. F. GILLBE.

**Works' tests on refractories and service conditions.** R. J. SARJANT (Trans. Ceram. Soc., 1931, 30, 46—65).—Various tests for refractories are discussed in relation to their serviceability in steel works' practice. The refractoriness-under-load test has definite value in

the case of firebricks and monolithic refractories; results with this test, using a load of  $50\text{ lb./in.}^2$ , are given and discussed. Useful data are obtainable from the change in porosity and density on firing refractories to a moderately high temp. ( $1450^\circ$ ). Simulative slag-attack tests are useful, provided that they are classified according to the type of refractory and use. Spalling tests are of doubtful value, whereas thermal conductivity tests provide important information in relation to industrial practice. The service test remains the ultimate arbiter in the selection of refractories. F. SALT.

**Mode of formation of anglesite.** V. L. BILLET [with M. BEULCKE] (Natuurwetensch. Tijds., 1931, 13, 67—68).—Anglesite, identical with the natural mineral, has been found deposited on the walls of ovens in which Pb-glazed articles have been fired.

H. F. GILLBE.

See also A., June, 700, **Silicate analysis.** 707, **Doubrovka kaolin.** Clay from Tshasov-Jar deposits.

#### PATENTS.

**Synthetic oriental emerald or emerald-green sapphire.** E. G. SANDMEIER, Assr. to SWISS JEWEL Co. Soc. ANON. (U.S.P. 1,775,867, 16.9.30. Appl., 28.2.28. Switz., 19.1.28).—A mixture of oxides or salts to give the composition  $Al_2O_3$  98.6%,  $CoO$  0.986%,  $V_2O_5$  0.119%,  $NiO$  0.295%, when fused according to the Verneuil process (cf. U.S.P. 1,004,505; B., 1911, 1254), gives a synthetic gem resembling the oriental emerald. M. PARKIN.

**Synthetic spinels.** E. G. SANDMEIER, Assr. to SWISS JEWEL Co. Soc. ANON. (U.S.P. 1,775,868—1,775,870, 16.9.30. Appl., [A, B] 28.2.28, [C] 3.5.28. Switz., [A, B] 19.1.28).—(A) Green spinels are produced synthetically by fusing according to the Verneuil process (see preceding abstract) a mixture of oxides or salts to give the composition  $Al_2O_3$  82.53%,  $MgO$  16.506%,  $TiO_2$  0.066%,  $Cr_2O_3$  0.099%. (B) Aspinel like Brazilian emerald is similarly produced by melting a batch to give the composition  $Al_2O_3$  82.6%,  $MgO$  16.5%,  $CoO$  0.004%,  $V_2O_5$  0.04%,  $MnO$  0.83%. (C) A spinel resembling blue zircon results from a similar fusion giving the composition  $Al_2O_3$  86%,  $MgO$  10%,  $MnO$  3%, and traces of  $CoO$  and  $TiO_2$ . M. PARKIN.

**Splinterless glass.** BRIT. CELANESE, LTD. (B.P. 347,219, 22.1.30. U.S., 22.1.29).—The glass sheets and reinforcing layer comprising a cellulose derivative are heated at above  $50^\circ$  (preferably at  $100$ — $200^\circ$ ), separately or after assemblage, before they are united by compression. L. A. COLES.

**Manufacture of compound transparent [glass] sheets.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 345,176, 21.6.30. Fr., 22.6.29. Addn. to B.P. 316,955; B., 1930, 1029).—Safety glass assembled as described in the prior process without the use of volatile solvents is subsequently heated to improve the adhesion of the glass to the core, e.g., for 42 hr. at  $45^\circ$ , or 52 at  $65^\circ$  for materials assembled without de-gassing *in vacuo*, or for 4 hr. at  $85^\circ$  when they had been so de-gassed. M. PARKIN.

**Compound transparent sheets.** DUPLATE CORP., Assees. of J. H. SHERTS and R. E. HAMILL (B.P. 347,455,

7.4.30. U.S., 9.4.29).—Edge defects in composite sheets comprising glass and a pyroxylin plastic are cured or prevented by subjecting the finished sheets to further compression in the presence of a heated liquid, preferably a high-boiling solvent of the plastic.

L. A. COLES.

**Ceramic materials.** W. W. TRIGGS. From R. POLLAK (B.P. 347,486, 30.4.30).—Mixtures of hydrated Mg silicates free from alkali compounds (serpentine, chlorite, soapstone, etc., or artificial products) with not more than 10% of alkaline-earth compounds and  $\text{Al}_2\text{O}_3$ , with the addition of graphite if desired, are calcined for the production of refractory linings, stills, emery wheels, etc.

L. A. COLES.

**Manufacture of ceramic products.** V. M. GOLDSCHMIDT (U.S.P. 1,782,295, 18.11.30. Appl., 11.12.28. Ger., 18.8.27).—The raw materials include Mg hydro-silicates, *e.g.*, serpentine, with the exception of talcum, and other substances rich in Mg, *e.g.*, asbestos waste, these being heated to a temp. at which Mg orthosilicate is formed without fusion.

W. J. WRIGHT.

**Manufacture or burning of bricks and the like, and fuel for use therewith.** J. ONIONS (B.P. 347,266, 24.1.30).—Perforated tiles or blocks of fuel are interspaced with the bricks as they are set up in the kiln for burning.

L. A. COLES.

**Refractory structure.** F. L. ARENSBERG and A. J. JACKMAN, Assrs. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,782,023, 18.11.30. Appl., 8.2.29).—The tendency of refractory structures to spall is reduced if materials of different thermal conductivity are used, one predominating near one face and extending to a considerable depth, and the other, with a lower thermal conductivity and consequent greater susceptibility to spalling, forming the remainder of the structure and being protected by the former.

W. J. WRIGHT.

**Lehrs for annealing glassware.** UNITED GLASS BOTTLE MANUFRS., LTD., and T. C. MOORSHEAD (B.P. 348,872, 10.5.30).

**Manufacture of glass tubes and rods.** J. R. C. JORGENSEN, Assee. of L. BOSC (B.P. 348,857, 28.4.30. Austr., 6.11.29).

**Brick-making machinery.** C. WHITTAKER & Co., LTD., and N. WHITTAKER (B.P. 349,187, 15.3.30).

**Lute.**—See IX. Treatment of surfaces.—See XIII.

## IX.—BUILDING MATERIALS.

**Dissolution velocity of Portland cements.** T. YOSHIOKA and K. KUMAGAE (J. Soc. Chem. Ind., Japan, 1931, 34, 89B).—Dyckerhoff Doppel, Rapid-hardening, Super, Special, Velo, and Japanese normal cements were extracted with  $\text{H}_2\text{O}$  in the absence of  $\text{CO}_2$  and determinations of the electrical conductivity and alkalinity of the extracts were made. Cements of the Portland type show a common and characteristic time-reaction tendency of hydration. Cements of similar chemical composition are not necessarily similar in velocity of hydration, and marked differences are observed in the rate of increase of alkalinity of the solution. The concentration of CaO in the aq. phase increases with time to a saturation limit, but the time curve shows a

point of arrest which is common to all the cements examined.

E. S. HEDGES.

**Effect of free lime in Portland cement clinker on the solubility of lime and alumina.** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1931, 34, 56—58B).—In well-burned clinker, low in free CaO, the solubility of  $\text{Al}_2\text{O}_3$  is comparatively high, that of CaO low. With increasing free CaO content, the solubility of the  $\text{Al}_2\text{O}_3$  decreases, whilst that of the CaO increases rapidly. Addition of  $\text{H}_2\text{O}$  to clinker containing much free CaO produces a large quantity of a flocky gel substance, and formation of  $\text{Ca}(\text{OH})_2$  takes place much more quickly than with well-burned clinker.

F. SALT.

**Rôle of calcium hydroxide in the hardening of Portland cement.** F. F. TIPPMMANN (Kolloid-Z., 1931, 55, 85—107).—The setting and hardening processes of Portland cement are illustrated by photomicrographs. The needle-like crystals which are observed in the presence of an excess of  $\text{H}_2\text{O}$  are not Ca hydrogen silicate, as was formerly supposed, but consist of  $\text{Ca}(\text{OH})_2$ . They can be produced by the hydration of pure CaO in the presence of gypsum, which stimulates the crystallisation. Gypsum also assists in the transformation of amorphous into cryst.  $\text{Ca}(\text{OH})_2$ , and the hitherto unexplained effect of gypsum on the hardening of Portland cement is attributed to this cause. Crystalloidal and colloidal theories of the hardening of cement are reviewed. It is claimed that the process can be understood only by considering both the cryst. and colloidal hydrated products.

E. S. HEDGES.

**Hydration phenomena of cements.** T. YOSHIOKA, K. KUMAGAE, and H. TAKATA (J. Soc. Chem. Ind., Japan, 1931, 34, 87—88B).—Microscopical observations of the slow hydration of 5 types of cement in a limited excess of  $\text{H}_2\text{O}$  are recorded. Velo cement and blast-furnace cement were rich in needle crystals, whilst Special, Super, and normal Portland cements formed needle-like crystals during the initial course of the process, which later disappeared.

E. S. HEDGES.

**Swelling of cement grains on hydration.** T. YOSHIOKA and H. TAKATA (J. Soc. Chem. Ind., Japan, 1931, 34, 88B).—Grains of normal Portland cement undergo marked swelling during hydration, the original linear dimensions increasing by as much as 45%. Special, Super, and Velo cements undergo a total decrease of vol. when mixed with  $\text{H}_2\text{O}$ . Probably swelling occurs with these cements also, but vol. increase due to this cause is more than counterbalanced by the decrease due to dissolution of certain constituents of the grains.

E. S. HEDGES.

**Durability and strength of bond between mortar and brick.** L. A. PALMER and J. V. HALL, JUN. (Bur. Stand. J. Res., 1931, 6, 473—492).—The porosity of the brick has but little influence on the strength of the mortar bond. The principal factors affecting the durability, assuming sufficient wetting of the brick, are the pressure on the joint, freedom of movement during shrinkage of the mortar, and the type of mortar used. The ratio strength of bond:tensile strength is greater for a 1:1:6 CaO-cement-sand mortar than for a 1:3 cement-sand mortar, but the percentage of

failures is rather greater with the former. Metal lugs in the mortar, by preventing movement as the mortar contracts on ageing, reduce the durability of the joint. Disintegration of the mortar or of the brick is not a common cause of failure of a joint. H. F. GILLBE.

**Retrogression of porous concretes.** G. NATTA and C. G. FONTANA (Giorn. Chim. Ind. Appl., 1931, 13, 173—178).—Investigation of the slow falling-off in the mechanical properties of the concrete in a reinforced-concrete structure revealed insufficient gravel or coarse sand, excess of fine sand, and excess of  $H_2O$ . The very high porosity of the concrete and the large sp. area of the mortar, due mainly to the faulty granulometric composition of the sand, led to pronounced hydrolysis, with carbonation of the  $CaO$  produced. In slightly porous concretes rich in cement, the gradual increase in vol. accompanying such hydrolysis and carbonation results in greater compactness and mechanical strength; hydrolysis is slow, since the increase in vol. gives lessened porosity. Poor, porous cements undergo rapid hydrolysis and carbonation and show negligible increase in strength with time. T. H. POPE.

**Nature of the building stone employed at Salamanca; the patina and its artificial production.** I. RIBAS and (SRTA.) P. DE PRADA (Anal. Fis. Quím., 1931, 29, 211—220).—The stone quarried near Salamanca consists principally of quartz and felspar. The patina is of practically the same chemical composition as the underlying stone, and its formation is ascribed to dehydration and consequent change of structure or orientation of the  $Fe_2O_3$  in the surface layer; the Ti present (0.4—0.5%  $TiO_2$ ) does not take part in the change. The patina may be produced artificially by applying to the stone an aq. suspension of precipitated  $Fe_2O_3$  and powdered stone. H. F. GILLBE.

**Durability of Philippine woods against fungi.** O. A. REINKING and C. J. HUMPHREY (Philippine J. Sci., 1931, 45, 77—89).—The relative durability of Philippine woods against fungi, as determined by laboratory tests, agrees with that obtained under service conditions.

D. K. MOORE.

**Thermal conductivity.**—See I.

#### PATENTS.

**Rotary [cement] kilns.** J. S. FASTING (B.P. 347,665, 22.11.29).—A portion of the length of a rotary kiln is subdivided into a number of cylindrical sections grouped round the axis and spaced apart so that air can circulate around them; they may either be fixed to the main tube of the kiln or be separate from it and driven at a different speed. [Stat. ref.] B. M. VENABLES.

**Manufacture of Portland cement.** F. W. HUBER (U.S.P. 1,781,232, 11.11.30. Appl., 19.3.30).—In making cement from raw materials containing  $Ca_3(PO_4)_2$ ,  $Fe_2O_3$  is added in greater mol. ratio than the  $P_2O_5$  radical. During calcination all the  $P_2O_5$  is converted into  $FePO_4$  and the residual Fe becomes effective as a mineralising agent. C. A. KING.

**Hydraulic cement and other calcareous plastics.** W. A. COLLINGS, Assr. to SILICA PRODUCTS Co. (U.S.P. 1,755,502, 22.4.30. Appl., 17.11.27).—The rate of ageing and the final mechanical strength of concrete is

increased if 5—10% of a naturally swelling clay (bentonite) and NaCl are added to Portland cement. The proportion of NaCl required depends on the amount occurring naturally in the bentonite, but is usually about 10% of the wt. of the clay. C. A. KING.

**Asbestos mortar.** I. KOTANI (B.P. 345,727, 20.1.30).—A mixture of crushed, air-blown asbestos fibre with Portland cement and sand is stirred and kneaded with  $H_2O$ . II. ROYAL-DAWSON.

**Rendering porous structural materials [stone-work] water- or weather-proof.** ELEKTROCHEM. WERKE MÜNCHEN A.-G. (B.P. 347,514, 29.5.30. Ger., 22.7.29).—The materials are treated with aq. emulsions of fatty oils (linseed oil) prepared with the smallest quantity of an emulsifier necessary to yield a stable emulsion which coagulates irreversibly on drying.

L. A. COLES.

**Making a compound for mixing with cements.** R. BUHMAN (U.S.P. 1,781,267, 11.11.30. Appl., 5.4.24).—A mixture, used especially for pumping into the cases of oil wells, consists of a cement slurry to which about 1.5% of  $CaCl_2$  and 1% of  $FeCl_3$  have been added. The  $FeCl_3$  is thereby converted into  $Fe(OH)_3$ , which retards the initial set of the slurry, the final set being accelerated. C. A. KING.

**Utilisation of blast-furnace slags.** P. P. BUDNIKOV (B.P. 347,357, 17.2.30).—The slag is ground with anhydrite and/or gypsum cement, insol. anhydrite or Estrich gypsum, or dolomite burned at 500—800°;  $CaCO_3$ , dolomite, fluorspar, phosphorite, or mixtures of these, may also be added. Alternatively, the slag is ground wet and the other constituents are added during the drying. L. A. COLES.

**Asphalt emulsions.** P. M. TRAVIS and A. L. HALVORSEN, Assrs. to EMULSION PROCESS CORP. (U.S.P. 1,757,102—3, 6.5.30. Appl., [A] 10.1.28, [B] 29.12.28).—(A) An emulsion of asphalt with  $H_2O$  contains not more than 1% (0.1—0.4%) of Na silicate and not more than 0.5% of a fatty acid, preferably oleic acid. (B) As the emulsifying agent, not more than 0.75% of  $Na_3PO_4$  by wt. on the emulsion is used. C. A. KING.

**Manufacture of an emulsion of tar for road-making.** UNION CHIM. BELGE, SOC. ANON. (B.P. 344,490, 2.12.29. Belg., 9.7.29).—Coal tar, fractionally distilled up to 270° to remove  $H_2O$ , light oils, and  $C_{10}H_8$ , is mixed at above room temp. with soap solution and potato fecula. H. ROYAL-DAWSON.

**Manufacture of road-building material.** S. S. SADTLER, Assr. to AMESITE ASPHALT Co. (U.S.P. 1,758,913—4, 13.5.30. Appl., [A] 28.5.29, [B] 28.6.29).—(A) Suitable aggregate is treated in a pug mill with kerosene, light coal tar, or other asphalt solvent and an aq. dispersion of rubber latex. Later a substantial coating of bitumen of 70—130 penetration is applied and 0.2%—1% of  $CaO$  is added to toughen the asphalt. The proportion of rubber is 0.2—2% of the bitumen used. (B) To ensure a satisfactory coating of the aggregate with rubber before applying bitumen, 0.25—1 pt. of rubber in 8—12 pts. of solvent is used for 1900 pts. of aggregate, which is then treated with 35—140 pts. of bitumen. C. A. KING.

**Production of premixed asphalt composition.** J. S. DOWNARD (U.S.P. 1,781,105, 11.11.30. Appl., 6.3.26).—Asphalted aggregate is coated with  $\text{CaCl}_2$  solution to keep the surface wet and prevent adhesion. At any subsequent time the mass is treated with a precipitating agent, *e.g.*,  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ , which renders the mass adhesive and capable of being consolidated.

C. A. KING.

**Floor coverings.** N. KENYON, and SPEN RUBBER WORKS, LTD. (B.P. 345,700, 8.1.30).—Rubber, cork powder, S,  $\text{ZnO}$ , whitening, French chalk, and paraffin wax are mixed together and vulcanised.

H. ROYAL-DAWSON.

**Lute.** R. B. MCCARTY, ASSR. to GULF REFINING Co. (U.S.P. 1,782,932, 25.11.30. Appl., 6.9.27).—A luting material for withstanding the action of  $\text{Cl}_2$  at high temp. is prepared by mixing 65% of finely-ground, burnt fireclay, preferably obtained from the lining of  $\text{AlCl}_3$  chambers, with 35% of  $\text{NaOH}$  solution (*d* 1.384).

W. J. WRIGHT.

**Cementing together two surfaces of identical or different substances.** T. GOLDSCHMIDT A.-G. (B.P. 347,242, 20.12.29. Ger., 2.2.29).—A porous carrier, *e.g.*, tissue paper, impregnated with a liquid artificial resin or intermediate condensation product, to which glycerin may be added, is interposed between the surfaces of, *e.g.*, sheets of wood, and the whole is united by heat and pressure.

L. A. COLES.

**Colouring of concrete [by spraying with water paints].** J. F. BARR (B.P. 347,523, 5.1.30).

**Production of artificial marble.** A. BAUMANN (B.P. 349,348, 26.6.30).

**Workshops. Mixing of materials.**—See I.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Surface pyrometers [for metals].** W. CLAUS and R. HASE (Z. Metallk., 1931, 23, 120—123).—Methods of measuring the temp. of metal surfaces by means of special pyrometers are described and the sources of error discussed.

A. R. POWELL.

**Detection of sulphur in steel sections by the impression method.** H. J. VAN ROYEN and E. AMMER-MANN (Arch. Eisenhüttenw., 1930—1, 4, 435—438; Stahl u. Eisen, 1931, 51, 462—463).—The yellow colour of  $\text{HgCl}_2$  paper prints obtained from steel sections is invariably due to S and not to P, but the test is not sufficiently delicate as white or black Hg compounds containing S may also be formed under certain conditions. A more certain test comprises covering the surface of the steel with a gelatin paper impregnated with a solution containing 1.25%  $\text{Cd}(\text{OAc})_2$ , 8%  $\text{AcOH}$ , and 2.5%  $\text{H}_2\text{SO}_4$ , whereby  $\text{CdS}$  is formed in the paper covering the sulphide inclusions. The paper is washed in running  $\text{H}_2\text{O}$  and developed by immersion in a solution containing 120 g.  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and 120 c.c. conc.  $\text{H}_2\text{SO}_4$  per litre, which converts the  $\text{CdS}$  into black  $\text{CuS}$ .

A. R. POWELL.

**Factors affecting corrosion of buried steel.** F. L. BASSETT (J.S.C.I., 1931, 50, 161—166 T).—The

wt. losses of 480 specimens of two brands of mild steel in 80 samples of damp soils were recorded after periods of 1, 2, and 3 months. The soils were marls and clays from the Iraq-Syrian desert, with variable proportions of sand, and chloride salinity from zero to 27.8%  $\text{NaCl}$  equiv. The  $p_{\text{H}}$  values were all in the sub-alkaline range, and corrosion took the usual course of localised anodic pitting. Soils with very low chloride, up to 0.2%  $\text{NaCl}$  equiv., showed in each series a higher average attack than salt-free soils; above about 0.2%, however, the attack is diminished, and in soils containing more than 1%  $\text{NaCl}$  equiv. the repression of corrosion becomes strongly marked. Diminished corrosive attack in the salted soils is also found to be correlated on the average with increased water retentivity, and is ascribed to hindrance of O diffusion. Hygroscopicity of salted soils therefore becomes a controlling factor which at quite low concentrations may reverse the normal stimulation of Cl ion for corrosion, by reducing the O supply to the cathodic areas of the plate.

**[Tensile] resistance of ordinary steels at high temperatures.** L. GUILLET, J. GALIBOURG, and M. SAMSOEN (Compt. rend., 1931, 192, 861—863).—Tensile tests at 225°, 325°, 425°, and 525° carried out as previously (cf. B., 1929, 601, 752) are detailed regarding four ordinary steels containing, respectively, (in %) C 0.15, 0.17, 0.18, 0.35; Si 0.07, 0.10, 0.11, 0.0008; Mn 0.32, 0.53, 0.58, 0.60; S 0.028, 0.027, 0.022, 0.020; P 0.017, 0.038, 0.022, 0.027, and having breaking stresses at room temp. of 37.3, 44.2, 46.8, 55.2 kg./mm.<sup>2</sup> The results strengthen the conclusion that there is, properly speaking, no true elastic limit for steel even at room temp., apparent absence of change in dimensions being due only to imperfect means of measurement.

C. A. SILBERRAD.

**Resistance of chromium steel to alkali solutions.** A. KRÜGER (Chem.-Ztg., 1931, 55, 335).—Cr steel, although attacked by hot, dil. acid solutions, is quite resistant to boiling solutions of  $\text{NaOH}$ . No trace of Fe could be detected in the solution after treatment under various conditions. Vessels of Cr steel should therefore prove useful in quantitative analysis involving the use of alkaline solutions. The material is also resistant to  $\text{H}_2\text{O}_2$  and  $\text{Na}_2\text{S}$  solutions. E. S. HEDGES.

**Manganese-free zirconium-treated steels.** F. M. BECKET (Min. and Met., 1931, 12, 234—236).—Steels with a high content of S and P and with < 0.1% Mn cannot be rolled without cracking, but if 0.4% Zr is added to the ladle just before casting, the resulting ingots work well and have a higher ductility but lower yield point and ultimate strength than Mn steels. It is suggested that addition of Zr to steels with a moderate Mn content and relatively low in P and S would increase the ductility and confer good deep-drawing properties on the sheet.

A. R. POWELL.

**Influence of occluded gases on the mechanical properties of metals.** L. GUILLET and J. COURNOT (Compt. rend., 1931, 192, 787—789).—The conclusions arrived at by Guichard etc. (B., 1931, 592) are questioned in view of the results of Hugues (B., 1928, 159), Bogitch (B., 1928, 126), and others.

C. A. SILBERRAD.

**Outdoor corrosion of zinc and effect of rainfall and atmospheric pollution.** W. S. PATTERSON (J.S.C.I., 1931, 50, 120—123  $\tau$ ).—The deposit formed on Zn during outdoor corrosion in an urban atm. does not offer complete protection to the metal. There is appreciable evidence, however, to show that the deposit has some retarding effect on the corrosion, probably by preventing the direct access of acid rain-water to the metal. In the winter period, when the air of London contains appreciable amounts of  $\text{SO}_2$ , the curves for rainfall and corrosion are divergent; in the summer period, when atm. pollution is low, the curves are parallel. The velocity of the outdoor corrosion of Zn is largely governed by the character of the corrosion products and the manner in which the solubility of these in rain-water is affected by atm. pollution.

**Initial corrosion rates of metals.** R. H. BROWN, B. E. ROETHELI, and H. O. FORREST (Ind. Eng. Chem., 1931, 23, 350—352).—The initial rates of corrosion in oxygenated water have finite values and have been measured by rotating an oxide-free metal cylinder in oxygenated water and determining the decrease of O concentration during the test. The initial rates of corrosion of the metals examined decrease in the following order: Al, Zn, Fe, Cu, Ni, Sn, Ag. In the case of the first three metals the corrosion rate appears to depend on the rate of diffusion of O to the specimen, but with the remaining metals the rate of oxidation of the metal is the controlling factor. The initial corrosion rates of all the metals decrease after a relatively short time, indicating the formation of partly or completely protective films in all cases. Metals such as Ni, Sn, Cu, and Ag do not seriously deteriorate on exposure to oxygenated water, not because of a negative corrosion tendency, but because of the formation of an impermeable corrosion product which prevents further deterioration. The electromotive series is not considered a proper criterion for judging the probability of corrosion of a metal in oxygenated water, and the free energy decreases, assuming O and  $\text{H}_2\text{O}$  to react with the metals to form hydroxides in saturated solution are more exact measures of the tendencies to corrode.

A. R. POWELL.

**Method of testing the local-element theory of corrosion.** W. GUERTLER and B. BLUMENTHAL (Z. Metallk., 1931, 23, 118—119).—According to the theory of Palmaer and others the rate of dissolution of Zn in dil. acids should be proportional to the concentration of the acid. An apparatus is illustrated for alternately immersing Zn strips in 0.1*N*- and 0.2*N*-HCl and measuring the vol. of gas evolved; results obtained in this apparatus show that the rate of dissolution of Zn in 0.2*N*-HCl is only 1.5—1.8 times as great as in 0.1*N*-HCl. The bearing of these results on Palmaer's theory (cf. B., 1929, 921) is discussed briefly.

A. R. POWELL.

**Attack of insects on metals.** II. O. BAUER and O. VOLLENBRUCK (Z. Metallk., 1931, 23, 117; cf. B., 1930, 1071).—Specimens of Zn roofing perforated by beetles of the *Cerambycides* family or possibly by wood-wasps of the *Sirex* family are illustrated.

A. R. POWELL.

**Alloys resistant to heat and sulphur.** H. GRUBER (Z. Metallk., 1931, 23, 151—157).—The behaviour of 29 Fe, Co, and Ni alloys containing varying quantities of one or more of the metals Cr, Mn, Al, Mo, and W has been investigated in an atm. of  $\text{H}_2\text{S}$  at 700—1000°. The rate of corrosion by the gas depends on the fusibility of the sulphides formed, but in alloys containing Al a resistant S film is formed which is generally highly impervious to  $\text{H}_2\text{S}$ . An alloy of 61% Ni, 15% Cr, 20% Fe, and 4% Mn to which 10% of Al has been added is permanent in atm. containing S compounds and has sufficient strength and good casting properties for use in the manufacture of furnace parts and annealing boxes. Addition of 10% Al to pure Ni reduces the rate of absorption of S at 1000° to  $\frac{1}{3.5}$ th that of pure Ni and the resulting sulphide layer is fairly adherent; with 15% Al even better resistance is obtained. Similar results are obtained with Co-Al alloys. Although Mo itself is the most resistant of the metals mentioned above to the action of S at 1000°, addition of Mo to alloys of Fe, Co, Ni, and Cr tends to reduce their resistance to S at high temp.

A. R. POWELL.

**Röntgenographic investigation of the ageing of duralumin at room temperature.** J. HENGSTENBERG and G. WASSERMANN (Z. Metallk., 1931, 23, 114—117).—During the ageing of duralumin at room temp. slight broadening of the interference lines in the Debye-Scherrer diagrams takes place and their intensity increases, whilst the intensity of the diffused radiation diminishes slightly. These facts are interpreted as indicating that, during ageing, an enrichment in Cu atoms occurs in small regions of the Al lattice without the simultaneous separation of a new crystal phase.

A. R. POWELL.

**Chemical and physical phenomena in autogenous welding.** L. KNEZ (Arh. Hemiju, 1931, 5, 15—25).—A theoretical treatment of the chemical and physical processes which occur during welding of Fe and Cu with the  $\text{C}_2\text{H}_2$  flame. The effects of impurities in the gas, such as  $\text{PH}_3$  and  $\text{H}_2\text{S}$ , are summarised. H. F. GILLBE.

**Theory of addition agents [in electroplating].** S. KANEKO (J. Soc. Chem. Ind., Japan, 1931, 34, 103B).—The effect of addition agents on electroplating baths is ascribed to (1) reduction of surface tension of the solution, favouring the formation of a larger number of nuclei; and (2) positive adsorption of the addition agent, producing a smaller grain size. E. S. HEDGES.

**Buffer effect in nickel-plating baths.** J. BARBAUDY and A. PETIT (Compt. rend., 1931, 192, 834—837).—Titration curves and, therefrom, Van Slyke's (cf. A., 1922, i, 893) buffer value,  $\beta = dB/dp_{\text{H}}$ , have been determined at 20° and 50° for (1) a rapid Ni-plating bath (hot) of composition (per litre)  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$  450 g.,  $\text{NiCl}_2$  12 g., and  $\text{H}_3\text{BO}_3$  22 g.; (2) the same bath modified by use and additions of acid and  $\text{NiCO}_3$ ; and (3) bath (1) without  $\text{H}_3\text{BO}_3$ . The buffer effect of the  $\text{H}_3\text{BO}_3$  in maintaining the  $p_{\text{H}}$  below that of the precipitation of  $\text{Ni}(\text{OH})_2$  is clearly shown, and also the greater similarity *inter se* of the three  $\beta/p_{\text{H}}$  curves at the higher temp. Curves relating, at 20—80°, the working  $p_{\text{H}}$  to the  $p_{\text{H}}$  of  $\text{H}_2\text{O}$  at the same temp. show an almost constant relative acidity for (1) and (2), but a



rapid increase with temp. for (3). This is attributed to the unbuffered hydrolysis of  $\text{NiSO}_4$ . C. A. SILBERRAD.

**Protection of alloys and steel against atmospheric attack.** E. DE WINIWARTER (Bull. Soc. chim. Belg., 1931, 40, 129—143).—A lecture on Ni-, Co-, and Cr-plating. R. CUTHILL.

**Electrodeposition of chromium from ammonium chromioxalate.** A. MAZZUCHELLI (Atti R. Accad. Lincei, 1930, [vi], 12, 587—588).—A satisfactory deposit of Cr may be obtained on a brass cathode by electrolysis with a high c.d. (25—150 amp./dm.<sup>2</sup>) a conc. solution of  $\text{NH}_4$  chromioxalate. The current efficiency is very low, but rises somewhat with increase of c.d.; the coherence of the deposit decreases under the same conditions. F. G. TRYHORN.

**Hardness and hydrogen content of electrolytic metals.** GUICHARD, CLAUSMANN, BILLON, and LANTHONY (Compt. rend., 1931, 192, 1096—1098; cf. B., 1931, 592, 680).—Five samples of electrolytic Fe gave the following figures for hardness and H content (in c.c. per g. of Fe): 365, 2.1; 338, 1.7; 265, 1.5; 247, 8.3; 202, 3.0. A similar absence of parallelism between hardness and H content is observed in unannealed Ni and Co. The error in the determination of H in the authors' previous results cannot exceed 0.1 c.c. C. A. SILBERRAD.

**Refractories.**—See VIII. **Protection of Fe.**—See XI. **Cd and foodstuffs.**—See XIX.

See also A., June, 676, Pb-Bi and Sn-Bi alloys. Al bronze. Al-Ti, Cu-Sn, and W-Re alloys. 691, Electrochemistry of corrosion. 693, Ferrite from rusting of iron. 701, Test for Ag in minerals. 702, Analysis of Cu-Ag and Cu-Zr alloys. Determination of Cu in presence of Pt.

#### PATENTS.

**Production of malleable-iron castings.** L. H. MARSHALL, Assr. to A. A. GRUBB (U.S.P. 1,781,119, 11.11.30. Appl., 27.4.28).—White-iron castings are annealed above 360° in closed containers which are packed with a mixture of refractory material and a compound, e.g.,  $\text{FeCl}_3$ , which will evolve a non-oxidising and etching atm. when heated. C. A. KING.

**Forming [ferrous] alloys.** P. A. E. ARMSTRONG (U.S.P. 1,781,490, 11.11.30. Appl., 8.5.28).—Ferrous alloys are formed into articles, e.g., valves and tappets, by melting the constituents of the alloy by means of an electric arc in a mould crucible made of a metal of relatively low m.p., but of high conductivity. Thus an Fe electrode is inserted into the base of a Cu crucible the bottom of which is of the desired shape. The metal alloy and slag constituents are packed round the crucible walls and a second electrode down the central open space causes an arc between the two electrodes. As metal melts, the slag rises and forms a protective layer on the crucible walls, which cool it. The electrodes themselves may form part of the alloying metals. C. A. KING.

**Heating metallic and other electrically conducting bodies.** S. E. KUSNETZOV (B.P. 347,163, 17.10.29).—Two articles or groups of articles embedded in carbon-

aceous material mixed with, if desired, fireclay, asbestos, etc., are directly connected with the respective supply leads from a source of current. J. S. G. THOMAS.

**[Salt-bath] heat-treatment of metals.** A. E. BELLIS (B.P. 347,238, 16.12.29. U.S., 14.12.28).—Fusion of the salt-bath materials is initiated by combustion of an Fe compound, e.g., thermit, on the surface of the bath, and a low-voltage current is passed between an electrode immersed in the bath and the metal wall of the container. J. S. G. THOMAS.

**Carbonising of metal surfaces [used in electron-discharge devices].** WESTINGHOUSE LAMP Co., Assees. of C. V. IREDELL (B.P. 347,267, 24.1.30. U.S., 25.1.29).—The oxidised surface of the metal, e.g., Ni, is exposed at 800—1000° to a hydrocarbon gas, e.g.,  $\text{C}_2\text{H}_2$ , containing a vaporised liquid hydrocarbon, e.g., light petroleum or kerosene, and is degassed by heating *in vacuo*. J. S. G. THOMAS.

**Hg-vapour generators.**—See I. **Cleaning metals.**—See VII. **Blast-furnace slag.**—See IX. **Treatment of surfaces.** Coating compound.—See XIII.

#### XL—ELECTROTECHNICS.

**Protection of iron in aerated saline solutions : an Evans pile.** E. HERZOG and G. CHAUDRON (Compt. rend., 1931, 192, 837—839).—The pile, depending on differential aeration (cf. Evans, B., 1923, 983A), consists of two precisely similar Fe plates. The cathode, horizontal, is immersed in NaOH solution and is aerated by a current of air impinging on it; the anode is vertical and below the cathode in a solution of  $\text{FeCl}_2$  or  $\text{FeSO}_4$ , the two solutions being separated by a horizontal diaphragm. The p.d. increases in a few hr. from 0.15 to 0.43 volt, then becoming steady. Time-potential curves show the results of adding NaCl without (giving a p.d. of 0.4 volt), or with, simultaneous addition of an acetate, citrate, or phosphate (to illustrate their buffer effects). Citrates, oxalates, tartrates, phosphates, and arsenates all sooner or later cause the formation of a film reducing the p.d. to about 0.02 volt. With sea-water the p.d. is steady at 0.15 volt, a deposit of  $\text{MgO}$  forming. C. A. SILBERRAD.

**Positive electrode with gas circulation for air depolarisation.** C. FÉRY (Compt. rend., 1931, 192, 881).—To assist depolarisation by atm.  $\text{O}_2$  where the polarising gases (e.g.,  $\text{H}_2$  or  $\text{NH}_3$ ) are lighter than air, the positive electrode is made hollow and air brought to the bottom of the cavity, whilst the polarising gases escape by a vent at the top. C. A. SILBERRAD.

**Efficient accumulator not liable to sulphation.** C. FÉRY and REYNAUD-BONIN (Compt. rend., 1931, 192, 1035—1037; cf. B., 1925, 44).—To prevent access of  $\text{O}_2$  to the negative plates these are each surrounded by a protecting medium, permeable to the ions but not to the gases, and covered by a cap 15—20 mm. high, which completely prevents access of air from above. These caps at the end of charging are full of  $\text{H}_2$ , production of which continues slowly through the action of the electrolyte on the spongy Pb. Actual comparison of precisely similar commercial-type, 30AH-type batteries, the one thus protected, the other not, shows the very marked

superiority of the protected type, and confirms the authors' views on the cause of sulphation (cf. A., 1919, ii, 263).

C. A. SILBERRAD.

Continuous kilns.—See VIII. Electroplating. Ni-plating baths. Protection of alloys etc. Cr. Hardness etc. of electrolytic metals.—See X. Photoelectric control in waterworks' practice.—See XXIII.

See also A., June, 686, Conductivity of liquids. Potential of high-resistance cells. 702, Determination of Cu in presence of Pt. 703, Self-regulating induction furnace.

#### PATENTS.

[Gas-filled] photoelectric cells. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 347,544, 5.7.30. Holl., 24.7.29).—A grid raised to a suitable potential is arranged within the photoelectric cell at a distance from the photo-active electrode which is equal to or less than the free path of an electron in the gas-filling.

J. S. G. THOMAS.

Cathodes of electron-discharge tubes or thermionic valves. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 347,500, 16.5.30. Holl., 9.10.29).—A highly refractory metal core, *e.g.*, of W, covered, at least partly, with a layer of metal having a lower m.p., *e.g.*, Ni, is helically wound with metal wire, *e.g.*, Ni, at least partly covered with material of high electron-emitting capacity, *e.g.*, an alkaline-earth metal or oxide.

J. S. G. THOMAS.

[Indirectly heated] cathodes of vacuum-electric tube devices. E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 347,311, 31.1.30).—A filamentary heater, *e.g.*, of W, is, prior to assembly within an insulating sheath, *e.g.*, of SiO<sub>2</sub> or porcelain, coated with an adherent protective coating consisting of refractory metallic oxide or other non-injurious refractory insulating material.

J. S. G. THOMAS.

[Partition walls for] electrical accumulators. H. WEHRLIN (B.P. 347,012, 1.5.30. Ger., 29.5.29).—The edges of the walls (composed of glass wool, asbestos, or cellulose) are united by substances, *e.g.*, asphalt, artificial resins, or rubber, which are not attacked by the electrolyte.

H. ROYAL-DAWSON.

Precipitation electrode for electric gas purification. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 347,501, 16.5.30. Ger., 18.5.29).—The electrode, made in the form of a box with slot-like openings in its sides serving as precipitation surfaces, has the parts lying behind the trap-pockets and protected from the flow of gas entirely or partly cut out, and vertical or oblique stays are arranged in the inner trap chamber to prevent cross-currents.

J. S. G. THOMAS.

Mercury-vapour rectifiers. A.-G. BROWN, BOVERI & CO. (B.P. 349,411 and 349,413, [A] 14.10.30, [B] 23.10.30. Ger., [A] 5.12.29, [B] 19.4.30).

[Switching means for the heating device in] luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 347,559, 18.8.30. Ger., 19.10.29. Addn. to B.P. 316,611; B., 1930, 775).

Electric-discharge devices for producing ultra-violet rays [in foodstuffs etc.]. F. REYNOLDS (B.P. 349,294, 20.5.30).

Treatment of hydrocarbons.—See II. Forming alloys. Heating of metals. Carbonising metal surfaces.—See X.

#### XII.—FATS; OILS; WAXES.

Influence of the so-called *isooleic* acid on fat hardening from the viewpoint of the phase rule. W. KOCZY and F. GRIENGL (Monatsh., 1931, 57, 253—290).—The m.p.-composition relationships are studied for the possible binary systems comprising *isooleic* (prepared by Schmidt's method; A., 1890, 863), palmitic, oleic, linoleic, and stearic acids, and for the ternary systems comprising stearic and the above unsaturated acids, both liquidus and solidus values being determined. The system formed by an American cottonseed oil and a technical hydrogenated oil derived from it is also investigated. As the result of a study of "pseudo-binary" mixtures, chosen to represent the possible products obtainable in the course of hydrogenation of the free acids in such an oil, it is concluded that: (1) during the hydrogenation of a mixture of 40.5% of oleic and 59.5% of linoleic acids the formation of *isooleic* acid is at first without influence, but beyond a concentration of about 7% has a marked effect in increasing the m.p.; (2) the presence of a const. amount (1.8%) of stearic acid considerably reduces the hardening effect of the *isooleic* acid; (3) an increase in the content of stearic acid to 6.6%, as the result of hydrogenation, has a much greater hardening effect than has *isooleic* acid alone; and (4) the presence of a const. proportion (20.9%) of palmitic acid prevents any increase in m.p. as the result of hydrogenation. The last effect is also observed in the system formed by linoleic and oleic with 1.8% of stearic and 20.9% of palmitic acids.

H. A. PIGGOTT.

Carbohydrates affect mill values of cottonseed. G. S. MELOY (Oil & Fat Ind., 1931, 8, 187—189).—Tables are given connecting oil, protein, and NH<sub>3</sub> contents with the proportion of meats in the whole seed: for a given % of oil in whole seed, the increase of NH<sub>3</sub> content (due to increased % of meats) coincides with increase of material other than fat or protein. The connexion between composition and growing conditions is illustrated.

E. LEWKOWITSCH.

Splitting of castor oil. H. M. LANGTON (J.S.C.I., 1931, 50, 213—214 r).—After pointing out the unique character of castor oil, which is attributable to its containing upwards of 80% of the triglyceride of ricinoleic acid, the author enumerates the results of saponifying "firsts" castor oil in an autoclave using 2.5% CaO as the splitting base under a steam pressure of 120 lb./in.<sup>2</sup> for 6 hr. The course of the reaction was followed by hourly withdrawal of samples which were tested for free fatty acids. The consts. of the oil are given and the results are exhibited both numerically and graphically, and are compared with those of Jones, who operated at 200 lb./in.<sup>2</sup> (J.S.C.I., 1917, 36, 359). The author's results lie along a curve of the exponential type, and are thus in accord with his published records of

the splitting of other fatty oils. The average split obtained was apparently 75.2%, but a glycerin determination on the final product showed that the split was almost complete. The reduction in Ac value from 135.6 to 115.4 is evidence that some polymerisation has taken place during the splitting, but not to such a great extent as in the anomalous results obtained by Jones. The lower pressure used in the present case is given as the reason for absence of abnormality in the results.

**Composition of commercial palm oils. II. Fatty acids of some palm oils of high free acidity.** T. P. HILDITCH and (Miss) E. E. JONES (J.S.C.I., 1931, 50, 171—176 t).—Four commercial palm oils of high free acidity from various sources have been investigated with regard to the quantitative composition of the fatty acids present in (a) the crude oils and (b) the neutral portions of the oils; the results have been discussed in conjunction with those for four oils of low free acidity (cf. B., 1930, 956). Six of the eight crude oils were closely similar in composition, namely, myristic (1.2—5.9%), palmitic (37.5—43.0%), stearic (2.2—5.9%), oleic (40.2—45.2%), and linoleic (6.5—11.2%) acids; the remaining two, both from Drewin (Gold Coast) but of widely different free acidity, had myristic (2.2—2.3%), palmitic (34.3—35.3%), stearic (5.2—5.6%), oleic (49.5—52.3%), and linoleic (5.5—8.3%) acids. Broadly speaking, increase in unsaturated acids is compensated for by diminution in palmitic acid, and *vice versa*, the proportions of myristic and stearic acids being small and somewhat variable. Plantation oils, in addition to very low free acidity, tend towards const. fatty acid composition, probably in consequence of standardisation of the variety of oil palm cultivated. Except in one case (Niger), there was little difference between the component fatty acids of the crude oils and of the neutral portions prepared by alkali-refining the crude oils; it would appear that hydrolytic changes during rancidity affect the different fatty acids indiscriminately and non-selectively. The neutralised Niger oil was about 4% lower in palmitic, and higher in oleic, acid than the crude oil. The acetyl values of the neutralised oils indicated relatively little di- or mono-glycerides and correspondingly extensive hydrolysis to glycerol and fatty acids. On the other hand, the plantation palm oils of lowest free acidity had acetyl values of 11—12, indicating the presence of some mono- or di-glycerides in the fresh fat as matured in the palm fruit; the amount may correspond to 4—8% of the oil, expressed as diglycerides.

**Laboratory bleaching technique for fatty oils.** J. T. R. ANDREWS and R. G. FOLZENLOGEN (Oil & Fat Ind., 1931, 8, 183—185).—For laboratory bleaching tests the use of 3% of English earth and 1% of H<sub>2</sub>O (at 135° for 5 min. or 105° for 15 min.) gives results only slightly inferior to those by the official A.O.C.S. method (6% of earth) and comparable with plant practice; moisture control is essential when comparing bleaching-earth efficiencies. E. LEWKOWITSCH.

**Oils for chronometers.**—See II. Litharge-linseed oil.—See XIII. Oleum ricini.—See XX.

See also A., June, 679, Soap solutions. 691, Oxidation of fats by air. 711, Hydrogenation of fatty

acid esters. 712, Hydrogenation of linolenic acid esters. 773, Autoxidation of fats.

#### PATENTS.

**Margarine and other manufactured fatty-predominating substances.** J. L. KERR (B.P. 346,276, 3.1.30).—The melted mixed fats are sprayed upwards into a cooling chamber, from which the solidified granules are subsequently collected and amalgamated.

E. LEWKOWITSCH.

**Production of saturated fatty acids.** H. P. KAUFMANN (B.P. 345,626, 24.12.29. Ger., 27.12.28).—Catalytic hydrogenation (preferably with Ni) is conducted simultaneously with hydrolysis of the fat by any usual method (e.g., in the presence of MgO, with alkali). A working pressure of about 10 atm. is cited. The relative extent of hydrolysis and hydrogenation is controlled by the amounts of the catalysts used, the temp. and duration of the treatment, and the H pressure. [Stat. ref.] E. LEWKOWITSCH.

**Manufacture of sulphonation products of fats, fatty oils, fatty acids, or aliphatic hydrocarbons of high mol. wt.** I. G. FARBENIND. A.-G. (B.P. 346,945, 17.3.30. Ger., 15.3.29).—The fats or oils (C<sub>8</sub> or over), especially mineral lubricating fractions (saturated or unsaturated compounds, with or without OH groups), are dissolved in liquid SO<sub>2</sub> and treated with SO<sub>3</sub>, oleum, or chlorosulphonic acid at about −12° to −15°. After evaporating the solvent at 0° the product is worked up in the usual manner. E. LEWKOWITSCH.

**Manufacture of water-soluble condensation products of unsaturated fatty acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,425, 4.1.30).—Higher (poly-)unsaturated fatty acids (e.g., wood oil fatty acids) are treated with sulphonic acid derivatives of aromatic (polynuclear) hydroxy-compounds, e.g.,  $\alpha$ - or  $\beta$ -naphtholsulphonic acids, in the presence of dil. H<sub>2</sub>SO<sub>4</sub> at 120—140°. The neutralised products are H<sub>2</sub>O-sol. and stable to mineral acids and alkali, but can be salted out by NaCl. E. LEWKOWITSCH.

**Manufacture of soft soap.** K. HENTSCHEL (B.P. 346,389, 3.1.30. Ger., 29.1.29).—Coconut oil, palm-kernel oil, etc., or their mixtures with other fats (not over 35%), are saponified with KOH containing not more than 2.4% of constituents such as chlorides etc. with salting-out properties; the amount of H<sub>2</sub>O is adjusted to give a yield of 200—300% of the transparent stable product. E. LEWKOWITSCH.

**[Paste] soaps and cleansers [for paint etc.].** F. J. LOVEFACE (B.P. 347,433, 29.3.30).—Na silicate or glycerin, borax solution, light mineral oil containing petroleum jelly, powdered pumice, benzene, and perfume are added successively to a NaOH-tallow-vegetable oil soap. E. LEWKOWITSCH.

**Apparatus for treating oleaginous material.** J. DAVIDSON, Assr. to DAVIDSON-KENNEDY Co. (U.S.P. 1,782,714, 25.11.30. Appl., 15.4.29).—Plant for cooking oleaginous materials with direct superheated steam at 206—372° is detailed: an automatically controlled measuring kettle, a cooker, and receiver are superimposed concentrically. E. LEWKOWITSCH.

**Manufacture of waxes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,438, 10.1.30).—Natural or artificial waxes (*e.g.*, montan or shellac wax) are heated with alkylene oxides (*e.g.*,  $C_2H_4O$ ) at, *e.g.*, 120–170° in a closed vessel. Hydrocarbon waxes, and/or organic acids such as phthalic acid or anhydride, resinic acids, etc., may be included if desired. The products, which resemble beeswax, Japan wax, etc., are readily emulsifiable in alkaline solutions.

E. LEWKOWITSCH.

**Lubricating compositions.**—See II. **Polymerised alkylene oxides. Detergents. Basic products.**—See III. **Drying agents for paints.**—See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Viscous and elastic flow of paints.** H. WOLFF (Kolloid-Z., 1931, 55, 81–85).—A “turboviscosimeter” for use with very viscous liquids is described. For liquids showing viscous flow (glycerol, mineral oil, sucrose solutions) the product of the time of fall of the wt. and the load is const., but for the paints examined the product decreases with increasing load. The ratio of the product under a small load to the product under a heavy load gives a measure of the plasticity. E. S. HEDGES.

**Reaction between litharge and linseed oil at room temperature.** K. CHARISIUS and E. KINDSCHER (Farben-Ztg., 1931, 36, 1297–1300).—A mixture of litharge (3 pts.) and linseed oil (2 pts.) was stored for 7 weeks, in the absence of air, with vigorous daily shaking. The colour of the supernatant oil gradually changed to a dark red-brown and the vol. of sediment increased until, after 5 weeks, no sedimentation occurred during 24 hr. After storage it was found by progressive extractions that 29% of the litharge had reacted with the oil, comprising oil-sol. Pb, and  $Et_2O$ - and  $C_6H_6$ - $EtOH$ -sol. organic Pb compounds containing 46 and 50.4% Pb, respectively. The quantity of fatty acids isolated from the Pb compounds (greatly in excess of the fatty acid content of the original oil) indicates that definite splitting of the oil must have occurred. Analyses of the raw materials and of the various products isolated are given.

S. S. WOOLF.

**Application of low-temperature tar in the production of phenol-formaldehyde resins.** G. T. MORGAN and N. J. L. MEGSON (J.S.C.I., 1931, 50, 191–193 r).—Hardening resins have been prepared from crude phenolic fractions of a typical low-temp. tar and purified sufficiently for use in electrical tests. These resin preps. when tested either on single impregnated sheets or in laminated boards have breakdown voltages at least equal to those of trade resins and of resins made under comparable laboratory conditions from pure phenol. Soluble non-hardening resins of Novolak type accompany hardening resins in  $CH_2O$  condensations with crude phenols.

**Machinery for nitrocellulose lacquer manufacture.** F. ZIMMER (Farben-Ztg., 1931, 36, 1342–1344).—Centrifuges used for clarification of clear nitrocellulose lacquers and refining of pigmented lacquers, and of suitable grinding mills for the latter, are briefly described.

S. S. WOOLF.

**Nitrocellulose.**—See V. **Ti film-forming materials.**—See VII.

### PATENTS.

**Coating compound.** S. I. CHARLESWORTH and A. W. HARRIS (U.S.P. 1,782,341, 18.11.30. Appl., 4.2.27).—The molten resinous residue from the distillation up to 300° *in vacuo* of the “pure still residue” remaining after the distillation of the light oils recovered from by-product coke-oven gas is mixed with approx. 30 wt.-% of benzol etc., forming a coating composition for nails etc.

S. S. WOOLF.

**Compositions of cellulose compounds containing softeners of the acetal type.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 318,980, 13.9.29).—Compositions of cellulose derivatives, *e.g.*, cellulose acetate, and, as softener therefor, an acetal of relatively low v.p. having outside the acetal group more than one ether group, *e.g.*, an acetal of an alkyl ether of diethylene glycol, together with a solvent mixture are claimed.

S. S. WOOLF.

**[Coating composition for] treatment of surfaces.** BRIT. CELANESE, LTD. (B.P. 346,269, 4.12.29. U.S., 4.12.28).—An ester or ether of cellulose is precipitated from a solution in a mixed solvent, the higher-boiling constituents, *e.g.*, amyl alcohol, “cellosolve,”  $PhMe$ ,  $H_2O$  being non-solvents for the cellulose derivative, and the lower-boiling constituents, *e.g.*,  $COMe_2$ ,  $AcOEt$ ,  $CHCl_3$ , being solvents or “latent” solvents therefor. Clouded or opaque coatings are formed.

S. S. WOOLF.

**Coating compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 319,590, 23.9.29. U.S., 22.9.28).—Styrene is polymerised by heating in the presence of a catalyst, *e.g.*,  $Bz_2O_2$ , and an inert solvent, *e.g.*, aromatic hydrocarbons, at below 140° (*e.g.*, 5 hr. at 115–120°). The resulting vinyl polymeride is used as the base of lacquers free from “cobwebbing” tendency when sprayed.

S. S. WOOLF.

**Preventing spontaneous combustion [of spray dust from coating compositions].** E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,782,126, 18.11.30. Appl., 13.7.28).—The deposits of spray dust containing oxidisable matter, *e.g.*, linseed oil, are treated with anti-oxidants, *e.g.*, quinol, tannic acid.

S. S. WOOLF.

**Production of drying agents with a high metal content for use in paints.** GEBRÜDER BORCIERS A.-G. (B.P. 346,812, 3.1.30. Ger., 23.1.29).—Metals are substituted in the OH and  $CO_2H$  groups of mono- or polybasic hydroxy-fatty acids or their glyceryl esters (*e.g.*, ricinoleic acid, glycollic acid, castor oil) by reaction with metal oxides, preferably in the presence of saturated or unsaturated non-hydroxylated fatty acids or fatty oils. Such products are sol. in turpentine, oils, etc.

E. LEWKOWITSCH.

**Manufacture of litharge.** E. KNAPP and C. G. ALLGRUNN, Assrs. to NIAGARA SPRAYER Co. (U.S.P. 1,779,003, 21.10.30. Appl., 5.5.25).—Air is forced upwards through molten Pb maintained at approx. 550°, and the  $PbO$  floating to the surface is skimmed off continuously and conveyed to a finishing furnace, where it is kept in const. agitation in contact with air for 3–4 hr. at approx. 600°.

S. S. WOOLF.

**[White] pigment.** E. C. HOLTON, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,778,975, 21.10.30. Appl., 19.10.27).—White pigments comprising a Zn-Pb fume pigment and a smaller proportion of a Ti-O compound and substantially free from non-opaque extenders, *e.g.*, BaSO<sub>4</sub>, are claimed. Such pigments have high opacity and show little tendency to "chalk." S. S. WOOLF.

**Metal-oxide pigments.** GOODGLASS WALL & LEAD INDUSTRIES, LTD., and N. J. READ (B.P. 347,129, 18.1.30).—A metal salt, inert towards paint vehicles and capable of combining with S compounds to form a white sulphide and liberate an inert white oxide, *e.g.*, Zn sesquitanate or orthosilicate, is incorporated as colour-preserving agent for white pigments, particularly Sb oxide, containing the oxide of a metal forming a coloured sulphide. S. S. WOOLF.

**Manufacture of colour lakes from petroleum.** G. ALLEMAN, Assr. to SUN OIL Co. (U.S.P. 1,781,772, 18.11.30. Appl., 24.8.26).—The H<sub>2</sub>O-sol. soaps in the soda-sludge from petroleum refining are separated in an oil-free condition (resins may also be removed, if desired) and precipitated with an aq. solution of a sol. salt of a suitable metal, *e.g.*, CaCl<sub>2</sub>, in the presence of a H<sub>2</sub>O-sol. dye. The lakes produced are insol. in H<sub>2</sub>O, but sol. in AcOEt, petroleum spirit, etc., and may be used in paints, wall-paper inks, etc. E. LEWKOWITSCH.

**Manufacture of colour lakes.** J. Y. JOHNSON From I. G. FARBENIND. A.-G. (B.P. 346,263, 7.10.29).—Basic dyes which may contain acid groups are precipitated by solutions of salts of complex inorganic acids, *e.g.*, phospho-molybdates or -tungstates, the *p<sub>H</sub>* values of which (whilst definitely above 7) have been predetermined by the addition of basic substances, *e.g.*, caustic alkalis, carbonates, acetates, to solutions of the complex acids, or their acid salts, or by other suitable means. Strong acids, *e.g.*, HCl, chloroacetic acid, may, if desired, be added before or after the precipitation.

S. S. WOOLF.

**Printing processes.** H. WADE. From AMER. MULTIGRAPH Co. (B.P. 345,263, 17.12.29).—The surface of a printing plate prepared with an image is treated with a substance, *e.g.*, metal or NH<sub>4</sub> nitrates and/or sulphates, which will render the plate initially ink-repellent on the non-image portions thereof without injuring the image. Printing is then carried out using an ink which will adhere to the image only and which has been treated with a similar substance to the above and will continue the surface-treatment, maintaining the non-image portions ink-repellent. If a Zn plate be used, suitable material, *e.g.*, NH<sub>4</sub> phosphate, is added to wash and ink, forming a protective film on the plate. S. S. WOOLF.

**Manufacture of lacquers, varnishes, or the like.** BAKELITE GES.M.B.H. (B.P. 347,611, 28.1.30. Ger., 29.1.29. Addn. to B.P. 295,335; B., 1930, 469).—The "A" resins ("resols") of the prior patent are converted into "B" resins ("resitols") by heating in suitable solvents, *e.g.*, hydrogenated phenols, with or without hardening agents, the H<sub>2</sub>O of reaction being removed by distillation or desiccation. Org. non-solvents, *e.g.*, Tetralin or PhBr, may be added. S. S. WOOLF.

**Production of rosin oil.** J. N. BORGLIN, Assr. to HERCULES POWDER Co. (U.S.P. 1,782,401, 25.11.30.

Appl., 2.1.29. Renewed 29.3.30).—Wood rosin is heated at 215–250° in the presence of 0.05–1.0% of a B compound, *e.g.*, H<sub>3</sub>BO<sub>3</sub>, and then destructively distilled under reduced pressure. S. S. WOOLF.

**Removing colour from rosin.** W. B. LOGAN, Assr. to NEWPORT Co. (U.S.P. 1,782,267, 18.11.30. Appl., 2.8.28).—A solution of gum or wood rosin in a light petroleum solvent (petroleum naphtha) is treated with a metal halide, *e.g.*, SnCl<sub>4</sub>, capable of forming an insol. compound with the colour substances in the rosin, and the ppt. thus formed is removed by filtration etc. The metal halide remaining in the solution is removed therefrom, *e.g.*, by precipitation with H<sub>2</sub>O.

S. S. WOOLF.

**Mixed esters of colophony etc.** H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. Co. (U.S.P. 1,783,165, 25.11.30. Appl., 8.10.28).—Glycerol (etc.) is esterified with a mixture of resin acid and a dibasic aliphatic acid between C<sub>6</sub> and C<sub>10</sub>, *e.g.*, colophony (30 pts.), sebacic acid (15 pts.), and glycerin (5 pts.) are heated at 195–260° to give a C<sub>6</sub>H<sub>5</sub>-sol. mixed ester gum which remains flexible at –5°.

C. HOLLINS.

**Manufacture of synthetic resins.** BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. NEWBOUND, and A. T. WARD (B.P. 346,333, 29.11.29).—Resins of the polyhydric alcohol-polybasic acid type, *e.g.*, glycerol-phthalate, are acylated by treatment in a fusible state with an aliphatic or aromatic acyl chloride or acid anhydride, *e.g.*, BzCl, Ac<sub>2</sub>O, excess of the latter substances being subsequently removed by distillation under normal or reduced pressure. Plasticisers and natural or other synthetic resins may, if desired, be incorporated. The time taken to polymerise the resin is reduced and the product is free from cavities or blisters.

S. S. WOOLF.

**Synthetic resin varnishes and synthetic resins.** IMPERIAL CHEM. INDUSTRIES, LTD., A. A. DRUMMOND, and H. H. MORGAN (B.P. 345,276 and 345,310, 13.9.29).—(A) CH<sub>2</sub>O and *m*-xylenol (or its isomerides) or coal-tar distillates (b.p. 216–223°) are heated under normal or increased pressure with or without an acid catalyst, in the absence of alkali, and, if desired, in the presence of excess of a solvent or diluent, *e.g.*, industrial methylated spirit, which is distilled off after the initial reaction is complete. The temp. is then allowed to rise to 120° and maintained thereat until a suitable resinous product is obtained on cooling. This is sol. in a wide range of solvents and may be heated at 170° for 1–2 hr. with a drying oil and converted into varnish by the addition of driers, thinners, etc. (B) Homogeneous synthetic resin products derived from phenols, CH<sub>2</sub>O, and fatty acid esters (> 20% of the final product; cf. B.P. 329,313 [B., 1930, 780]), sol. in alcohol, ketone, or ester solvents but substantially insol. in hydrocarbon solvents, are heated at 110–160°, preferably with continuous removal of solvent vapours liberated, until gelation is imminent (1–2 hr.). The product is then insol. in the original solvents, but sol. in hydrocarbon solvents, *e.g.*, heavy naphtha and, for compositions containing large proportions of oils, white spirit, giving air-drying varnishes with the addition, if desired, of driers.

S. S. WOOLF.

**Production of urea-formaldehyde condensation products.** E. G. BUDD MANUFG. CO., Assecs. of G. L. KELLEY and M. W. DEISLEY (B.P. 319,687, 11.9.29. U.S., 26.9.28).—A mixture of urea,  $\text{CH}_2\text{O}$ , and  $\text{NH}_3$  solution, of  $p_{\text{H}}$  5.2—7.0 is heated under reflux for a short time; thiourac is added to increase the  $\text{H}_2\text{O}$ -repellent properties of the final product. The gelation of the resulting homogeneous fluid is retarded by addition of a salt having a strong basic ion and a weak acid ion, e.g.,  $\text{AcONa}$ , and  $\text{H}_2\text{O}$  is removed, an acid or acid-yielding substance, e.g., formic acid,  $\text{KH}_2\text{PO}_4$ , or  $\text{AlCl}_3$ , being added during removal of  $\text{H}_2\text{O}$  and after the removal of free  $\text{CH}_2\text{O}$ . Heat is applied, if desired, to free the product from bubbles. [Stat. ref.] S. S. WOOLF.

**Phenolic resinous products and compositions containing them.** BAKELITE CORP., Assecs. of V. H. TURKINGTON and W. H. BUTLER (B.P. 347,737, 28.1.30. U.S., 29.1.29).—Hydroxydiphenyls are condensed with methylene-containing substances, e.g.,  $\text{CH}_2\text{O}$ , in the presence, if desired, of fatty oils, e.g., tung oil, giving oil-sol. resins. S. S. WOOLF.

**Fibrous resinous compositions.** BAKELITE CORP., Assecs. of G. E. WIGHTMAN (B.P. 345,332, 17.12.29. U.S., 18.12.28).—Mouldable compositions comprising wood fibres containing approx. 90% (preferably 94% or more) of  $\alpha$ -cellulose, and a potentially reactive resinoid binder, e.g., a phenol-fatty oil-methylene reaction product, are claimed. S. S. WOOLF.

**Manufacture of [resinous] condensation products.** KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 347,228, 24.1.30. Austr., 21.12.29).—Thiodicyanodiamidine ready-formed, or prepared *in situ* by acting on cyanamide or dicyanodiamide with  $\text{H}_2\text{S}$ , is condensed with  $\text{CH}_2\text{O}$  in alkaline solution. The product is neutralised or acidified and further heated, and, if desired, a urea- $\text{CH}_2\text{O}$  or other condensation product is incorporated. The solution is then thickened *in vacuo*, and mixed with fillers etc. S. S. WOOLF.

**[Synthetic resin-]bonded materials.** BAKELITE CORP., Assecs. of V. H. TURKINGTON (B.P. 345,228, 8.11.29. U.S., 10.11.28).—A resinoid binder to be incorporated with a filler, e.g., a woven fabric, and subsequently set by heat without the aid of a polymerising catalyst, has imparted to it a reticulated structure by dispersing in it, before setting, a material, e.g.,  $\text{H}_2\text{O}$ , that is liquid and insol. in the binder or gaseous at the temp. of setting, the volatilisation of this material being controlled so that a small proportion, e.g., 2—6%, is left in the bonded material when set. A hygroscopic substance, e.g.,  $\text{CaCl}_2$  or glycerin, may be added to the  $\text{H}_2\text{O}$  to retard volatilisation until the binder sets. The reticulated structure may be imparted by incorporating with the binder a resinous material immiscible therewith, e.g., a flexible resinous product of the phenol-tung oil type. High impact strength and a minimum of swelling are claimed for the products. S. S. WOOLF.

**Resin plasticiser.** H. A. BRUSON, ASSR. to RESINOUS PRODUCTS & CHEM. CO. (U.S.P. 1,783,166, 25.11.30. Appl., 5.7.29).—A condensation product of a polyhydric alcohol, e.g., glycerol, and pimelic, suberic, azelaic, or

sebacic acid is incorporated with a  $\text{PhOH-CH}_2\text{O}$  resin as plasticiser. S. S. WOOLF.

**Preparation of resin-like plastic masses.** J. BAER (B.P. 345,894, 20.6.30. Ger., 24.6.29).—To a solution of Ca (or other) polysulphide ( $d$  1.07) is added "sulphite-turpentine" or "Tallöl," and the mixture is heated to the b.p. H. ROYAL-DAWSON.

**Moulding powder.** W. H. BOORNE (B.P. 345,472, 10.3.30).—A mixture of rosin (4 pts.),  $\text{CaO}$  (1 pt.), and formalin (4 pts.) is heated in an open vessel and ground. The powdered mixture (450 pts.) is admixed with a cellulose filler or waste, e.g., wood flour (300 pts.), Portland cement (4 pts.), and heavy mineral oil (10 pts.) which has been mixed to paste with  $\text{CaCO}_3$  (5 pts.). For increased hardness and brilliance of surface, hexamethylenetetramine (< 8%) is added, and synthetic phenolic resins may be incorporated with the rosin. S. S. WOOLF.

**Mouldable compositions containing mica.** BAKELITE CORP., Assecs. of C. A. NASH (B.P. 346,349, 8.1.30. U.S., 17.1.29).—Compositions of reactive phenolic resinoids (2 pts.) and a filler (more than 3 pts.), the latter containing 5% or more of finely-divided mica (passing 80-mesh), are claimed. S. S. WOOLF.

**[Liquid] adhesives.** BRIT. CELANESE, LTD. (B.P. 347,445, 1.4.30. U.S., 2.4.29).—A liquid adhesive containing a cellulose derivative, e.g., cellulose acetate, a synthetic resin, e.g., a  $\text{PhOH-CH}_2\text{O}$  condensation product, suitable solvents, and, if desired, a plasticiser, is applied to surfaces to be united. When this coating has become tacky, a finely-divided powder comprising a cellulose derivative, e.g., the acetate, and a plasticiser, e.g., ethyl phthalate, is sprinkled on the surface and the articles are pressed together under increased temp. S. S. WOOLF.

**Manufacture of inlaid linoleum or the like.** ARMSTRONG CORK CO., Assecs. of J. A. PHELAN (B.P. 348,892, 21.5.30. U.S., 22.6.29).

**Nitrocellulose compositions.**—See V. Colouring cellulose esters etc.—See VI. Cementing of surfaces.—See IX. Soaps for paints.—See XII. Nitro-starch.—See XXII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See A., June 683, Degradation of rubber solutions.

##### PATENTS.

**Manufacture of ribbon, sheet, etc. from aqueous dispersions of organic substances, particularly rubber latex.** J. T. SHEVLIN. FROM SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 347,691, 31.1.30).—Heat-stabilised dispersions are forced through a space of the required thickness formed between two plate-like elements heated to the coagulation temp. Fibrous material such as woven or cord fabric may be impregnated by passage together with the dispersion between the plates. D. F. TWISS.

**Treatment of rubber.** NAUGATUCK CHEM. CO., Assecs. of S. M. CADWELL (B.P. 347,955, 23.6.30. U.S., 2.7.29).—The mixture of aliphatic bases of the empirical composition  $\text{C}_{2n+2}\text{H}_{5n+8}\text{N}_{n+2}$  obtainable by the

interaction of an ethylene dihalide and  $\text{NH}_3$ , is used as an accelerator of vulcanisation. The dihalide of a homologue of ethylene, *e.g.*, a propylene dihalide, may be used as an alternative in the primary reaction.

D. F. TWISS.

**Treatment [prevention of scorching] of rubber.** S. M. CADWELL, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,777,874, 7.10.30. Appl., 2.3.28).—Scorching is prevented by the use of acid (chloroacetic, sulphanilic, oxalic, trichloroacetic acids,  $\text{H}_3\text{PO}_4$ , toluene-*p*-sulphonyl chloride, pine tar) in conjunction with a dithiocarbamate derivative hydrolysable by alkali, *e.g.*, 2:4-dinitrophenyl dimethylthiocarbamate, triphenylmethyl ethyl xanthate, 4-chloro-2:6-dinitrophenyl dithiobenzoate, 2:4:6-trinitrophenyl 2-benzthiazyl sulphide, benzylidene dimethylthiocarbamate, etc.

C. HOLLINS.

**Vulcanisation of rubber and accelerator therefor.**

D. H. POWERS, ASSR. to E. I. DU PONT de NEMOURS & Co. (U.S.P. 1,780,149, 28.10.30. Appl., 26.1.26).—Products obtained by the action of an aliphatic aldehyde, particularly  $\text{CH}_2\text{O}$ , on the condensation product of an aliphatic amine and an aliphatic aldehyde, such as  $\text{CH}_2\text{O}$ -ethylamine or heptaldehyde-methylamine, are capable of so aiding vulcanisation that this will occur at  $120^\circ$ ; below  $100^\circ$  their effect is only slight, so that they are free from scorching tendencies.

D. F. TWISS.

**Vulcanisation accelerators.**—See III. Rubber-coated fabrics.—See VI. Road-building material. Floor coverings.—See IX.

## XV.—LEATHER; GLUE.

**Vegetable tanning materials of Erythrea.** G. A. BRAVO (Annali Chim. Appl., 1931, 21, 141—146).—The leaves of *Osyris Abissinica* (Hochst) or Erythrean sumac contain 18.43% of tans of the pyrocatechol group and 16.72% of sol. non-tans; the pods of *Acacia nilotica* (Del) 35.25% of tans; and the pods with (without) seeds of *Casalpinia tinctoria* (Dombey, Benth) tans 45.62 (51.88—55.75), sol. non-tans 20.45 (20.45—22.38), insol. matter 24.85 (14.02—14.12), and  $\text{H}_2\text{O}$  9.08 (9.68)%. Leather tanned with the last of these is very light in colour, resembling that obtained with sumac, and when mordanted with Fe (Ti) assumes a bright black (red) colour.

T. H. PORE.

**Biochemistry of tan-liquor fermentation.** P. D. DALVI (J. Indian Inst. Sci., 1930, 13A, 173—192).—Microflora from tan liquors of avaram bark (*Cassia auriculata*), divi-divi (*Casalpinia coriaria*), and myrobalans (*Terminalia chebula*) were isolated and their physiological characteristics studied. Of the moulds, *Aspergillus niger*, *Penicillium glaucum*, and an unknown *Penicillium* developed strongly even in presence of 10% of tannic acid. Tannase was prepared from *A. niger* grown in an extract of myrobalan powder together with Freudenberg's medium, and purified by precipitation with EtOH and dialysis. The preparation, which was protein-free, hydrolysed Me gallate, the hydrolysis being hastened by addition of  $\text{KH}_2\text{PO}_4$ . The optimum temp. for the enzyme is  $60^\circ$ . Hydrolysis of tannic to gallic acid by tannase is accelerated by presence of  $\text{K}_2\text{HPO}_4$  and by decrease in concentration of substrate and is inhibited by very acid reactions. In checking either the growth

of *A. niger*, *A. luchuensis*, and *P. glaucum* or the loss of tannic acid,  $\text{CS}_2$ ,  $\text{CH}_2\text{O}$ , and paraffin are ineffective, whilst thymol and camphor are highly efficient.

F. O. HOWITT.

See also A., June, 752, Determination of tannic and gallic acids. 777, Tannin of barley husk.

## PATENTS.

**Leather-stretching test.**—See I. Albumose-like products.—See III. Articles having pearl effect.—See V.

## XVI.—AGRICULTURE.

**Soil structure.** M. KRAUSE (Landw. Jahrb., 1931, 73, 603—690).—A comprehensive description and discussion of Russian investigations in connexion with pore space,  $\text{H}_2\text{O}$  relationships, and the physical condition of soils and their effects on crop production.

A. G. POLLARD.

**Elutriation, water capacity, and compression tests in the characterisation of soils.** VON NOSTITZ (Landw. Jahrb., 1931, 73, 407—429).—In a discussion of soil survey results, mechanical analyses are shown to be valuable in soil classification.  $\text{H}_2\text{O}$ -capacity measurements are of little value for this purpose. Compression tests with Puchner's apparatus were made with soil blocks prepared by kneading the sieved soil (2 mm.) with just sufficient moisture to form a compact mass. The blocks were dried at room temp. for 24 hr. and then at  $50^\circ$  until of constant wt. The crushing pressure of the blocks increased with the clay content of the soils. Freezing did not appreciably affect the crushing strength of surface soils, but markedly increased that of subsoils.

A. G. POLLARD.

**Study of the flocculation of a sodium clay soil with the objects of reclaiming sodium clay soils and the amelioration of gault, Kimmeridge and Oxford clays.** E. M. TAYLOR and R. M. WOODMAN (J.S.C.I., 1931, 50, 203—206 r).—The flocculating concentrations of solutions of  $\text{CaSO}_4$  and  $\text{Ca}(\text{OH})_2$  for a Na clay soil have been determined. When flocculation of a suspension of this soil has once been obtained by solutions of these substances, replacement of the liquid medium of the suspension by water, followed by reshaking, is accompanied by reflocculation, the number of such reflocculations depending on the concentration of the original flocculating solution. It is also demonstrated that flocculation can be maintained so long as any water in contact with the sediment contains a small percentage—much less than would flocculate the original soil—of these substances. The bearing of these two findings on the reclamation of "black alkali" soils is discussed.

**Effect of water on the reaction of clay soils.** G. ROTHE (Arb. biol. Reichsanstalt Land- u. Forstwirts., 1931, 18, 425—429).—Liming practice is discussed in relation to fruit tree culture, with special reference to blue clay soils. Fruit trees may be grown successfully in acid soils of good permeability. The compact nature of acid blue clays is unfavourable to root development, but carefully regulated liming is essential. An alkaline reaction in these soils involves the coagulation of colloidal Fe and Al compounds, but other colloids may be dispersed and washed out by  $\text{H}_2\text{O}$ .

A. G. POLLARD.



**Soil acidification by ammonium sulphate.** R. W. BELING (*Landw. Jahrb.*, 1931, **73**, 491—501).—In soils treated with dicyanodiamide to prevent nitrification, the acidifying effect of  $(\text{NH}_4)_2\text{SO}_4$  is small. The definite increase in acidity of normal soils resulting from treatment with  $\text{NH}_4$  salts results directly from the nitrification process.

A. G. POLLARD.

**Soil acidification and superphosphate.** C. KRÜGEL (*Superphosphate*, 1931, **4**, 117—124).—Experimental evidence is quoted to show that the supposed acidity produced in soil by superphosphate treatment is insignificant.

A. G. POLLARD.

**Interrelationships of certain single-valued soil properties.** G. B. BODMAN and E. P. PERRY (*Soil Sci.*, 1931, **31**, 365—378).— $\text{H}_2\text{O}$ -vapour adsorption methods indicated a higher proportion of colloid in soils than that represented by the clay fraction ( $< 2 \mu$  diam.), after  $\text{H}_2\text{O}_2$ -HCl pretreatment. Of the clay fraction nearly 90% of the particles were of  $< 1 \mu$  diam. Pretreatment of soil with  $\text{NH}_3$  yielded much smaller amounts of clay with diam.  $< 1 \mu$  than did the  $\text{H}_2\text{O}_2$ -HCl treatment. Close correlation was found between the following values, arranged in descending order of correlation coeff.: moisture equiv. and sticky point, colloid and moisture equiv., colloid and sticky point, moisture equiv. and non-sticky plastic range, colloid and air-dry moisture content, colloid and clay  $< 2 \mu$ , moisture equiv. and rolling-out limit. Correlations of a lower order were obtained for clay and sticky point, clay and moisture equiv., colloid and rolling-out limit. The sticky point and rolling-out limit should be valuable in characterising field soils. In soils having a moisture equiv. of 9.8% approx., an inversion or coincidence of values for the sticky point and rolling-out limit (normally the lower) may be anticipated.

A. G. POLLARD.

**Soil solution experiments of Wrangell and the views of Němec.** L. MEYER (*Forts. Landw.*, 1930, **5**, 745—748; *Bied. Zentr.*, 1931, **60A**, 70).—Determinations of the available P in soil cannot be based on a single extraction with  $\text{H}_2\text{O}$  (Němec) since the proportion of the total P thus removed varies with the adsorptive power of the soil. Moreover, more P is removed if the extraction is prolonged beyond the stipulated 30 min.

A. G. POLLARD.

**Solubility of the adsorptively bound bases in soils in relation to their lime condition and degree of saturation.** II. A. GEHRING, U. CREUZBURG, E. POMMER, O. WEHRMANN, A. WOLTER, and H. VON STOCKHAUSEN (*Z. Pflanz. Düng.*, 1931, **20A**, 183—217; cf. B., 1931, 455).—The action of Mg salts on crop yields varies with the degree of saturation of the soil with Ca, being most marked where the Ca saturation is 20—25% and decreasing as the value is raised. The solubility of soil potash also depends on the degree of Ca saturation. This effect is masked in pot cultures, but is apparent in the field. Difficulties attending potash-requirement determinations by means of pot cultures are discussed.

A. G. POLLARD.

**Composition of the soil solution and the growth of plants in very dilute solutions.** M. VON WRANGELL (*Ergeb. Agrik.-Chem.*, 1930, **2**, 9—20; *Bied. Zentr.*, 1931, **60A**, 81).—Interrelationships between the com-

position of soil solutions and plant growth are examined and discussed.

A. G. POLLARD.

**Determinations of  $p_H$  in soil suspensions.** C. DREYSPRING and F. HEINRICH (*Z. Pflanz. Düng.*, 1931, **20A**, 155—169).—Details of the technique for determining  $p_H$  values in aq. and KCl soil suspensions are recommended. The sample (10 g. passing 2-mm.) is shaken in a hard glass tube with 10 c.c. of  $\text{H}_2\text{O}$  in a rotary apparatus (40 r.p.m.) for 15 min. Quinhydrone (50 mg.) is added to the tube and the measurements are made 3—5 min. later.

A. G. POLLARD.

**Application of soil analyses.** I. J. BEHR and R. KÖHLER (*Mitt. Lab. Preuss. Geol. Landesanst.*, 1930, No. 11, 15—34; *Chem. Zentr.*, 1931, **i**, 1346).—A discussion of the results of elutriation analyses and the differentiation of soil classes and constituents.

A. A. ELDRIDGE.

**Nutrient content of soils.** K. NAUMANN (*Diss.*, T.H., Dresden; *Bied. Zentr.*, 1931, **60A**, 66—67).—The logarithmic nature of Mitscherlich's growth curves and the constancy of growth factors in respect of K for various crops and under different climatic conditions are confirmed. The effect factor for K based on the K intake of plants agreed with that calc. from the K applied, but the value was lower than that adopted by Mitscherlich. A logarithmic relationship in respect of P was not observed and the effect factors from different series of experiments showed poor agreement. The effect factor for P, calc. from the amount of P applied, was less than that indicated by the P intake of plants. In Neubauer tests the whole of the available P and K is not removed by one crop of barley. In a comparison of methods the values for K were in the order: Neubauer  $>$  Weissmann  $>$  Mitscherlich. Values for P were less divergent, except in Neubauer tests, where values were markedly low.

A. G. POLLARD.

**Biochemical determination of the nutrient content of soils.** H. NIKLAS (*Ergeb. Agrik.-Chem.*, 1930, **2**, 21—34; *Bied. Zentr.*, 1931, **60A**, 67).—Christensen's *Azotobacter* method and Neubauer's seedling method for determining nutrient contents show close agreement in soils of low P content, diverged somewhat in soils of moderate P content, and were reasonably concordant in rich soils. In very heavy soils the *Azotobacter* test was less satisfactory. A method described for determining the assimilable P and K in soil by means of *Aspergillus* gave more const. results for K than for P.

A. G. POLLARD.

**The *Aspergillus* method [of determining soil nutrients].** H. SÖDING (*Z. Pflanz. Düng.*, 1931, **20A**, 129—130).—The method of Niklas and co-workers (B., 1931, 37) is criticised. The preparation of the samples for examination and the  $p_H$  changes involved are discussed.

A. G. POLLARD.

**Determination of easily soluble nutrients in soils by electro-ultrafiltration.** R. DIEHL (*Ernähr. Pflanze*, 1930, **26**, 453—456; *Bied. Zentr.*, 1931, **60A**, 124).—Electro-ultrafiltration methods offer a satisfactory means of determining the proportion of sol. nutrients in soils and also yield information as to the rate at which nutrients become sol. The "easily-sol." portion of the soil reserves may thus be differentiated.

A. G. POLLARD.

**Photosensitised oxidation of ammonia and ammonium salts and the problem of nitrification in soils.** G. G. RAO and N. R. DHAR (Soil Sci., 1931, 31, 379—384).—Photochemical oxidation of  $\text{NH}_3$  to nitrate occurs in the presence of sensitisers. The activity of sensitisers examined was in the order  $\text{TiO}_2 > \text{ZnO}, \text{CdO} > \text{Na}_2\text{U}_2\text{O}_7 > \text{Al}_2\text{O}_3 > \text{SiO}_2$ . Alkalinity favours oxidation and *vice versa*. A. G. POLLARD.

**Determination of the assimilable nitrogen in soils by means of grass seedlings.** R. WAGNER (Wiss. Arch. Landw., 1930, 5A, 166; Bied. Zentr., 1931, 60A, 80—81).—Western ryegrass may be used for determining the available N content of soils under conditions similar to those of Neubauer tests. Utilisation of nitrates and  $\text{NH}_4$  salts by ryegrass is dependent on the reaction of the medium. Fresh, moist soil samples must be used, since drying under any conditions leads to changes in the N content of the soil. Results do not agree with those obtained by König's method.

A. G. POLLARD.

**Soils. VIII. Assimilable potassium. IX. Calcium and the soil.** J. CLARENS and H. MARGULIS (Bull. Soc. chim., 1931, [iv], 49, 380—384, 384—389; cf. B., 1930, 294).—VIII. It is shown that solubilisation of K in soils occurs in definite stages, and an attempt is made to correlate these with the requirements of the plant during the stages of its growth. The total alkali metals were determined as chlorides in the usual way in extracts of 4 samples of the soil in solutions containing suitably increasing quantities of acid (*e.g.*, for a soil containing 3%  $\text{CaCO}_3$ , 0.25, 0.5, 0.75, and 1.0 c.c. of 0.1N-HCl per g. respectively).

IX. On the basis of previous work,  $\text{CaCO}_3$  in a soil poor in humus may be determined from the change in level of a Hg manometer attached to a closed vessel containing 5 g. of soil with an excess of dil.  $\text{HNO}_3$ . Details are also given for the determinations of acidic functions actually saturated by so-called displaceable bases, and free acidic functions. The quantity of  $\text{CaCO}_3$  necessary for recalcification is found by agitation of the soil with saturated  $\text{CaSO}_4$  solution, or with an equiv. solution of  $\text{CaCl}_2$ . Solid  $\text{CaCO}_3$  recalcifies only very slowly on account of its insolubility (cf. B., 1930, 474, 629); examples are quoted. Acidic functions are liberated and appreciable quantities of bases displaced by treatment with HCl in concentrations less than that required for saturation of the  $\text{CaCO}_3$ , the whole of the bases being displaced by excess of acid.

J. GRANT.

**Determination of the lime requirement of soils on the basis of their hydrolytic acidity.** J. VON CSIKY (Z. Pflanz. Düng., 1931, 20A, 142—155).—Addition to soils of the CaO required to neutralise hydrolytic acidity as calc. by Woltersdorf (B., 1930, 734) does not remove acidity completely. Relationships between the author's lime-water method for determining CaO requirement (B., 1930, 256); the limiting values of Vageler and Woltersdorf (B., 1930, 295); the (*T*—*S*) value, of Hissink; and the presence of free  $\text{CaCO}_3$  in soils, are discussed.

A. G. POLLARD.

**Phosphate requirement of soils in relation to soil type.** E. LAUTERBERG (Z. Pflanz. Düng., 1931,

20A, 302—356).—Discrepancies in the results of soil-nutrient determinations by various methods may depend, in part, on the physical composition of the soil, and such results should be considered in conjunction with mechanical analyses. The phosphate content of soils is largely concentrated in the finer particles, there being practically none in particles of  $>0.05$  mm. diam. The adsorptive power of soils for phosphate varies with the nature of the phosphate applied. Soils containing much assimilable phosphate (Neubauer) have a low adsorptive power and *vice versa*.

A. G. POLLARD.

**Soil examination and the use of artificial fertilisers.** H. VAGELER (Z. Pflanz. Düng., 1931, 10B, 238—253).—Field-trial results are utilised in a general discussion of the rational use of fertilisers.

A. G. POLLARD.

**Effect of artificial nitrogenous fertilisers on the occurrence and development of *Azotobacter chroococcum* in soils.** E. SCHNEIDER (Arch. Pflanz. enb., 1931, 5, 304—373; Bied. Zentr., 1931, 60A, 70).—Application to soil of nitrogenous fertilisers, especially  $\text{NaNO}_3$  and, to a smaller extent, of urea, favours the growth of *A. chroococcum*.  $(\text{NH}_4)_2\text{SO}_4$  is not effective until nitrified. Continuous dressings of  $(\text{NH}_4)_2\text{SO}_4$  reduce the development of *Azotobacter*.

A. G. POLLARD.

**Effect of increasing applications of nitrogen on pure and mixed herbage plants of meadows and pastures.** J. VASTERS with T. REMY (Landw. Jahrb., 1931, 73, 521—602).—The growth of grasses, clovers, and weeds was affected to different proportional extents by increasing applications of nitrogenous fertilisers. No relationships were apparent between the effects on plants of similar families. The yield and chemical composition of hay was mainly the result of the different botanical composition of the herbage brought about by fertiliser treatment. Nitrogenous fertilisers increased the crude protein content of all non-legumes, but simultaneously depressed the yield of legumes. The protein content of the mixed herbage depended on the balance of these two effects and, in general, increased with the heavier applications of fertiliser. The proportion of root to top growth increased with fertilisation, but the N content of the root was lower than that of the green matter.

A. G. POLLARD.

**Effect of potash on the stiffness of cereal straws.** F. BERKNER and W. SCHLIMM (Landw. Jahrb., 1931, 73, 503—520).—Applications of potash fertilisers to soils had no consistent effects on the potash content, diam., and tensile strength of oat and barley straws grown in them. No relationship between the  $\text{SiO}_2$  content and bending strength of straw was observed. Applications of potash fertilisers reduced the  $\text{SiO}_2$  content of straw. CaO applied with small dressings of potash fertilisers increased the bending strength of straw, but phosphatic fertilisers had no very definite effect in this respect. In most cases potash manures increased the length and thickness of the vascular bundles and thickened the sclerenchymatous tissue. Kainit produced a considerable increase in the number of vascular bundles.

A. G. POLLARD.

**Hay and silage.** E. BROUWER (Vereen. Exploit. Proofzuivel. Hoorn, 1929, 1—34; Chem. Zentr., 1931,

i, 1378).—Losses are recorded and feeding experiments described.

A. A. ELDRIDGE.

**Characteristics of various methods of utilising peat in manure making in the Schenkursk district of the northern area.** A. A. KUPRIANOV and N. S. ROSANOV (Arb. Zentr. Torfstat., 1930, 2, 41—60; Bied. Zentr., 1931, 60A, 74).—Where peat is trampled in cattle pens to a depth of about 1 m. the exclusion of air from the lower layers has little effect on the properties of the final product since only the surface layer undergoes change. Where aeration of the peat is maintained, the penetration of dung and urine is more thorough and the product has a lower  $H_2O$ -holding capacity. The best peat manure is obtained by filling successive layers of mossy peat into the pens. Good-quality manure is produced by composting moor soil with dung from the stalls. Peat manuring increases the bacterial numbers of soils to a greater extent than does straw manuring.

A. G. POLLARD.

**Conservation and availability of the nitrogen in farmyard manure.** A. F. HECK (Soil Sci., 1931, 31, 335—363).—During the storage of farmyard manure under anaerobic conditions  $H_2O$ -sol. N compounds are transformed into  $NH_3$ , which combines with the org. acids produced by acid fermentation. Losses of N from manure during handling and storage may amount to 50% of the total, or the whole of the liquid-manure N, and are due to the volatilisation of free  $NH_3$  in the early drying period and of  $NH_4$  salts in the later stages. Changes in  $p_H$  during acid fermentation are directly proportional to the amount of cellulosic (energy-producing) material present. When liquid manure is stored its N compounds are steadily transformed into  $(NH_4)_2CO_3$ , which may be lost by volatilisation. Covering the liquid with oil prevents this loss even in open cisterns. The use of straw litter in manure reduces N losses during drying, but also reduces its availability. Available N in manure may be expressed as the total  $H_2O$ -sol. N less the loss during handling and storage.

A. G. POLLARD.

**Nitrogen losses and action of cold- and hot-fermented manures.** D. MEYER (Z. Pflanz. Düng., 1931, 10B, 253).—A correction (cf. B., 1931, 558).

A. G. POLLARD.

**Manuring of meadows.** EICHINGER (Superphosphat, 1931, 7, 83—91).—The proportion of mineral nutrients applied to soil which are recovered in successive cuttings of meadow herbage is examined. No appreciable loss of nutrient is incurred by applying the whole dressing of phosphates and potash in the spring. Nitrogenous fertilisers are better applied in two portions, one in spring and the other following the first cutting. The value of applications of farmyard manure and of  $CaO$  are discussed. Superphosphate is superior to basic slag on low-moor soils.

A. G. POLLARD.

**Effect of phosphate manuring on the yield and quality of malting barley.** C. DREYSPRING, H. KURTH, and F. HEINRICH (Z. Pflanz. Düng., 1931, 10B, 217—237).—In field trials with numerous varieties of barley, applications of superphosphate increased the grain : straw ratio and the starch content of the grain, decreased the protein content of the grain, and acceler-

ated ripening. The extent of these effects varied considerably with the variety examined.

A. G. POLLARD.

**Increased yield of starch from potatoes by phosphate manuring.** OSKJERSKI (Superphosphat, 1931, 7, 102—103).—Numerous field trials are recorded in which applications of superphosphate increased the yield and starch content of potatoes and, more consistently, the yield of starch per acre.

A. G. POLLARD.

**Analogy between the course of growth and nutrient intake of oats and physico-chemical laws.** II. WAGNER (Landw. Jahrb., 1931, 73, 453—490).—Pot cultures of barley in which varying depths of soil, but the same nutrients, were used showed that the dry matter yields increased with the depth of soil, gradually approaching a maximum value. The percentage nutrient content of all plants remained practically identical. The ratio of nutrients ( $K_2O : P_2O_5 : N = 1$ ) in the green matter of the plants widened as the flowering stage was approached. Assimilation of potash, phosphate, and N by the plant increase the rate of production of organic matter; Ca had but little effect. Mathematical expression of growth processes must be based on an exponential junction. The S-form of growth curve observed, e.g., in the K intake, results from a temporary limitation of the growth process. Various mathematical treatments of growth curves are examined.

A. G. POLLARD.

**Premature heading of cauliflower as associated with the chemical composition of the plant.** W. R. ROBBINS, G. T. NIGHTINGALE, and L. G. SCHERMERHORN (New Jersey Agric. Exp. Sta. Bull., 1931, No. 509, 14 pp.).—Premature "heading" of cauliflower was produced in sand cultures by restricting the supply of N in the nutrient. In comparison with vigorous plants those with "button" heads had high carbohydrate content and low N contents of which the proportion present as protein was abnormally high.

A. G. POLLARD.

**Biology of the potato.** F. MERKENSCHLAGER. VI. **Ecological deterioration of the potato.** II. WARTENBERG (Arb. biol. Reichsanstalt Land- u. Forstwirtschafts., 1931, 18, 405—423).—Symptoms typical of ecological deterioration in potatoes (leaf curl, mosaic, etc.) are associated with a high sol. salt concentration in the soil and in particular with heavy potash manuring. Affected plants frequently have high ash contents and a high potash content in the ash. The bearing of these results on the ultimate cause of deterioration is discussed.

A. G. POLLARD.

**Protecting stored grain from insects by the use of oils.** W. P. FLINT (J. Econ. Entom., 1931, 24, 425—427).—Successful results obtained by the use of standard and proprietary oil emulsions are described. A thin film of oil over the surface of the grain prevents the entry of newly hatched larvae.

A. G. POLLARD.

**Lethal power of certain insecticides against the European corn borer.** F. L. SIMANTON, F. F. DICKE, and G. T. BOTTGER (J. Econ. Entom., 1931, 24, 395—404).—Trials with arsenates, pyrethrum, nicotine, fluosilicates and numerous other F compounds are described. F compounds proved the most satisfactory,

although Na and Ba fluosilicates caused serious injury to the grain. The use of carriers, adhesives, spreaders, activators, etc. did not appreciably improve the insecticides. A. G. POLLARD.

**Summer oil [insecticide] for the control of European elm scale (*Gossyparia almi*, L.).** C. R. CLEVELAND (J. Econ. Entom., 1931, 24, 349—355).—Successful trials with 2% white-oil emulsions are described. No foliage injury was observed. A. G. POLLARD.

**Spray-residue removal.** D. F. FISHER (J. Econ. Entom., 1931, 24, 526—531).—A discussion of recent developments. A. G. POLLARD.

**Eradication of prickly-pear by chemicals, with particular reference to emulsions in the systems gelatin-aqueous arsenic acid.** R. M. WOODMAN and W. J. WILEY (J.S.C.I., 1931, 50, 187—189 t).—It is argued that when spraying for the eradication of prickly-pear, emulsions containing aq.  $H_3AsO_4$  solutions and the cresols as liquid phases with gelatin or glue as the emulsifier, should preferably be of the aq.  $H_3AsO_4$ -cresols type. The  $H_3AsO_4$  present is demonstrated to favour this desirable type, any tendency to formation of the opposite type possessed in the absence of this acid being thus suppressed.

**Cottonseed.**—See XII. **Sugar beet.**—See XVII. **Sewage sludge as manure. Control of the house fly.**—See XXIII.

See also A., June, 699, **Glass electrode for  $p_H$  determinations.** The "pehameter." 705, **Kjeldahl N distillation.** 708, **Air in low-moor soils. Hungarian lowland soils. Soil structure in N. Germany.** 769, **Nitrifying bacteria. N cycle in forest soils. Decomposition of carbamide in soil.** 774, **Petroleum oils and the respiration of bean leaves. Permeability to iodine of vegetable plants.**

#### PATENTS.

**Manufacture of mixed fertilisers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,991, 14.4.30. Addn. to B.P. 326,529; B., 1930, 475).—Similar results to those claimed in the prior patent are obtained by adding up to 6% of  $H_2O$ -sol. Mg salts to the liquid mixture containing  $NH_4NO_3$ . H. ROYAL-DAWSON.

**Non-hygroscopic seed disinfectant.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,776,423, 23.9.30. Appl., 6.2.28).—A mercuriated phenol (*o*-chloro- or *o*-nitro-phenol) or acid etc. (benzoic anhydride) is mixed with an alkaline-earth oxide or hydroxide and an alkali salt which can react with the latter to give an insol. salt (Na oxalate, phosphate, sulphate, fluoride). C. HOLLINS.

**Weed-destroying compositions.** G. LITSCHKE (B.P. 344,701, 22.4.30).—Cryst.  $CuSO_4$  (1 pt., powdered) is made into a paste with  $H_2SO_4$  (d 1.70, 3 pts.).

H. ROYAL-DAWSON.

**Sol. cellulose esters.**—See V.

#### XVII.—SUGARS; STARCHES; GUMS.

**Sampling, analysis, and composition of sugar beet.** G. MILNE, H. T. JONES, and J. S. WILLCOX (J.S.C.I., 1931, 50, 155—160 t).—Experiments were made to

investigate the accuracy of various methods for the sampling and analysis of sugar beet, and the effect of such factors as size, exposure of the upper part of the root above the soil, clamping, and fanginess on the composition of beet. The following conclusions are reached. The standard error calculated from 212 duplicate determinations of sugar in beet by the Pellet hot-water process is  $\pm 0.136$ . Except in the case of small crops, a sample of sugar beet to be representative should contain at least 50 roots. The use of a boring tool for obtaining material for pulping from a field sample is unreliable. The juice-and-factor method leads to higher results than those obtained by the pulp method. The possible causes are discussed. "Bolters" are significantly lower in sugar and in dry matter contents than are normal roots. "Crowns" constituting about 13% of the entire root are much inferior in sugar and richer in N, fibre, and ash than is the rest of the root. Small beet are definitely richer in sugar and dry matter than larger beet grown under identical conditions. Evidence is adduced to show that the factory method of sampling a bulk is inaccurate. Greening of the upper part of the root due to exposure above the soil does not materially affect the composition. Development of fangs on beet is not accompanied by a significant alteration in the sugar content.

**Rapid determination of sugar in exhausted [beet] slices.** V. STANEK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1930, 55, 175—182).—By passing 0.5—0.75 kg. of the slices through a modified hand fruit-press ("Cosmos"), rejecting the first 50 c.c. of juice, and polarising the next 100 c.c. after clarification with 0.75 g. of a well-ground mixture of 1 pt. of fine CaO with 25 pts. of neutral  $Pb(OAc)_2$  (cheap substitute for dry basic Pb acetates), results are obtained in good agreement with those by official methods and in a much shorter time, viz., 9 min. J. H. LANE.

(A) **Conductometric formula for determining ash in both raw and refinery syrups and molasses.** (B) **Ash and electrical conductivity of refined cane sugars.** F. W. ZERBAN and L. SATTLER (Ind. Eng. Chem. [Anal.], 1931, 3, 38—40, 41—43. Cf. B., 1928, 422; 1930, 387, 925).—(A) The formula proposed is:  $\text{ash (\%)} = 0.0191369 K - 0.002249 K_2 - 0.001210 K_3 + 3.07$ , in which  $K$  is the sp. conductivity of a solution containing 0.5 g. of the sample plus 4.5 g. of pure sucrose per 100 c.c., and  $K_2$  and  $K_3$  are those of similar solutions treated with 5 c.c. of 0.25*N*-KOH and 5 c.c. of *N*- $H_3PO_4$  respectively, per 200 c.c. With 133 samples, including one of beet molasses, of ash contents 4.84—14.23%, this formula gave a max. deviation from the chemical ash (sulphated minus  $H_2O$ ) of 0.3 and an average deviation of 0.134. A nomograph for simplifying calculations is illustrated.

(B) On the basis of 34 samples tested, from 4 refineries, it is concluded that for granulated and remelt cane sugars containing up to 0.3% of ash, in solutions of 25 g./100 c.c., the sp. conductivity, corr. for that of the  $H_2O$  and multiplied by the  $C$ -value 530, agrees satisfactorily with the chemical ash content. With soft sugars (41 samples containing 0.18—2.31% of ash were tested, from 3 refineries) at a concentration of

5 g. (or 2.5 g. plus 2.5 g. of sucrose) per 100 c.c. it is necessary to use different  $C$ -values for different refineries or districts, or the following general formula may be used:  $\text{ash (\%)} = 0.001695(0.913K + 193.5 - 0.1K_1)$ , in which  $K$  is the sp. conductivity of a solution of 5 g. of the sample in 100 c.c., and  $K_1$  that of a similar solution treated with 5 c.c. of 0.25*N*-HCl per 200 c.c. The authors conclude with some practical recommendations for equipment and methods to be used in routine work on all types of cane products.

J. H. LANE.

**Effect of amino-acids on rotation of glucose [dextrose] and fructose [lævulose] and its significance to determination of sucrose by double-polarisation methods.** D. T. ENGLIS and F. A. DYKINS (Ind. Eng. Chem. [Anal.], 1931, 3, 17—21).—The combination between dextrose and amino-acids in alkaline solutions (A., 1926, 151, 714, 1061) may occur to some extent in solutions of sugar products clarified with basic Pb acetate, and affect the determination of sucrose. Mixtures of equal vols. of 0.0666*M*-dextrose and glycine solutions buffered at  $p_H$  8.11, 9.52, and 10.3 lost, respectively, 2.77, 16.66, and 22.22% of their rotatory power in 48 hr. at 25°, and their iodometric values decreased to about the same extent. With asparagine in place of glycine similar slow changes were observed, depending on the alkalinity, but the falls in rotation and iodometric value did not correspond. The changes produced in solutions of low alkalinity are very slowly reversed on acidification, but at higher alkalinities the reversibility becomes incomplete. At very low temp. the interaction of dextrose and glycine is much restricted, whilst at 50° it is relatively rapid and non-reversible and marked discoloration occurs. Lævulose showed no optical change in presence of glycine and asparagine with increasing  $p_H$  values, which could not be attributed to the alkalinity alone.

J. H. LANE.

**Determination of glucose [dextrose] in presence of fructose [lævulose] and glycine by the iodometric method.** F. A. DYKINS and D. T. ENGLIS (Ind. Eng. Chem. [Anal.], 1931, 3, 21—23).—In the iodometric determination of dextrose in presence of amino-acids the oxidation of the sugar is too rapid for any appreciable condensation with the amino-acid to occur (cf. preceding abstract). Low results from this cause are likely only if the sugar solution has remained alkaline for some time before the analysis. Amino-acids and lævulose both tend to produce positive errors, which are greatest when the alkalinity is highest and due to NaOH alone. Of the various alkaline agents employed with I, phosphate buffer is preferred. The authors add 10 c.c. of 25%  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  and 5 c.c. of 0.5*N*-NaOH to a mixture of 25 c.c. of sugar solution and 25 c.c. of 0.1*N*-I, and leave in a stoppered bottle for 1 hr. before acidifying.

J. H. LANE.

**Measurement of consistency of starch solutions.** J. C. RIPPERTON (Ind. Eng. Chem. [Anal.], 1931, 3, 152—154).—To measure the swelling of starch without rupturing the granules, nearly boiling  $\text{H}_2\text{O}$  is allowed to drop gently on to a suspension of the starch grains in cold  $\text{H}_2\text{O}$ . The degree of swelling cannot be taken as a direct measure of the viscosity, but the viscosity is

proportional to the swelling power multiplied by the concentration, and a given starch may therefore be evaluated without the use of a viscosimeter. The method is particularly applicable to tuber starches having a high swelling power, such as those of potato, sago, and arrowroot.

E. S. HEDGES.

**Starch in cereals. Fodder for milk goats.**—See XIX.

See also A., June, 691, Oxidation of carbohydrates by air. 703, Sodium light for refractometry. 776, Carbohydrate in root of *Arctium lappa*. New sources of inulin. Sorbitol in Norwegian rowan berries. 699, Glass electrode for  $p_H$  determinations.

PATENTS.

**Cellulosic product.**—See V. Yeast.—See XVIII.

### XVIII.—FERMENTATION INDUSTRIES.

**Proteolytic enzymes of malt preparations.** K. A. TISSUE and C. H. BAILEY (Cereal Chem., 1931, 8, 217—226).—Removal of the proteolytic enzymes from diastatic malt extracts by treatment with safranine increases their bread-improving qualities.

E. B. HUGHES.

**Fermentation of millet.** A. HEIDUSCHKA and P. DANTSCHKEFF (Pharm. Zentr., 1931, 72, 337—339).—A very thick mash of finely-ground millet is boiled and cooled several times and inoculated with an infusion of ground chick-pea and air-dried embryos of millet. After keeping overnight at room temp., the diluted mash is filtered through wire gauze and allowed to ferment further. The resulting yellowish-brown beverage in which are suspended the finest particles of the millet is termed "Bosa" and contains at the end of 13 days 0.515 wt.-% of EtOH, 0.493% of lactic acid, and 0.0975% of AcOH.

C. RANKEN.

**Formation, presence, and detection of  $\beta\gamma$ -butylene glycol in wine and cider.** J. PRITZKER and R. JUNGKUNZ (Ann. Falsif., 1931, 24, 139—144).— $\beta\gamma$ -Butylene glycol is present as a by-product in alcoholic fermentation. When oxidised by Br in  $\text{Na}_2\text{CO}_3$  solution,  $\beta\gamma$ -butylene glycol forms diacetyl, which may be distilled off in presence of  $\text{FeCl}_3$ . A sensitive test for diacetyl is to in distil into a mixture of  $\text{NH}_4\text{OH}$ , HCl, NaOAc, and  $\text{NiCl}_2$ , when a ring of Ni dimethylglyoxime is formed. The reaction may be made quantitative, but only one third of the glycol is oxidised to diacetyl. The production of  $\beta\gamma$ -butylene glycol is considered to be characteristic of an alcoholic fermentation.

E. B. HUGHES.

**"Casse" of white wines.** R. MARCILLE (Ann. Falsif., 1931, 24, 167—168).—The presence of Fe is not the usual cause of "casse" in white wine, as is usually supposed, but it is due to  $\text{H}_3\text{PO}_4$  present. The ppt. is shown to consist of  $\text{Ca}_3\text{PO}_4$  and protein matter.

E. B. HUGHES.

**Fluorescent indicators for the determination of free acidity of red wines.** Y. VOLMAR and J. M. CLAVERA (Anal. Fis. Quím., 1931, 29, 247—254).—Comparative experiments have been made with umbelliferone and acridine and with ordinary indicators for the

determination of the free acidity of wines of which the colour obscures the end-point, the potentiometric method being used to establish the true end-point. Titration to  $pH$  7 may be carried out by the use of 5–6 drops of a 0.001% alcoholic solution of umbelliferone in 100 c.c. of a solution containing 4 c.c. of the wine; the error is 0–3%. With phenolphthalein the results are high (5–30%), and with acridine low (7–15%).

H. F. GILLBE.

**Composition of Morocco wines.** L. CHAUVEAU and A. VASSEUR (Ann. Falsif., 1931, 24, 152–164).—Analyses are given. It is concluded that in 1930 26% of the samples did not conform to the usual standards. Analyses of samples taken in 1926–1929 are also discussed.

E. B. HUGHES.

**Tan liquor fermentation.** See XV.

See also A., June, 759, **Urease**.

#### PATENTS.

**Production of yeast.** A. BRAUN, J. and E. FISCHL (M. FISCHL'S SÖHNE), and F. ROSENBERG (B.P. 346,361, 4.12.29. Austr., 18.12.28).—Yeast is propagated in a series of fermentations directly following one another. Apart from the first phase in which seed yeast is used, the subsequent phases are seeded with the yeast crop from the preceding phase. The dilution of the molasses solution and the volume of the fermentation liquid increase with each phase, together with a corresponding increase in the amount of aeration. Each phase is supplied with sufficient nutrients and the propagation carried on until the sugar has been consumed as completely as possible. The quantities of sugar and nutrient required for the amount of yeast yielded by any phase are calc. by means of a growth factor, which is constant for the second and following phases, but slightly lower for the first phase.

C. RANKEN.

**Increasing the fermenting power of yeast.** H. WINDESHEIM and F. W. THIELE (B.P. 347,542, 2.7.30).—Cholesterol or a similar compound in the form of an emulsion with fat is added with phosphates to yeast. Brewer's yeast is stored before treatment in dil. malt extract and purified with charcoal.

C. RANKEN.

**Production of absolute alcohol from unpurified spirit.** G. B. ELLIS. From E. MERCK (B.P. 347,268, 24.1.30).—Raw spirit and an entraining liquid are distilled with or without pressure in a dehydrating column. The entraining liquid is separated from the aq. EtOH and low-boiling impurities in the azeotropic distillate and is returned to the dehydrating column with the EtOH obtained by rectification of the alcoholic layer of the distillate. The abs. alcohol and high-boiling impurities are removed from the dehydrating column and separated from each other and from traces of the entraining liquid in two further columns.

C. RANKEN.

**Rectification of alcohol.** SOC. ANON. DES. DISTILLERIES DES DEUX-SÈVRES, E. RICARD, and H. M. GUINOT (B.P. 347,122, 18.11.29).—Liquid low in EtOH content is heated in a small auxiliary column so that less than 3% of the feed liquid is vaporised. Impurities insol. or only slightly sol. in  $H_2O$  are thus eliminated before the "refined wort" enters the main distillation column.

C. RANKEN.

**Treatment [concentration] of ethyl alcohol.** C. HANER, Assr. to PUBLICKER, INC. (U.S.P. 1,783,086, 25.11.30. Appl., 3.10.28).—NaOH or KOH (e.g., 0.8 lb./gal.) is added to the aq. EtOH, which is distilled; 98% or more conc. EtOH is recovered in the first stages. Distillation is discontinued at a point (90–98% of total EtOH recovered) when the still residue remains liquid when hot, but solidifies on cooling to room temp.

E. LEWKOWITSCHE.

**Dye preparation.**—See IV. **Cheese.**—See XIX.

#### XIX.—FOODS.

**Determination of starch in cereal products.** C. W. HERD and D. W. KENT-JONES (J.S.C.I., 1931, 50, 15–22 T).—Methods of starch determinations are classified broadly into two classes: (1) hydrolytic method; (2) non-hydrolytic method. Many of the methods in each group are examined and discussed and it is suggested that a modification of Rask's method is very suitable for cereal work. Results of application to commercial starches, commercial flours and mill stocks, wheat offals, and whole wheats are given. Reference is made to the uncertainty of the constituents of starch, and it is suggested that discrepancies in various methods are due to the inclusion or exclusion of one or other of these constituents.

**Hygroscopicity of wheat.** L. PAP (Cereal Chem., 1931, 8, 200–206).—A tabulation of the hygroscopicity of Hungarian and other foreign wheats, showing the deviations in moisture content of samples stored under the same conditions.

E. B. HUGHES.

**Fermentation period of Northwest and Pacific Northwest flours as indicated by carbon dioxide production and dough expansion.** J. L. ST. JOHN and M. HATCH (Cereal Chem., 1931, 8, 207–216).—Owing to complicating factors, the application of the Bailey-Johnson method (B., 1925, 111) is limited to determining the effect of added ingredients on fermentation.

E. B. HUGHES.

**Relation of wheat protein to baking quality. II. Saskatchewan hard red spring wheat crop of 1929.** R. K. LARMOUR (Cereal Chem., 1931, 8, 179–189; cf. B., 1930, 583).—A statistical study of results of baking tests on wheats of greatly varying protein content. The correlation factor between protein content and loaf vol. is high for the  $KBrO_3$  and blend-bromate methods, the latter giving better results with high-protein flours.

E. B. HUGHES.

**Wheat proteins in relation to peptisation and baking strength.** R. H. HARRIS (Cereal Chem., 1931, 8, 190–200).—Peptisation of wheat protein by inorg. salt solutions gives results comparable with those obtained on flour.

E. B. HUGHES.

**Mildly toasted wheat germ—a possible merchandisable table cereal.** R. HERTWIG (Cereal Chem., 1931, 8, 226–228).—Wheat germ roasted at 120–130° to golden-brown keeps well and has a pleasant flavour.

E. B. HUGHES.

**Toasted wheat germ self-rising flour—a possible merchandisable cereal.** R. HERTWIG (Cereal Chem., 1931, 8, 229–231).—Toasted wheat germ is

blended with flour, baking powder, salt, and cerclose (dextrose) for use in cakes and biscuits.

E. B. HUGHES.

**Use of oxygen in the quick determination of ash [in flour].** R. B. PORRIS (Cereal Chem., 1931, 8, 232—233).

—The flour is burned in an open furnace at 427° and transferred to an airtight furnace at 593° into which O<sub>2</sub> is introduced. The total time occupied is 1½—2½ hr.

E. B. HUGHES.

**Fermentation and proofing cabinet [for baking tests] giving low temperature variability.** R. K. LARMOUR, F. D. MACHON, and S. F. BROCKINGTON (Cereal Chem., 1931, 8, 233—241).—A cabinet of the rotating-shelf type with a temp. variation of not more than ± 0.5° is described and scale diagrams are given.

E. B. HUGHES.

**F.p. of milk and its applications.** G. D. ELSDON and J. R. STUBBS (J.S.C.I., 1931, 50, 135—141 r).—The authors review the published literature on this subject, showing that the f.p. is the most const. of all the properties of cows' milk and therefore the most useful for detecting added water. The theoretical and practical aspects of the subject are discussed and a detailed description of the Hortvet apparatus is given. From their own results the authors show that up to the present the indications obtained by the process have not once been in error. An extensive bibliography is given.

**Acido-proteolytes and thermophiles in the pasteurisation of milk.** C. GORINI (Atti R. Accad. Lincei, 1931, [vi], 12, 519—593).—Milk pasteurised for 30 min. at 63° and at once cooled to 5°, bottled, and stoppered mostly kept good at room temp. (in Milan) for 42—48 hr., but during the summer of 1930, with air temp. reaching 28—30°, this period was sometimes reduced to 28—34 hr. Many of the bacterial forms, like *B. acidificans presamigenes casei*, developing were capable of acido-proteolytic action.

T. H. POPE.

**Nitrogen distribution in Kingston cheese-ripening.** B. A. EAGLES and W. SADLER (Nature, 1931, 127, 705—706).—The N distribution indicates that after the first few hr. of ripening, the proteolytic breakdown in the ripening of Kingston cheese is of a peptic-tryptic-like nature.

L. S. THEOBALD.

**Ammonium hydrogen carbonate given with acid slices of sugar beet [from the diffusion process of sugar manufacture] as a substitute for protein in fodder for milk goats.** F. H. ZIEMER (Biochem. Z., 1931, 232, 352—422).—Provided that the fodder which they receive is otherwise appropriate and sufficient, up to 50% of the digestible protein of such fodder can be replaced by NH<sub>4</sub>HCO<sub>3</sub> without detrimental effect on the health of the animals or on their capacity to yield satisfactory milk.

W. MCCARTNEY.

**Determination of diacetyl in butter.** G. TESTONI and W. CRUSA (Annali Chim. Appl., 1931, 21, 147—150).—The butter (200 g.) is slowly distilled in a current of steam, 10 c.c. of distillate are collected, and these are heated gently with excess of hydroxylamine carbonate, 10 drops of 10% NiSO<sub>4</sub> solution, and aq. NH<sub>3</sub>, a red precipitate of Ni dioximine being formed if diacetyl be present. The precipitate may be either weighed or

converted, without separating it, into the sol. complex containing Ni<sup>++</sup> by heating it with PbO<sub>2</sub> and a few drops of alcoholic KOH. The ppt. is then filtered off and the clear red filtrate made up to a definite vol. and compared in a colorimeter with a similar solution obtained from a known wt. of freshly prepared Ni dioximine. Diacetyl was not detected in fresh milk, edible cacao butter, or molten or pasteurised butters, but about 0.0005% was found in various samples of fresh butter (cf. A., 1928, 924; 1929, 1099).

T. H. POPE.

**Digestibility and fodder value of tapioca and fish meal.** H. EDIN, G. KIHLEN, and A. GUSTAFSSON (Kungl. Landtbruks-Akad. Handl. Tidskr., 1930, 69, 882—904; Chem. Zentr., 1931, i, 1378—1379).—Digestion coefficients of tapioca constituents are: crude protein 26, crude fat 80, crude fibre 47, N-free extract 99, carbohydrate 98. Coarsely-powdered material is less digestible (2%) than finely powdered.

A. A. ELDRIDGE.

**Adulteration of fish meal with bone meal.** CLAUSSEN (Z. Fleisch- Milch-hyg., 1931, 41, 137—139; Chem. Zentr., 1931, i, 1379).—On addition of 10% of bone meal to fish meal the number of bone particles in 0.01 g. of the finest powder rose from 4—36 to 98.5.

A. A. ELDRIDGE.

**Cadmium in contact with foodstuffs.** O. C. FORMENTI (Boll. Chim. Farm., 1931, 70, 313—315).—Cd produces chronic poisoning and sometimes death when ingested in small amounts as salts. As regards the possibility of using Cd as a coating for food containers and utensils, the metal is appreciably dissolved in a few hr. by 1% AcOH, tartaric, citric, or lactic acid solution, and by preserved peaches or tomatoes.

T. H. POPE.

**Malt preparations.**—See XVIII.

See also A., June, 757, **Determination of Ca, Mg, and acid-sol. P of milk.** **Residual N of cows' milk.** 774, **Ripening of Japanese persimmon.** 776, **Ether extractives of white leaves of cabbage.** **Carotene of lettuce.** **Lipoids from spinach and cabbage.** **Active substance from raw coffee.**

#### PATENTS.

**Production of bakery products.** POMOSIN-WERKE GES.M.B.H. (B.P. 347,512, 27.5.30. Ger., 1.11.29).—Liquid or powdered pectin (Pomosin) added to the mix for cakes and other bakery products assists in keeping the crumb moist and delaying staling.

E. B. HUGHES.

**Baking of bread or the like and baking powders therefor.** C. F. H. SCHOTT (B.P. 348,509, 2.12.29).—K salts, particularly KHCO<sub>3</sub>, may be used in baking powder if a moisture-absorbing material such as kieselsguhr or lycopodium be added.

E. B. HUGHES.

**Colouring material for oleomargarine, butter, and the like.** J. SCHAU, Assr. to BEST FOODS, INC. (U.S.P. 1,783,154, 25.11.30. Appl., 6.7.25).—Starch grains are coated with Yellow-OB or -AB by mixing with a solution of the colour in a volatile solvent (CO<sub>2</sub>Me<sub>2</sub>). The solvent is evaporated and recovered.

E. B. HUGHES.

**Egg products.** A. K. EPSTEIN (B.P. 346,794, 12.11.29).—The viscosity of liquid egg materials is



modified by the addition of a hydrophillic colloid, *e.g.*, 0.2% of gum tragacanth for whole egg or 1% for yolk. The colloid is dispersed first in a non-aq. medium such as glycerin or edible oil. The product may then be frozen without impairing the viscosity on subsequent thawing. The gum may be used in conjunction with NaCl or sugars as preservatives. E. B. HUGHES.

**Manufacture of cheese.** A. AXELROD (B.P. 347,244, 24.12.29).—Milk is fermented with lactic acid-producing and peptonising bacteria, partly condensed under reduced pressure, and treated with acid and rennet as for normal cheese. E. B. HUGHES.

**Treatment of coffee beans.** K. LENDRICH (B.P. 346,784, 17.1.30).—The excess of chlorogenic acid, which gives rise to the astringent flavour in coffee, may be removed by decomposition into caffeic and quinic acids by treatment of the raw beans with steam under pressure, with or without addition of mineral acid or alkali. E. B. HUGHES.

**Chocolate material.** E. B. WORKING (U.S.P. 1,781,672, 11.11.30. Appl., 16.7.30).—It is claimed that the addition to chocolate of up to 1% of lecithin (usually 0.2%) retards or prevents "greying" or bloom, gives better gloss, increases the temp. range for coating, and renders the material less affected by moisture. The fat content of the chocolate can be reduced (*e.g.*, from 35% to 30%) without decreasing fluidity, and the fat is rendered more digestible. E. B. HUGHES.

**Production of chocolate and chocolate goods.** H. BOLLMANN and B. REWALD (B.P. 347,088, 17.10.30. Ger., 14.5.30. Addn. to B.P. 330,450; B., 1930, 792).—By incorporating 0.25–1% of lecithin in chocolate, 10–25% less cacao butter is required and the tendency for the chocolate to deteriorate is diminished. E. B. HUGHES.

**Manufacture of chewing-gum material.** W. W. TRIGGS. From SWEETS LABORATORIES, INC. (B.P. 347,376, 25.2.30).—Chewing-gum base is prepared by drying a mixture of rubber latex, powdered coumarone resin, and hard hydrogenated oil above 100° in a mixer. Flavouring, sugar, and filling materials may be added subsequently. E. B. HUGHES.

**Manufacture of dried food products from raw meat.** INTERNAT. PRODUCTS PROPRIETARY, LTD. (B.P. 346,200, 12.5.30. Austral., 11.5.29).—Raw meat is trimmed of fat, minced, immersed in H<sub>2</sub>O (15–26°), and stirred. The blood matters separate and are strained from the meat tissue, which is pressed dry, again placed in H<sub>2</sub>O, stirred into a spongy mass, and heated to 55–65°, when the water substance soon comes away freely. The meat protein is pressed to a dry cake, granulated or powdered, and "conditioned" by exposure for a few hr. to the atm. The product is practically flavourless, but the liquid expressed at 55–65°, containing the meat flavours, may be concentrated, powdered, and diffused over the meat proteins. E. B. HUGHES.

**Preservation of foodstuffs [meat].** G. M. CLARK. From J. F. STELLA and L. TESSIER (B.P. 346,520, 21.2.30).—Meat is preserved (*e.g.*, in the preparation of canned beef) by passing it, by means of a screw conveyor, first through water at 100° for 25 min. and then through

pickling solution at 77° for 1 hr. Apparatus is described. E. B. HUGHES.

**Preservation of [edible] emulsions.** A. MUSER (U.S.P. 1,782,011, 18.11.30. Appl., 26.10.28).—Paraffin wax is used to fill the air space usually found in bottles containing mayonnaise and salad cream. E. B. HUGHES.

**[Capsules for] flavouring foods.** P. LINDNER (B.P. 348,698, 17.2.30. Ger., 20.2.29).

**Margarine.**—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Chemical examination of *p*-hydroxyphenyl-methylaminoethanol hydrochloride.** S. M. GORDON (J. Amer. Pharm. Assoc., 1931, 20, 437–443).—Chemical and physical characteristics of the compound are described and compared with those of related substances. Standards are suggested. E. H. SHARPLES.

**Colour reactions of some medicaments.** J. SIVADJIAN (J. Pharm. Chim., 1931, [viii], 13, 528–529).—Plasmoquin forms a blue colour when its epichlorohydrin solution is boiled with chloranil; percaïne, under similar conditions, forms an emerald-green colour. Na 5-iodo-2-hydroxypyridine acetate ("Uroselectan") in aq. solution (1 c.c.) gives a blue colour when heated with 6–7 drops of Br water; addition of conc. H<sub>2</sub>SO<sub>4</sub> causes its disappearance and the formation of a ppt., but the colour reappears on adding NH<sub>3</sub> solution. An ammoniacal solution of pyrocatechol forms a cherry-red colour when boiled with a little nitroprusside. E. H. SHARPLES.

**Absorptive power of medicinal carbon.** G. FRERICHs (Apoth.-Ztg., 1930, 45, 913–914; Chem. Zentr., 1930, ii, 2416).—The accepted method of determination is criticised and a modification proposed. L. S. THEOBALD.

**Application of the analytical quartz lamp to the examination of perfumes.** E. EKMAN and A. SAMY-SCHLAXEVA (Riechstoffind., 1930, 5, 164–165, 182–184; Chem. Zentr., 1931, i, 1375).—Data for alcohols, aldehydes, ketones, phenols, acids, and esters are tabulated. When pure, few alcohols fluoresce. A. A. ELDRIDGE.

**Sterilisation of cocaine solutions.** W. HÖK (Dansktidsskr. Farm., 1931, 5, 37–42).—During the technical sterilisation of local anæsthetic solutions containing cocaine, H<sub>2</sub>O, EtOH, and phenylurethane are lost and the residual solution becomes richer in cocaine and NaCl. Experiments with a 1% aq. solution of pure cocaine hydrochloride showed that no appreciable decomp. of the alkaloid took place during 15 min. sterilisation at 125°, but by more prolonged heating at this temp. or by sterilisation at higher temp., partial decomp. resulted with production of BzOH; in one experiment 30% of the cocaine was decomp. during sterilisation for 30 min. at 150°. H. F. HARWOOD.

**Ash structures of important drug-containing leaves. Recognition of drugs by means of their ash structures.** II. K. OHARA and Y. KONDO (Arch. Pharm., 1931, 269, 292–306; cf. J. Pharm. Soc. Japan, 1929, 49, 573).—The distribution and shape of the CaC<sub>2</sub>O<sub>4</sub> crystals in the ash of leaves prepared in the Werner apparatus (B., 1929, 260) may be used to

distinguish certain drugs. Detailed descriptions of the ash structure of a number of common drugs are given.

A. A. LEVI.

I. "Etrates," a new highly-active type of drug.  
II. Determination of the extractive content of drugs. C. GRIMME (Pharm. Zentr., 1930, 71, 657—659; 1931, 72, 193—197).—The "Etrate," obtained by evaporating to dryness a concentrated extract of a drug with a proportion of the drug itself, presents advantages for oral administration over other preparations. The compositions of Etrates from cinchona bark, bearberry leaves, valerian root, rhubarb root, senna leaves, and ergot of rye are compared with those of the corresponding fluid extracts and tinctures.

II. Previously employed procedures are reviewed and the amounts of extractive obtained by 12 methods from the first 4 drugs mentioned above and the 4 corresponding Etrates are tabulated. Exhaustive extraction with boiling  $H_2O$  gives the highest results. The results with EtOH vary considerably with its  $H_2O$  content and are, therefore, of little value, except in special cases.

H. E. F. NOTTON.

**Hyoscyamus extract, Ital. P. V.** A. SCOCCIANI (Boll. Chim. farm., 1931, 70, 355—358).—The alkaloid content of 0.5% cannot be attained by the method prescribed in the Italian pharmacopœia V. From survey of foreign pharmacopœias and from experimental data it appears that the max. limit for the alkaloid content of hyoscyamus extract should be 0.3%. This standard should be adopted by the International Commission for Dangerous Drugs.

O. F. LUBATTI.

**Mitchella repens, L., N.F.V.** W. P. BRIGGS (J. Amer. Pharm. Assoc., 1931, 20, 224—226).—The EtOH extract of the drug contains a resin, but no alkaloid, glucoside, or saponin. The drug contains moisture (10.2%), ash (6.2%), insol. ash (2.2%), reducing sugars, tannins, starches, waxes, fats, and protein (4.9%). Aq. or alcoholic extracts have no marked pharmacological activity. H. E. F. NOTTON.

**Hydrastis canadensis (Golden seal).** I. E. GILLIS and H. A. LANGENHAN (J. Amer. Pharm. Assoc., 1931, 20, 210—224).—The % of Et<sub>2</sub>O-sol. alkaloid in the root usually runs parallel with, and about one unit lower than, that in the rhizome (4.3—2.5%; mean, 3.5%). The alkaloid content of the rhizome remains fairly constant (4.2%) from November until June, rising slightly in May and declining to a minimum (3.3%) in September; that of the root shows similar but less regular variations. The alkaloid contents on a moisture- and soil-free basis are also tabulated. H. E. F. NOTTON.

**Tests of Oleum ricini, D.A.B. VI.** W. MEYER (Chem.-Ztg., 1931, 55, 279).—Results of tests of samples of pharmaceutical castor oil are described and compared with the Biechele-Brieger specifications for preparations described in D.A.B. VI. No cryst. material separated, as required by the specification, nor did the oil become pasty, even at  $-16^{\circ}$ . A small quantity of free acid was present, and it is recommended that a test for acidity be applied, especially if the oil is to be used for the manufacture of explosives.

H. F. GILLBE.

**Comparison of the chemical and biological assays of Oleum chenopodii.** W. F. REINDOLLAR and J. C.

MUNCH (J. Amer. Pharm. Assoc., 1931, 20, 443—445).—The U.S.P. assay confirms the greater ascaridol content (87—100%) and the higher sp. gr. (0.9883—0.9993) reported for "high-test" oil of chenopodium, as compared with those of "normal" oil (50.0—75.6% and 0.9549—0.9765). No biological assay which bore any relationship to the ascaridole content could be established.

E. H. SHARPLES.

**Determination of oil and alkaloids in delphinium seed.** L. N. MARKWOOD (J. Amer. Pharm. Assoc., 1931, 20, 454—455; cf. A., 1927, 1227).—Alkaloids are separated from the light petroleum extract of the ground seed with 5%  $H_2SO_4$  and the acid extract is made alkaline with 15% NaOH and extracted with  $CHCl_3$ . This last extract is treated again with 5%  $H_2SO_4$  and the acid extract treated as above. The procedure is repeated and the final  $CHCl_3$  extract is washed and evaporated to give the total alkaloids. The oil content is obtained by evaporation of the light petroleum extract after separation of the alkaloids. *D. consolida* gave 28.3—28.7% of oil and 1.01—1.06% of alkaloids and *D. staphisagria* 34.7—35.4% and 1.22—1.35%, respectively.

E. H. SHARPLES.

**Essential oil from the wood of Cedrela Toona, Roxb.** P. P. PILLAI and B. S. RAO (J.S.C.I., 1931, 50, 220—221 r).—The essential oil from the wood of the tree *Cedrela Toona*, Roxb., yield 0.44%,  $d_{20}^{25}$  0.9287,  $n_D^{25}$  1.5007,  $[\alpha]_D^{25}$   $+2.5^{\circ}$ , acid val. 1.1, ester val. 2.1, acetyl val. 13.3, has been shown to have the approximate composition *l*-copaene 35%, *l*-cadinene and other dicyclic sesquiterpenes 45%, *l*-cadinol 13.0%. The semicarbazone of the ketonic acid,  $C_{15}H_{24}O_3$ , from the oxidation of *l*-copaene has m.p. 231—232° and that of its Me ester 199—200° (cf. Semmler and Stenzel, A., 1915, i, 427).

**Camphor and camphor oil from Cinnamomum camphora grown in New Zealand.** I. R. SHERWOOD and W. F. SHORT (J.S.C.I., 1931, 50, 195—198 r).—The leaves of 8-year-old camphor trees yielded 1.9% of camphor, *d*- $\alpha$ -pinene, camphene, dipentene, caryophyllene (more than 20%), and cadinene (probably). Wood-oil from 40-year-old trees (lower and middle stem) contained camphor,  $\alpha$ -pinene, camphene, dipentene, cineole, terpineol, safrol (10%), eugenol, cadinene (probably), and a substance m.p. 162°. The sesquiterpene alcohol fraction contained neither primary nor secondary alcohols. The phenylhydrazone and semicarbazone methods of determining camphor are discussed.

**Hydrocarbon constituents of Japanese peppermint oil.** K. J. DUNCAN and W. F. SHORT (J.S.C.I., 1931, 50, 198 r).—Besides *l*-limonene (Schimmel Ber., Apr., 1912, 103), Japanese peppermint oil contains  $\alpha$ -pinene, caryophyllene, and a small quantity of another sesquiterpene (dextrorotatory).

**Hungarian essential oils.** M. JANICSEK (Mezőgazdasági Kutatások, 1929, 2, 153—157).—Oils from experimental plots gave high characteristic numbers: peppermint oils, menthol content 69.7—71.4%; coriander oil, alcohol content (coriandrol) 72.6%; *Salvia sclarea*, ester content 70%; *Lavandula vera*, ester content 62.5%. Oils of marjoram, chenopodium, and *Salvia officinalis* were also examined.

H. NICOL.

**Ethereal oils from *Bæckea frutescens*, L., and from *Dalbergia parviflora*, Roxb.** D. B. SPOELSTRA (Rec. trav. chim., 1931, 50, 433–440).—Steam-distillation of the leaves of *B. frutescens*, L., at 120° gives 0.9–1.8% of an oil from which by fractional distillation are isolated: *l*- $\alpha$ - and - $\beta$ -pinene (58%); *l*-limonene and dipentene (3.1%); *p*-cymene (0.7%); cineole (7.4%); *l*-linalool, fenchyl alcohol, *l*-borneol, and *l*- $\alpha$ -terpineol (approx. 10%); a dicyclic sesquiterpene, b.p. 126–135°/12 mm. (1.4%), dehydrogenated to cadalene; a dicyclic sesquiterpene alcohol, b.p. 152–157°/13 mm.,  $[\alpha]_D^{20}$  –12° (3%), also giving cadalene on dehydrogenation; phenolic components (2.4%); and a phenolic ether, m.p. 104°, probably identical with that isolated by Penfold (B., 1924, 576). Fractionation of the ethereal oil (0.45–0.8% yield) obtained by steam-distillation of the wood of *D. parviflora*, Roxb., affords *l*-nerolidol, which is the main constituent, whilst traces of furfuraldehyde and farnesol are also probably present.

J. W. BAKER.

**Essential oil of *Taiwania Cedar*.** K. KAFUKU and R. KATO (Bull. Chem. Soc. Japan, 1931, 6, 65–74).—The wood from *Taiwania cryptomerioides*, Hayata, gave 0.23% of an oil having  $d_4^{25}$  0.9593,  $n_D^{25}$  1.5104,  $\alpha_D^{25}$  –2.00°, acid value 0.38, ester value 10.52, ester value after acetylation 83.00. Caryophyllene, humulene, and cadinene were identified, and there was also obtained a sesquiterpene alcohol “taiwanol,” b.p. 141–143°/6 mm.,  $d_4^{20}$  0.9692,  $n_D^{20}$  1.5045,  $\alpha_D^{20}$  –42.56° (urethane, m.p. 134–135°). With HCl the substance gave cadinene dihydrochloride, whilst oxidation gave a substance, m.p. 170°, and dehydration with formic acid gave cadinene. The alcohol is a tertiary alcohol either identical or isomeric with cadinol (A., 1914, i, 63). Cadinene oxidised with  $\text{KMnO}_4$  in aq.  $\text{COMe}_2$  gives a substance,  $\text{C}_{12}\text{H}_{22}\text{O}_2$ , m.p. 142–143°.

A. A. LEVI.

**Essential oil of leaves of *Chamaecyparis obtusa*, Sieb. et zucc., f. *Formosana*, Hayata, or Arisan “Hinoki.”** II. K. KAFUKU and T. NOZOE (Bull. Chem. Soc. Japan, 1931, 6, 111–118; cf. B., 1931, 565).—The high-boiling portions of this oil contain a new sesquiterpene, *sesquichamene*, b.p. 122.5–123.5°/12 mm.,  $n_D^{25}$  1.5021,  $d_4^{25}$  0.9277,  $\alpha_D^{25}$  –89.85°, [nitroschloride, m.p. 77.5–78.5° (decomp.); nitrobenzylamine, m.p. 165–166° (decomp.)], oxidation of which with  $\text{KMnO}_4$  in  $\text{COMe}_2$  gives a substance (semicarbazone, m.p. 233°), a substance,  $\text{C}_{15}\text{H}_{26}\text{O}_2$ , m.p. 89–91°, and a substance, m.p. 240–245° (decomp., sinters 220°). Treatment of sesquichamene with alcoholic  $\text{H}_2\text{SO}_4$  gives isosquichamene, b.p. 129–131°/12 mm.,  $n_D^{21.5}$  1.5109,  $d_4^{21.5}$  0.9320,  $\alpha_D^{21.5}$  –8.52°. Other constituents are cadinene and cadinol.

A. A. LEVI.

**Volatile oil of *Pycnanthemum miticans*.** H. W. WERNER (J. Amer. Pharm. Assoc., 1931, 20, 445–448).—Steam-distillation of the dried plant gave 1.57% of oil having:  $d_4^{20}$  0.9250,  $\alpha_D^{20}$  +16.49°,  $n_D^{20}$  1.4807, ester val. 12.64 (equiv. to 4.47% of  $\text{C}_{10}\text{H}_{19}\text{OH}$  as acetate), ester val. after acetylation 61.29 (equiv. to 17.90% of  $\text{C}_{10}\text{H}_{19}\text{OH}$  in original oil), solubility in 70% EtOH 1 in 1.82, ketones ( $\text{Na}_2\text{SO}_3$  method) 71.75%. Menthone, pulegone (60–75.75%), menthol or *d*-neomenthol, and another ketone ( $\beta$ -thujone?) were present, together

with AcOH and formic acid, terpenes, and sesquiterpenes.

E. H. SHARPLES.

**Determination of hexamethylenetetramine.**—See III. Solution of Pb subacetate (U.S.P.) Alkali bitartrates. Sodium bismuth citrate.—See VII. Ethereal oils as germicides.—See XXIII.

See also A., June, 684, Dibismuthyl monosodium citrate solutions. 698, Capillary analysis. 725, *N*-Phenylalkyl-*N*-methylamino-alkyl benzoates and *p*-aminobenzoates. 731, Anaesthetics of the diphenyl ether series. 742, Antimalarials. 750, Colour reactions of brucine. Fluorescence of alkaloids. 752, Determination of narcotine, santonin, etc. Titration of alkaloid salts. 753, Determination of iodobismuthate. 763, Toxic action etc. of ethylene glycol. Styryl- and anil-benzthiazole derivatives as antiseptics etc. 764, Toxicity of *Corydalis caseana*. 769, Bacterial lipins. 771, Extraction of insulin. 775, Rhusinic acid. 777, Constituents of “Salpamisri.” 778, Microchemical detection of colchicine, piperine, etc., and of choline in plants.

## PATENTS.

**Arylamino-alcohol [therapeutic] compound.** R. ADAMS, Assr. to ABBOTT LABORATORIES (U.S.P. 1,767,423, 24.6.30. Appl., 27.1.28).—Compounds of the type,  $\text{Ph}\cdot\text{CH}(\text{OH})\cdot\text{CHR}\cdot\text{NR}'\text{R}''$ , where R and R' are alkyl and R'' is H or alkyl above  $\text{C}_1$ , have a pressor and sympathomimetic effect. They are produced by reduction of the corresponding ketones. In the examples, R = Me, R'' = H, and R' = Et (ketone, m.p. 183–184°; alcohol, m.p. 190–191°), Pr<sup>a</sup> (m.p. 167–168°, and 205–206°), Pr<sup>b</sup> (m.p. 213–213.5°, and 193°), Bu (m.p. 158–159°, and 220–221°), or *n*-amyl (m.p. 150°, and 219°); or R = Me, R' = R'' = Et (m.p. 167–168°, and 205–206°); or R' = Me, R'' = H, and R = Et (m.p. 190–192°, and 197–199°) or Pr<sup>a</sup> (m.p. 182–184°, and 224–225°).

C. HOLLINS.

**Preparation of stable solutions of acetylcholine salts.** G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 345,713, 13.1.30).—Solutions of acetylcholine salts (hydrohalides) in incompletely acylated polyhydric alcohols, e.g., in glycerol mono- or di-acetate or  $\beta$ -hydroxyethyl acetate, are stable cold or hot and are suitable for injections.

C. HOLLINS.

**Manufacture of double compounds of the acridone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,631, 24.12.29. Addn. to B.P. 328,212; B., 1930, 741).—Technical mixtures produced in the alkylation of 3:7-diaminoacridine are used, after analysis, for the preparation of the double compounds of the prior patent.

C. HOLLINS.

**Mercury compound of *m*-cresol.** D. MACD. COPLEY and J. P. SNYDER, Assrs. to NORWICH PHARMACAL Co. (U.S.P. 1,782,090, 18.11.30. Appl., 14.9.27).—*m*-Cresol, dissolved in alcoholic KOH, is mixed with alcoholic  $\text{HgCl}_2$  at 15° and heated at 100° until pasty. The product is filtered off, washed, and dried at 100°.

C. HOLLINS.

**Preparation of cineole-containing oil mixtures.** RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 345,517, 5.4.30. Ger., 15.4.29).—In the production of cineoles

from terpineol, terpinenol, terpin, terpin hydrate, etc. by isomerisation and/or dehydration, 1:8-cineole is the main product below 80°, *e.g.*, with 30–40%  $\text{H}_2\text{SO}_4$  at 35° or 60% acid at –10°. The product must be removed as formed, *e.g.*, by distillation in vac., or by extraction with  $\text{PhMe}$  or  $\text{CHCl}_3$ . Hydration products from pinene or sabinene are suitable starting materials.

C. HOLLINS.

**Basic products. Albumose-like products.**—See III. **Sol. cellulose esters.**—See V.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., June, 722, **Light-sensitive diazo compounds.** 752, **Determination of diazo-N in light-sensitive compounds.**

### PATENTS.

**Light-sensitive layer and manufacture of photographic images.** J. EGGERT, R. SCHMIDT, and B. WENDT, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,782,259, 18.11.30. Appl., 12.7.29. Ger., 17.7.28).—The conversion of *cis*- into *trans*-isomerides by light is utilised, the print being fixed by means of a selective solvent etc. *trans*-Dibenzoyl ethylene on black paper gives a positive from a positive on washing with  $\text{EtOH}$ . Maleic acid on white paper with a trace of Br as catalyst gives a positive from a negative on conversion of the fumaric acid into insol. Ag or Hg salt, followed by washing and development with chromate. Phenylcitraconic acid on collodion with phenacyl bromide as catalyst is washed with  $\text{CHCl}_3$ , and developed with a solution of a Hg, Ag, or Tl salt to a white image of the phenylmesaconate, which becomes black in a  $\text{Na}_2\text{S}$  bath. C. HOLLINS.

**Production of colour-record kinematograph films.** TECHNICOLOR MOTION PICTURE CORP., Assces. of L. P. TROLAND (B.P. 349,318, 31.5.30. U.S., 15.6.29).

**Reproduction of colour-record images on lenticular films.** SOC. FRANÇ. DE CINÉMAT. ET DE PHOT. FILMS EN COULEUR KELLER-DORAIN, and J. L. VIDAL (B.P. 349,267, 2.5.30. Fr., 2.5.29).

**Nitrocellulose compositions. Films.**—See V.

## XXII.—EXPLOSIVES; MATCHES.

**Nitrocellulose.**—See V. **Oleum ricini.**—See XX.

See also A., June, 689, **Transmission of detonation.**

### PATENTS.

**Manufacture of nitrostarch.** O. A. PICKETT, Assr. to HERCULES POWDER CO. (U.S.P. 1,779,825, 28.10.30. Appl., 4.5.26).—The nitration acids contain 12–15%  $\text{H}_2\text{O}$ , so as to promote swelling of the starch during nitration. The dried starch is sieved into the acid and forced beneath the surface by stirrers, the temp. of the acid being maintained at 20–30° by means of a cooling jacket. After nitration, the mixture of acid and nitrostarch is run into  $\text{H}_2\text{O}$ , the acid wash-water is drawn off, and the nitrostarch stabilised, its viscosity being reduced, if desired, by boiling with steam. The product is readily sol. in the usual solvents employed in lacquer manufacture. W. J. WRIGHT.

**Priming mixtures.** J. D. McNUTT, Assr. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,774,992,

1,779,820—1, and 1,779,851, [A] 2.9.30, [B–D] 28.10.30. Appl., [A] 21.11.27, [B] 24.12.27, [C] 21.6.28, [D] 31.3.27). —(A) A mixture of 20–40% of  $\text{PbO}_2$ , 5–10% of  $\text{BaCrO}_4$ , 25–40% of Hg fulminate, and 3–15% of tetranitromethylaniline, together with 15–20% of ground glass, is claimed. (B) The aniline derivative in (A) may be replaced by pentaerythritol tetranitrate, and this latter, wholly or partly, by trinitroresorcinol; also  $\text{Ba}(\text{NO}_3)_2$  is substituted for  $\text{BaCrO}_4$ . (C) A mixture of 45% of  $\text{Ba}(\text{NO}_3)_2$ , 35% of Hg fulminate, 16% of  $\text{Pb}(\text{CNS})_2$ , 2% of  $\text{BaCO}_3$ , and 2% of charcoal is claimed. (D) The composition may consist of 475 pts. of Hg fulminate (16%  $\text{H}_2\text{O}$ ), 240 pts. of  $\text{Ba}(\text{NO}_3)_2$ , 255 pts. of Sb sulphide, 30 pts. of trinitrotoluene, and  $\frac{1}{2}$ –2 pts. of a stabiliser, *e.g.*, diphenylamine. W. J. WRIGHT.

**Explosive priming mixtures.** L. MELLERSH-JACKSON. FROM WINCHESTER REPEATING ARMS CO. (B.P. 346,092, 11.2.30).—The use of witherite, instead of artificial  $\text{BaCO}_3$ , as a constituent of priming mixtures containing Sb sulphide, eliminates the use of ground glass. A suitable mixture consists of Hg fulminate 35–50, basic Pb nitrate 23–33,  $\text{Pb}(\text{CNS})_2$  5–15, Sb sulphide 5–20, and witherite 5–10%. W. J. WRIGHT.

**Gelignite and gelatin dynamite.** R. L. HILL, Assr. to ATLAS POWDER CO. (U.S.P. 1,778,718, 21.10.30. Appl., 14.12.28).—Part or all of the  $\text{NaNO}_3$  or  $\text{KNO}_3$  of the explosive is added in the form of pellets, to give the explosive improved plasticity and higher sensitiveness to detonation. W. J. WRIGHT.

**Manufacture of propellent explosives.** IMPERIAL CHEM. INDUSTRIES, LTD., R. BURNS, and O. W. STICKLAND (B.P. 346,295, 6.1.30).—In order to prevent metallic fouling in firearms, an organo-metallic compound, such as a substituted stannane, having approx. the same sp. gr. as have the other constituents of the explosive, is incorporated with the explosive. Suitable compounds for powders of the (a) solventless, (b) nitrocellulose, and (c) nitroglycerin type are the tetraphenyl, tetra-*n*-butyl, and propyl tri-*n*-butyl compounds of Sn, respectively, the last two compounds being dissolved in the volatile solvent employed. W. J. WRIGHT.

**Smokeless powder.** F. I. DU PONT, Assr. to U.S.F. POWDER CO. (U.S.P. 1,780,911, 11.11.30. Appl., 29.10.26).—In addition to the usual solvent employed in the production of propellent powders there is added about 1–5% of a substance, such as glycerin, which contains C and H, is liquid at room temp. and non-volatile at the drying temp. of the powder, forms a stable mixture with  $\text{H}_2\text{O}$ , and is a non-solvent for nitrocellulose. Undue loss of residual solvent during drying and the consequent development of dangerous pressures in practice are thus avoided. W. J. WRIGHT.

**Absorbent material for explosives.** H. C. BUGBIRD (U.S.P. 1,779,530, 28.10.30. Appl., 1.11.24. Renewed 17.3.27).—Carbonised lignin residue, after leaching to remove alkaline and other water-sol. material and heating to redness in absence of air, is used as an absorbent for liquid  $\text{O}_2$ . W. J. WRIGHT.

**Preparation of absorbents for liquid-oxygen explosives.** H. C. BUGBIRD (U.S.P. 1,781,214, 11.11.30. Appl., 28.9.25).—Lignin residues from the manufacture of wood pulp are carbonised, leached, and ground to a

relatively coarse granular product, the voids being then filled with a finely-divided absorbent material, *e.g.*, carbon black, having an absorptive capacity for liquid  $O_2$  equal to at least 2.6 times its wt. W. J. WRIGHT.

Nitrocellulose.—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Organic constituents of fresh and ripe sewage sludge.** H. HEUKELEKIAN (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 31–36).—Fresh solids contain mainly fats and crude protein with smaller proportions of  $H_2O$ -soluble and  $Et_2O$ -sol. matter, cellulose, hemicellulose, and lignin. During ripening, the principal loss of solids falls on fatty matter and cellulose.

A. G. POLLARD.

**Distribution of iodine in sewage and use of sewage sludge as iodine manure.** R. KÖHLER (Mitt. Lab. Preuss. Geol. Landesanst., 1930, No. 11, 1–14; Chem. Zentr., 1931, i, 1332).—Town sewage is relatively rich in I. Removal of the org. matter in the course of sewage purification reduces the I content of the effluent. The I content of crops may be increased by fertilisation with the sludge.

A. A. ELDRIDGE.

**Effect of certain chemicals on the vacuum filtration and gravity drying of ripe [sewage] sludge.** W. H. BAUMGARTNER (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 17–25).—Addition of  $FeCl_3$  to ripe sewage sludge increases the rate of filtration and of the drainage from drying beds. The resulting sludge has a more open texture. Optimum effects corresponded with definite  $p_H$  values, peak values being obtained by the addition of 2 lb. and of 7 lb. of  $FeCl_3$  per cu. yd. of sludge.  $FeCl_3$  adjusts the reaction of the sludge to produce more complete flocculation of colloids and also liberates  $CO_2$ , which renders solids more porous and assists their flotation.

A. G. POLLARD.

**[Sewage-]sludge drying; relation between drainage and evaporation.** W. RUDOLFS and I. O. LACY (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 26–29).—At temp. up to  $55^\circ$  the rate of drainage of  $H_2O$  from sewage sludge is a more important factor in drying than is the rate of evaporation.

A. G. POLLARD.

**Drying of fresh sewage solids.** A. J. FISCHER (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 29–31).—The drying of fresh solids in the open after treatment with  $CaO$ , alum, or a mixture of these is unsuccessful on account of poor drainage and objectionable odours.

A. G. POLLARD.

**Effectiveness of sodium fluosilicate as compared with borax in controlling the house fly.** S. MARCOVITCH and M. V. ANTHONY (J. Econ. Entom., 1931, 24, 490–497).— $Na_2SiF_6$  proved superior to borax for the treatment of manure heaps for house-fly control, and the risk of subsequent soil injury was much reduced.

A. G. POLLARD.

**Disinfectant action of trichloroethylene.** W. RIMPAU (Z. Hyg., 1931, 112, 202–221).—Stable emulsions of  $C_2HCl_3$ ,  $CHCl_3$ , and  $C_2H_2Cl_4$  have strong disinfectant properties. Solutions of  $PhOH$  derivatives, salicylic acid, etc. in  $C_2HCl_3$  are active disinfectants only when emulsified.

A. G. POLLARD.

**Germicidal action of ethereal oils.** S. L. MALOWAN (Z. Hyg., 1931, 112, 93–94).—The bactericidal action of ethereal oils is closely related to their solubility in  $H_2O$ . Oils consisting principally of esters are less bactericidal than those mainly composed of alcohols.

A. G. POLLARD.

**Ferric iron coagulation [for river water].** A. POTTER and W. I. KLEIN (J. Amer. Water Works' Assoc., 1931, 23, 719–727).— $Fe^{+++}$  has proved a more satisfactory coagulant than  $Al_2(SO_4)_3$  in the 5 million gal./day plant at Mamaroneck, N.Y., which treats river water from a small watershed without impounding reservoirs and so is subject to rapid variations in colour, taste, etc. The advantage is largely due to the better settling qualities of the  $Fe(OH)_3$  floc and the consequent effect thereof on the condition of the sand filters.

C. JEPSON.

**Unusual colour-removal plant [for well-water supplies].** C. WILSON (J. Amer. Water Works' Assoc., 1931, 23, 689–695).—An experimental plant to treat 69,000 gal. per day has demonstrated that at Los Angeles a peaty well-water may be freed from colour at low cost by means of  $FeCl_3$ . The plant permits a continuous return of the  $Fe(OH)_3$  sludge to the reaction chamber, with mechanical agitation to remove gases and so facilitate settlement. The sand filters are of coarse material, 10 in. deep, and have 10 in. freeboard for backwashing. The influent is applied by means of a grid laid on the sand surface with holes so placed as to deliver the  $H_2O$  horizontally.

C. JEPSON.

**Photoelectric control of chlorine feed [in water-works' practice].** J. H. HARRINGTON (J. Amer. Water Works' Assoc., 1931, 23, 736–739).—The dosage of  $Cl$  is automatically varied by means of apparatus controlled by differences in the intensity of the light falling on to two light-sensitive cells after passing through two colour cells. Each colour cell contains a similar sample of the water and is placed opposite and equidistant from a common source of light, but while in one cell a colour is developed by a suitable reagent proportional to the amount of residual  $Cl$ , the other has coloured glasses interposed to represent the colour which would be produced by the residual desired. Any variation from this standard is communicated by the photoelectric cells to the apparatus controlling the rate of feed and is thus corrected.

C. JEPSON.

**Fluorescence of water in filtered ultra-violet light as an indicator of its purity.** H. IVEKOVIC (Z. Hyg., 1931, 112, 54–61).—The bluish-white fluorescence frequently shown in filtered ultra-violet light by  $H_2O$  samples is due to dissolved organic matter. The intensity of the fluorescence increases with the  $O$  consumption value of the sample. Comparative tests of purity may be obtained by means of the intensity of fluorescence of gelatin solutions made with the samples.

A. G. POLLARD.

#### PATENTS.

**Water-softening plants.** T. W. ELEY (B.P. 348,749, 1.3.30).

**Removing solid particles from liquids.**—See I. **Distillation of material.**—See II. **Sol. cellulose esters.**—See V.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 31 and AUG. 7, 1931.\*



### I.—GENERAL; PLANT; MACHINERY.

**Reactions of sulphur compounds in boiler furnaces.** H. F. JOHNSTONE (Ind. Eng. Chem., 1931, 23, 620—625).—Air containing 0.428%  $\text{SO}_2$  was passed over powdered flue dust on asbestos at various temp. The max. oxidation of  $\text{SO}_2$  was 1.8% at  $400^\circ$ , the gases being dry. With moist gas and low temp. the oxidation is much greater, being catalysed by dissolved Fe salts. A number of analyses of stack gases, using the absorption method with  $\text{CH}_2\text{Ph}\cdot\text{OH}$  to inhibit secondary oxidation of  $\text{SO}_2$ , showed that only 2—3% of the S was present as  $\text{SO}_3$ , regardless of temp., O content, or the position from which the sample was drawn. The oxidation was rather less with powdered-fuel installations. 70—90% of the S in the fuel enters the gas. In one case, where acid petroleum residues containing much sulphate were used as fuel, the  $\text{SO}_3$  content was only slightly higher. Serious corrosion can therefore occur only when a metal surface is at a temp. lower than the dewpoint of the gas. The dewpoint of the gas is raised by high moisture content and by the presence of deposits containing  $\text{Fe}^{+++}$  salts, and may reach 80— $100^\circ$ . The presence of  $\text{FeS}$  is probably the cause of the formation of hard deposits in economisers. The better oxidation produced by powdered-fuel firing prevents its occurrence. C. IRWIN.

**Prevention of silica scale [in boilers] with sodium aluminate.** C. H. CHRISTMAN, J. A. HOLMES, and H. THOMPSON (Ind. Eng. Chem., 1931, 23, 637—646).— $\text{Na}_2\text{Al}_2\text{O}_4$  used as a coagulant for boiler feed-water reacts with  $\text{SiO}_2$  and Ca or Mg to form complex insol. aluminosilicates. At high pressures the reaction with Mg is much more complete than with Ca.  $\text{SiO}_2$  present in  $\text{H}_2\text{O}$  at concentrations of 5—50 p.p.m. is not precipitated by  $\text{CaSO}_4$  or  $\text{MgSO}_4$  alone, and  $\text{Na}_2\text{Al}_2\text{O}_4$  is more effective in precipitating it than  $\text{NaOH}$ . A lengthy series of tests on %  $\text{SiO}_2$  removed by different proportions of the reagents is described. At 400 lb. 2 pts. of  $\text{SiO}_2$  are completely precipitated by 2 pts. of  $\text{Al}_2\text{O}_3$  and 1 pt. of  $\text{MgO}$ . It is necessary to have excess  $\text{Al}_2\text{O}_3$  remaining in solution, and if the  $\text{MgO}$  is deficient it should be added. The procedure recommended is to use  $\text{Na}_2\text{Al}_2\text{O}_4$  as coagulant in external softening and to add further aluminate to the softened  $\text{H}_2\text{O}$ .  $\text{SiO}_2$  is of great importance in the formation of scale in boilers fed with softened  $\text{H}_2\text{O}$ . C. IRWIN.

**Properties of dispersions of the quicksand type.** R. V. WILLIAMSON and W. W. HECKERT (Ind. Eng. Chem., 1931, 23, 667—670).—Certain dispersions become more viscous when stirred or shaken vigorously. This "inverted plasticity" was measured with a Stormer viscosimeter with a 2-bladed stirrer, using a dispersion

of corn starch in  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ , and other liquids. The critical rate of stirring increases linearly with dilution of the starch. It is suggested that the phenomenon is due to the motion of the paddles causing the particles around them to arrange themselves in concentric shells, and that the critical rate of stirring corresponds to the disruption of these shells. This inverted plasticity occurs only with good dispersions with a slow rate of settling, and is shown by pigments in certain vehicles.

C. IRWIN.

**Heat transfer to liquids in viscous flow.** C. G. KIRKBRIDE and W. L. MCCABE (Ind. Eng. Chem., 1931, 23, 625—631).—The Nusselt-Gröber theory states that for heat transfer into a liquid flowing at less than the isothermal critical velocity  $hD/k = \psi(kL/D^2vc)$ , where  $h$  = film coeff. at a given point,  $D$  = tube diam.,  $k$  = thermal conductivity,  $L$  = tube length,  $v$  = mass velocity, and  $c$  = heat capacity of fluid. It is assumed that a preliminary "calming" zone in the pipe is provided and transverse convection currents are ignored. Experiments with  $\text{H}_2\text{O}$  and two oil samples yielded curves showing the relation of the two sides of the equation, taking the average film coeff., and these are compared with an integrated curve based on Nusselt's theory. With increasing length of tube the quantity  $hD/k$  approaches a limiting value of 3.65, and under these conditions there is good agreement between experiment and theory, but with low values for  $L$  this is not the case. The authors, however, used electrical heating equivalent to const. heat input per unit length, and data based on steam heating or theories assuming const. wall temp. required modifications, which have been made. C. IRWIN.

**A viscosity paradox.** E. N. DA CUNHA ANDRADE (Trans. Faraday Soc., 1931, 27, 201—203).—The rate of flow of a liquid in a cylindrical tube is influenced to an unexpected degree by the presence of even a very fine axial wire; e.g., if the radius of the wire is  $10^{-10}$  of that of the tube the flow is diminished by over 4%.

F. L. USHER.

**Volume relations of gases at high pressures.** J. R. DILLEY (Chem. Met. Eng., 1931, 38, 280—281).—At high pressures the ideal gas law is inaccurate. Curves are given by means of which the vol. relations of  $\text{H}_2$ ,  $\text{N}_2$ ,  $\text{CO}$ , and a 3 : 1 mixture of  $\text{H}_2$  and  $\text{N}_2$  at high pressures may be calc. D. K. MOORE.

**Solid  $\text{CO}_2$ .**—See VII. **Grinding pottery material.**—See VIII. **Dust removal from gases.**—See XI.

### PATENTS.

**Tunnel ovens.** J. DANIELS (B.P. 348,225, 17.2.30).—In a tunnel kiln the gas and air regenerators alternate

\* The remainder of this set of Abstracts will appear in next week's issue.

frequently along the length of the kiln, and the air and gas ports in the heating chamber alternate still more frequently, so that continuous sheets of flame are produced.

B. M. VENABLES.

**Regenerative [recuperative] ovens or kilns.** GAS LIGHT & COKE CO., C. VYSE, and W. DIETERICH (B.P. 348,607, 12.2.30).—A muffle is heated by groups of burners situated below its sides at points diagonally opposite. The gases from each group of burners pass up one side, across the roof, down the other side, across the bottom, and then zig-zag sideways and downwards through a recuperator. Secondary air passes zig-zag front and back upwards through the recuperators. An auxiliary burner is provided for creating a draught on starting the furnace.

B. M. VENABLES.

**Feeding materials to rotary kilns.** A. BUSCH (B.P. 348,799, 27.3.30).—In, *e.g.*, a cement kiln, the slurry is fed in the form of one or more compact jets substantially parallel to the axis of the kiln, and the jets impinge on chain curtains arranged in lines at a slight angle to the axis and attached to the interior wall of the kiln, the curtains nearest the end being short enough to permit the jets of slurry to pass under them.

B. M. VENABLES.

**Devices for heating liquids.** J. U. HEMINGWAY (B.P. 349,320, 2.6.30).—The liquid is heated by admixture of steam which is supplied partly to an injector device and partly to a conical coil surrounding the outlet of the injector.

B. M. VENABLES.

**Means for breaking foam in steam boilers and other liquid evaporators.** S. B. BILBROUGH (B.P. 349,705, 31.3.30).—The apparatus is situated preferably within the steam drum. It comprises a casing the earlier part of which is completely filled by a bundle of small tubes, and the later part contains baffles and divides upwardly into a steam outlet and downwardly into a water outlet, the latter preferably extending below the water-level of the boiler.

B. M. VENABLES.

**Evaporation systems.** J. A. REAVELL (B.P. 349,567, 13.2.30).—The mixture of vapour and permanent gases emitted by a submerged-flame evaporator is rendered useful either by separating the gases and vapours by a diffusion diaphragm so that the latter may be used in the same way as steam, or the unseparated mixture is passed through preheaters and/or evaporators of the thoroughfare type, in which a strong flow of exhaust gas is permitted, preferably upwards and in the opposite direction to that of the condensing vapours.

B. M. VENABLES.

**Drying plant.** N. PANZIREFF (B.P. 349,583, 26.11.29).—Forms of apparatus which may be made semi-portable are described. The goods are conveyed in a zig-zag manner by conveyors and are dried by indirectly heated currents of air. The exhaust gases may be passed over either the entering or the leaving goods, as desired; in the latter case, an adjustable amount of the hottest air is used to preheat the goods.

B. M. VENABLES.

**Drying and/or cooling apparatus.** W. DRYDEN (B.P. 348,558, 12.12.29).—Material in the form of nuts or small cakes is guided by an upper perforated cone on to the periphery of a lower, inverted, perforated cone and is raked down to the apex by means of rotating

spiral blades while drying air is forced upwards through it. Provision is made for the separate removal of dust which passes through the perforations.

B. M. VENABLES.

**Absorption or adsorption refrigerating processes.** ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 348,350, 24.4.30. Ger., 3.5.29).—In refrigerating apparatus of this type, vapour of refrigerant is prevented from flowing back into and condensing in the evaporator (which effects the desired cooling) by the presence of another fluid ("sealing medium") which is either a liquid of different sp. gr. from, and only slightly miscible with, the refrigerant, or is a non-condensable gas. Examples are: (inert sealing medium, refrigerant, and absorbent, respectively),  $H_2$ ,  $NH_3$ , and charcoal or  $CaCl_2$ ; paraffin oil,  $SO_2$ , and  $SiO_2$  gel or charcoal; pentane,  $NH_2Me$ , and  $CaCl_2$  or  $SrBr_2$ .

B. M. VENABLES.

**Jaw crushers.** NORDBERG MANUFG. CO., Assees. of E. B. SYMONS (B.P. 348,851, 23.4.30. U.S., 9.4.30).—A jaw crusher is constructed to operate on the principles of the Symons cone crusher (*cf.* B.P. 243,926; B., 1926, 111), the jaws being inclined to the horizontal and the space between them towards the outlet end being parallel for a sufficient distance to ensure that every particle is nipped at least once in the parallel zone. The swing jaw is pivoted at the inlet end and the cheeks are attached to it. The fixed jaw is normally held up against abutments by strong springs which yield only to uncrushable pieces.

B. M. VENABLES.

**[Gyratory] crusher.** TRAYLOR ENG. & MANUFG. CO. (B.P. 348,402, 20.5.30. U.S., 24.5.29).—The top bearing of the crusher shaft is rigidly supported in horizontal directions, but can yield to uncrushable pieces in a vertical direction, being supported by a lever and spring.

B. M. VENABLES.

**Hammers for centrifugal crushing mills.** G. P. LUCAS (B.P. 348,736, 25.2.30).—A form of renewable hammer head in which the securing pin is freed from shearing stresses is described.

B. M. VENABLES.

**Grinding or pulverising machines.** BUCKLEY & TAYLOR, LTD., and T. BUCKLEY (B.P. 349,516, 27.2.30).—The apparatus comprises mills of the ball-mill and disintegrator types communicating and co-axial, but driven at different speeds suitable to their functions.

B. M. VENABLES.

**Effecting reaction between solids and gases.** SULPHUR & SMELTING CORP., Assees. of E. W. WESCOTT (B.P. 348,651, 13.2.30. U.S., 28.2.29).—The apparatus resembles a pebble mill, but it is nearly filled with pebbles, to such an extent that internal circumferential ribs extend right into the mass of pebbles and prevent direct flow of gases through the free spaces at the upper part; means are provided for admitting the gaseous and powdered solid reagents (preferably at opposite ends) and for withdrawing the reaction products while retaining the pebbles. To regulate the temp. the rotating cylinder may be surrounded by a stationary cylindrical casing containing in the lower part a heavy liquid, *e.g.* molten Pb, and in the upper an inert atm. Forms of specially-shaped "pebbles" are described.

B. M. VENABLES.



**Sifting of materials.** C. A. GESNEL (B.P. 348,166, 2.1.30. Fr., 19.10.29).—Material is delivered intermittently on to various parts of the bolting cloth (which is in the form of a vertical cylinder) by means of a turbine-like device. B. M. VENABLES.

**Centrifugal separators.** AKTIEB. SEPARATOR (B.P. 349,865, 8.9.30. Swed., 10.9.29).—An accessory for the cleanly discharge of collected solid matter when the bowl is stationary is described. B. M. VENABLES.

**Discharge of sludge from settling tanks.** A. BORSIG GES.M.B.H., and T. STEEN (B.P. 349,720, 11.4.31).—The settling tanks are situated radially round a sludge outlet to which the floors slope, and the inlets are above the lower ends, the decantation taking place at or close to the circumference. In operation only so much sludge as will run by gravity is discharged down the slope; the outlet is then closed and the remaining clay-like sludge from the upper end is pushed to the lower where it will act as a cushion and lubricant for the sand deposited from the next charge. B. M. VENABLES.

**Settling apparatus for separation of solid and liquid matters.** C. STILL (B.P. 348,888, 20.5.30).—A receiver, e.g., for ammonium salts from a saturator, is formed as a tilting vessel with one trunnion solid and attached to the tilting mechanism and the other hollow and forming an overflow for mother-liquor. B. M. VENABLES.

**Mixing and centrifuging machine for moulding substances.** K. LEHMANN (B.P. 348,968, 5.8.30. Ger., 5.8.29).—A rotating pan for moulding sand, mortar, and the like is provided with fixed inclined stirring blades and with blades rotating at centrifugal speed on a transverse shaft, which dip into the pan and fling the material about, either back into the pan or out of it according to the position of a curved flap-door in a surrounding hood. B. M. VENABLES.

**Filters.** A. C. HANDLEY (B.P. 349,028, 19.2.30).—A closed tank is divided into three compartments containing prefilter, filter medium, and filtrate, respectively; the filter medium comprises vertical walls of coir and/or cellulose silk the space between which is kept filled with powdered coir or sand fed in from a hopper so as to prevent short-circuiting of unfiltered liquid across the top. The device is primarily intended for filtering oils. B. M. VENABLES.

**Construction of filters.** F. G. RILEY (B.P. 349,262, 1.5.30).—In a filter composed of compressed plaques of paper pulp or other material, the pressure is regulated by a nut on the end of an axial outlet pipe, which nut also serves to break the force of the incoming prefilter. B. M. VENABLES.

**Filtration of liquids.** OLIVER UNITED FILTERS, INC., Assees. of C. W. MOORE (B.P. 348,556, 9.12.29. U.S., 10.12.28).—The pulp in the reservoir of a vacuum filter is kept homogeneous by injecting filtrate or a similar liquid into the lower part where the sand collects. A stirring device may also be used. B. M. VENABLES.

**Filtration or straining of liquids.** C. B. THORNE (B.P. 349,466, 25.2.30).—Apparatus suitable for the treatment of waste water from paper mills comprises a tank or enlargement of a launder containing a number of elements with vertical filtering walls which, when

they are choked, are lifted out by a belt which also seals the opening for outflow of filtrate belonging to that element. Mechanism for automatic operation is described. B. M. VENABLES.

**Devices for distillation and evaporation *in vacuo*.** O. ANGELUCCI (B.P. 348,442, 27.6.30. Ger., 8.7.29).—Within an all-containing vacuum chamber the distillate is allowed to flow down through one or more larger spiral tubes surrounding smaller heating tubes and provided with vents so that the distillate can pass into the vacuum chamber, where it is condensed on a central cooled element. The condensate and residue are withdrawn through separate outlets. B. M. VENABLES.

**Controlling the pressure of vapours produced in stills.** H. W. ROBINSON (B.P. 348,397, 19.5.30).—More vapour is generated than is required to maintain the desired const. pressure in the delivery pipe, the excess being allowed to pass up a long pipe through a relief valve (which determines the pressure) at the top, then condensed, and allowed to flow back into the still through a barometric column sufficient to balance the pressure in the still. B. M. VENABLES.

**Purification of feed water.** O. BÜHRING (B.P. 349,554, 28.2.30. Ger., 7.3.29. Addn. to B.P. 324,428).—The sludge is taken from the boiler at a higher pressure than that in the thermic vessel, its pressure is reduced by ebullition, and the steam passed to the thermic vessel together with steam direct from the upper part of the boiler. The sludge is passed to the chemical stage together with any sludge formed in the thermic vessel. Alternatively, the sludge from the boiler may pass direct to the thermic vessel. B. M. VENABLES.

**Mixing apparatus for liquids.** O. RITSCHEL (B.P. 348,391, 16.5.30).—A reagent or adjuvant, which may be corrosive and is to be added in small but strictly proportional quantities to a continuous stream of a main liquid, is placed in a completely filled closed container and displaced therefrom by small quantities of the main liquid delivered by a small pump operated by a meter-motor in the main stream. The whole system is subjected to the pressure of the main liquid, the motor and pump being within one casing without high-pressure glands, and the suction of the pump and delivery of the adjuvant being both on the outlet side of the meter, the former slightly before the latter. If the two liquids are nearly equal in sp. gr. a bellows-like diaphragm may be used to separate them in the closed container, or the latter may be divided into two and a lighter inert liquid used as a diaphragm. B. M. VENABLES.

**Prevention of excessive frothing in vats and other containers.** DISTILLERS CO., LTD., and E. A. MEYER (B.P. 349,288, 16.5.30).—A trough of froth-killing agent of lower sp. gr. than that of the liquid, e.g., molten fat for yeast, is placed in the vat above the liquid level. When froth rises and spills into the trough it is condensed by the agent, falls below it and displaces an equal vol. of the agent into the main body of liquid, the action continuing automatically until the agent is used up. B. M. VENABLES.

**Treating liquids with gases.** W. E. DURRAD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,244, 21.2.30).—A number of tubes of decreasing diam.

and equal length are loosely assembled within each other, are rotated in an inclined position, and are supplied with liquid which flows either con- or counter-current to the gas. The tubes (except the outermost) may be perforated or made of gauze. B. M. VENABLES.

**Purification of gases.** W. H. OWEN (B.P. 349,233, 10.4.30).—The gases enter the large end of a perforated cone, at which point a whirling propeller is provided; at the smaller end are liquid sprays, and just beyond, if desired, another whirling propeller and, finally, a fan for producing draught only are provided. The separated matter leaves transversely through the perforations and beyond the second propeller.

B. M. VENABLES.

**Air-conditioning plants.** A.-G. BROWN, BOVERI & CIE. (B.P. 349,355, 11.7.30. Ger., 19.7.29).—In a conditioning system comprising a mixing valve for old and new air, an injection chamber, moisture remover, and reheater, when a temp. below that obtainable by the injected sprays is desired, the air itself is used as the refrigerant. By the movement of a pair of valves, it is subjected to compression before spraying and to expansion (with recovery of some work) after spraying, and is thus cooled to below the temp. of the spray-water.

B. M. VENABLES.

**Oil extractor and filter [for compressed air].** W. H. TAYLOR and C. SHAW (B.P. 349,729, 22.4.30).—A casing is provided with a number of concentric baffles and vanes which afford a large surface for the collection of oil and cause abrupt changes in direction. The air finally passes through a filter medium.

B. M. VENABLES.

**Storing and transporting liquefied gases of low b.p.** C. W. P. HEYLANDT (B.P. 348,838, 14.4.30).—That portion of the liquefied gas that it inevitably vaporised is led through a jacket surrounding the necessary pipe connexions to the vessel, whereby heat conducted along them is prevented from reaching the vessel.

B. M. VENABLES.

**Treatment of gases with liquids.** SOC. ANON. INDUSTRIA AMMONIACA, and F. LOPEZ (B.P. 349,230, 9.4.30).—The apparatus comprises a number of superposed chambers with bubble caps in the divisions, these caps comprising upflow pipes with loose hoods having resilient washers inside the top, forming non-return valves for the gases. The down-flow of liquid takes place through coils which are cooled in a surrounding liquid bath, so that the apparatus is suitable for the exothermic dissolution of ammonia. B. M. VENABLES.

**Hardness-testing machine.** C. H. WILSON (B.P. 348,915, 10.6.30. U.S., 16.4.30).—In an apparatus of the type in which the penetration of a sample is measured between the application of a minor and major load, the plunger is released from its guides, except a very narrow one at the upper end, by the application of a preliminary load even less than the minor load.

B. M. VENABLES.

**Apparatus for the rapid charging of a furnace.** H. GARREAU (B.P. 349,918, 1.3.30. Fr., 7.10.29).

**Preheater and degasifying plant for steam turbines and steam power units.** MASCHINENFABR. OERLIKON (B.P. 349,878, 24.9.30. Switz., 30.10.29).

**Liquid heating and storage installations [for domestic hot water etc.].** G. N. HADEN & SONS, LTD., and S. S. JEWSEBURY (B.P. 349,762, 16.5.30).

**Means for compensating for changes in liquid volume due to temperature.** M. CHARLES (B.P. 349,834, 22.7.30. Fr., 14.8.29).

**Heat-transferring devices for use with refrigerators.** ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 349,798, 16.6.30. Ger., 19.6.29).

**Evaporators for refrigerating machines.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. STEENSTRUP (B.P. 349,860, 28.8.30. U.S., 28.8.29).

**Pretreatment of fuel for furnaces.**—See II. **Retort furnace.**—See X. **Intensity of colours.**—See XI. **Decolorisation of solutions.**—See XVII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Austrian coals and the possibilities of their economic utilisation.** E. GALLE (Sparwirtschaft, 1931, 9, 185—189).—Low-temp. carbonisation, the production of lubricating oil from the tar, catalytic hydrogenation, pulverised-fuel firing, complete gasification, etc. are discussed in relation to the utilisation of Austrian coals.

A. B. MANNING.

**Clean coal in the gas industry.** R. LESSING (Gas J., 1931, 94, 815—825).—The case for coal cleaning and an account of the "Clean Coal" process are presented. Tests quoted show the advantage of cleaned coal for carbonisation and water-gas production.

A. KEY.

**Vegetable fuels.** II. T. BATUECAS (Anal. Fis. Quím., 1931, 29, 294—299).—Detailed analyses and the calorific val. are given for charcoals obtained from *Quercus lusitanica*, *Erica arborea*, and *Alnus lutinosa*. Dulong's formula yields low values for the calc. calorific val., whilst with the formulæ of Mahler and of Nikitin the error rarely exceeds 1%.

H. F. GILLBE.

**Processes of formation and combustion of fuels.** E. BERL (Petroleum, 1931, 27, 399—407).—Factors influencing combustion, thermal decomp., and condensation of hydrocarbons and the reduction of CO to aliphatic compounds are surveyed. The influence of pressure and length of C chain on the explosive limits of hydrocarbon vapours is discussed. Investigation of the influence of PbEt<sub>4</sub> and Fe(CO)<sub>5</sub> and of finely-dispersed Pb and Fe indicates that these materials inhibit self-ignition or knocking in motor fuels by reacting with intermediate products of partial combustion.

H. E. BLAYDEN.

**Routine test of the inflammability of mine dusts.** A. L. GODBERT (Safety in Mines Res. Board, 1931, Paper No. 68, 9 pp.).—The apparatus consists of a transparent SiO<sub>2</sub> tube, 7 in. ×  $\frac{3}{4}$  in., heated externally by either gas or electricity to 820°, through which a mixture of O<sub>2</sub> and 1 g. of 100-mesh (I.M.M.) dust is blown at a definite pressure. Inflammable dusts give a white flame; those just unflammable reddish-orange sparks. The inflammability of a coal dust is determined by adding just sufficient incombustible dust to render it incapable of propagating flame, and expressing the incombustible dust as a percentage; the

uninflammability is ascertained by blending the dust with coal dust.

A. H. EDWARDS.

**Intermittent vertical chambers and coal- and coke-handling plant at Southall.** J. S. THORMAN (Gas J., 1931, 94, 763—783).—Descriptive, with an account of satisfactory tests, showing flexibility of output and calorific val. of gas (which contains little  $N_2$ ), high steaming efficiency, and the production of a coke which is denser and has a higher resistance to shatter than that obtained from continuous vertical or horizontal retorts.

A. KEY.

**Controlling pressure conditions within coal-gas retorts.** R. N. WEBB (Gas J., 1931, 94, 826—830).—The pressure conditions prevailing in the foul-gas main of a setting of horizontal retorts are reproduced inside the exhaustor governor bell by means of an impulse pipe 3 in. in diam. The varying pressures on the bell are arranged to operate the steam-throttle valve of the exhaustor, so that it requires very little attention to keep the pressure in the retorts almost constant.

A. KEY.

**Combustion processes in motors and the six-stoke principle.** E. TERRES (Z. angew. Chem., 1931, 44, 509—519). Curves are given showing the dependence of the degree of completeness of combustion on the compression, speed of revolution, and ignition point and the relation between the composition of the products of combustion and the water-gas and other equilibria. The velocity of combustion of explosive mixtures of fuel and air increases at first with increasing initial pressure, passing through a max. at 15 atm. The greatest velocities are attained in systems consisting of finely-dispersed liquid fuel in air, and in agreement with this observation is the striking result that pre-heating the mixture reduces the velocity of combustion. The results are discussed from the mechanical point of view, as applied to the construction of internal-combustion engines.

E. S. HEDGES.

**Peroxides in the gases of internal-combustion engines.** DUMANOIS, MONDAIN-MONVAL, and QUANQUIN (Compt. rend., 1931, 192, 1158—1160).—With hexane as fuel, when the temp. rose to about 270° knocking began, and simultaneously peroxides and aldehydes appeared in the gases. On stopping ignition the motor continued to work for 1 min. or so with much diminished noise and absence of flame (cf. A., 1930, 167, 1157). The immediate cause of knocking is therefore detonation of peroxides.

C. A. SILBERRAD.

**Manufacture of charcoal in Japan.** I. MIURA (Ind. Eng. Chem., 1931, 23, 631—634).—Charcoal as made in Japan is either black charcoal made in earthen ovens, or white charcoal made in stone ovens withdrawn while hot and covered with earth etc. In the former case the temp. remains low and the yield is 15—20% on undried wood. White charcoal is harder and is made by raising the temp. at the end of carbonisation, thus causing combustion of the bark. The yield is 11—13%. Physical properties of each sort are described. The author has devised a scale of hardness, using a series of metals and alloys which is compared with Mohs' mineral scale. It is widely used in Japan in evaluating charcoal.

C. IRWIN.

**Flame-pressure process for carbon black.** A. W. FRANCIS (Ind. Eng. Chem., 1931, 23, 612—617).—The best C black is made by deposition on a metal plate from a flame of natural gas burning in the air. The low yield (about 3%) is due to the fact that the upper limit of  $CH_4$  concentration in air for inflammability is 14%, and hence in all parts of the flame there is enough  $O_2$  present to burn C to CO. Dilution with incombustible gases or enrichment with  $O_2$  is useless, but the upper limit of inflammability is raised by increased pressure (at 50 atm. it is 29%); small-scale experiments show that this gives increased C yields. A bomb was used into which gas was passed from an auxiliary bomb under higher pressure. Yields up to 9% varying with the burner tip design were obtained. Theoretical calculations suggest an optimum yield of about 30% at 34 atm. The process is not likely to be economical with the present low value of natural gas.

C. IRWIN.

**Some properties of carbon black. I. Adsorption.** W. B. WIEGAND and J. W. SNYDER (Ind. Eng. Chem., 1931, 23, 646—649; India-Rubber J., 1931, 81, 13—18).—The adsorptive qualities of C black, as measured by shaking 1 g. with 50 c.c. of a solution of 2 g. of diphenylguanidine in 1 litre of EtOH for 2 hr. and determining the amount of base in 25 c.c. of the filtrate, show a fair correlation with the effect of the C black on the vulcanisation of rubber, particularly when an org. accelerator is also used. The adsorption is much more marked in the alkaline than in the acid range. The test may with advantage be used to obtain a preliminary idea of the vulcanising characteristics of C black in organically accelerated mixtures, but it is not capable of predicting precisely the relative behaviour of successive samples. C blacks activated by heating in air or steam show a marked increase in adsorption of diphenylguanidine, whilst blacks deactivated by heating in absence of air show greatly reduced alkaline and slightly increased acid adsorption, which probably explains the known anomalous I adsorption of heated blacks. In litharge mixings, without added fatty acid, highly adsorbent blacks spoil vulcanisation by removing the natural fatty acids of the rubber; it is therefore necessary to adjust the relative proportions of added fatty acid and black in order to ensure the best results. If a min. proportion of fatty acid is desired in a litharge mixing a black of low adsorption must be selected. For entirely unaccelerated mixtures, a heat-deactivated black of low adsorption gives improved results. The adsorptive properties of C black are of advantage in the production of rubber insulating materials and in the treatment of insulating oils. Highly adsorptive, heat-activated blacks advance the vulcanisation of rubber with *m*-dinitrobenzene.

D. F. TWISS.

**Technical active charcoals and other adsorbents (with special reference to their application in the sugar industry).** K. SKUMBURDIS (Kolloid-Z., 1931, 55, 150—155).—Comparison of the adsorptive capacity of a number of active and inactive charcoals and 2 kinds of fuller's earth in aq. isoamyl alcohol solutions with the effect of these adsorbents on the inversion of sucrose indicates that the inversion effect noted in some cases is most probably due to small amounts of acid adsorbed

by the charcoal. Charcoals containing H and the basic fuller's earths do not invert sucrose and probably neutralise the traces of acid produced by the slow decomp. of sucrose. Active charcoals do not accelerate the inversion of sucrose by capillary active or inactive acids, but, on the other hand, they withdraw a portion of the acid and reduce the amount of sugar inverted. The greater is the adsorbability of the acid, the greater is the inhibitive effect of the charcoal. The addition of a capillary active, indifferent substance such as *iso*-amyl alcohol decreases the adsorption of the acid slightly.

E. S. HEDGES.

**Water-gas process: carbon and thermal balances.** J. G. KING and B. H. WILLIAMS (Dept. Sci. Ind. Res., Fuel Res., Tech. Paper No. 30, 1931, 31 pp.).—A test has been carried out on a standard Humphreys and Glasgow generator in which determinations of C have been made at various points. The C burned in the hot clinker during its removal from the generator was shown to be negligible. The period of test, 35–36 hr., comprised 4 complete clinkering to clinkering periods. The fuel used was a horizontal-retort gas coke. The total C in the blow gases was determined from the vol. of air delivered to the generator and the average composition of the blow gases. The vol. of water-gas produced was measured by means of the photographic recorder previously described (B., 1931, 282). The C balance shows a loss of 2.2%, which, in view of the possible errors of measurement in the determinations involved, is regarded as satisfactory. In order to be able to draw up a thermal balance also, measurements were made of the temp. of the air and coke entering the generator, and of the blow gas, water-gas, and clinker and ashes leaving the generator. These observations were made over one complete clinkering to clinkering period, which is regarded as a unit phase of the process. The value 96,700 C.H.U./lb.-mol. was adopted for the heat of reaction  $C + O_2 \rightarrow CO_2$ ; this value is higher than that adopted by Travers, who assumes that the C exists in the coke in the graphitic form (J.S.C.I., 1924, 43, 355 T), and is based on determinations of calorific val. of 23 gas cokes. The thermal balance shows a loss due to radiation and convection, and heat lost as heat of reaction and sensible heat of the gases issuing from the generator during clinkering operations (and errors), of 25.7 therms, or 2.9 therms/hr. This difference is of the same order as the sum of the radiation and convection losses calc. from measurements of the surface temp. of the generator (1.5 therms/hr.), and the estimated heat lost during clinkering operations (1–2 therms/hr.).

A. B. MANNING.

**Determination of phenols in water from coke-benzol manufacture.** L. I. KAZATCHKOV, M. M. KRASOVITSKAJA, and O. G. TEMIR (Ukrain. Chem. J., 1930, 5, [Tech.], 205–223).—Methods for determining traces of phenols in water are reviewed. Shaw's method (B., 1929, 771) is recommended.

E. B. UVAROV.

**Determination of phenols in the ammonia and spent liquors from gasworks, coke ovens, etc.** H. BACH (Gas- u. Wasserfach, 1931, 74, 331–334).—Liquors containing benzol are first made alkaline and then extracted with  $Et_2O$ , the  $Et_2O$  dissolved in the

liquor being finally removed by evaporation. 100 c.c. of liquor are then acidified and distilled until the whole of the phenols are in the distillate. If sulphides are present,  $CuSO_4$  is added in excess and the liquid boiled under reflux until no more  $H_2S$  is evolved.  $BaCl_2$  solution (5 c.c.) is added, the liquid made up to 500 c.c. and filtered, and 50 c.c. of the filtrate are treated with  $KBr-KBrO_3$  solution and acid. After  $\frac{1}{2}$  hr. excess Br is determined in the usual way with KI and  $N-Na_2S_2O_3$  solution. An accuracy of 25 mg./litre is claimed, hence the method is not suitable for liquors containing very little PhOH. To determine PhOH in benzol, it is extracted with NaOH solution, the alkaline solution washed with  $Et_2O$ , made acid, and distilled as above.

A. KEY.

**Semi-direct recovery of ammonia in gas-works' practice, and the recovery of tar acids from ammoniacal liquor.** A. L. HOLTON (Gas J., 1931, 94, 790–800).—A description of plant at Manchester, dealing with 3–10 million cu. ft. of gas per day. Violent oscillations of pressure in the saturator are prevented from communicating to the retort house by the substitution of "Connerville" for rotary meters. Rock salt formation in the saturator also caused trouble. Liquor circulating in the hydraulic mains is allowed to attain a concentration of 14–18%  $NH_4Cl$ , the salt being recovered by evaporation. The ammoniacal liquor is freed from phenols by washing with a countercurrent flow of benzol in a tower, and is then passed through active C to remove higher tar acids and dissolved benzol. The purification express by the O-absorption val. is 88%, and the loss of benzol should not exceed 6.6 gals. per 100 tons of liquor. The phenol is recovered from benzol by distillation.

A. KEY.

**Corrosion of tar stills.** W. A. DAMON (67th Annual Rep. on Alkali etc. Works, 1930, 29–37).—The loss in wt. of strips of mild steel immersed in tar samples and in aq. and org. solutions of various tar constituents was determined.  $NH_4$  salts and dihydric phenols both showed considerable effects in aq. solution, much less in xylene etc. Experiments with tars of known  $NH_4Cl$  content definitely established the corrosive effect of the latter, but it is not the only cause of corrosion, and the relation to  $NH_4Cl$  concentration is not linear. Phenols appear to exert a partial protective action against corrosion by  $NH_4Cl$ . During the stages of tar distillation, corrosion increases as distillation proceeds in most cases, and is maximal during steaming. The water-washing of tar reduces corrosion about 85%.

C. IRWIN.

**Lignite tar. III. Ketones of lignite tar oil. IV. Aromatic and hydroaromatic compounds of lignite tar.** J. HERZENBERG and E. VON WINTERFELD (Ber., 1931, 64, [B], 1025–1036, 1036–1044; cf. A., 1927, 551).—III. Although ketones form a considerable portion of the oxygenated compounds of lignite-gas benzene the nature of the preponderating remainder is unknown. This is true in still greater degree for the tar oils. Alcohols and esters comprise only a very small proportion of the oxygenated compounds, and, although terpene-like compounds are present, it appears probable that furan derivatives exist therein. The neutral gas benzene is

fractionated and the ketones are isolated from the individual fractions by phenylhydrazine; ethyl *n*-amyl ketone (semicarbazone, m.p. 115–116°), methyl *n*-hexyl ketone (semicarbazone, m.p. 121–122°), acetophenone, and cyclopentanone (dianisylidene derivative, m.p. 215°) are identified.

[With W. FISCHER.] The ketones from refined lignite generator tar oil, b.p. 70–150°/12 mm., are separated as their hydroferrocyanides. The following products are isolated: semicarbazone  $C_9H_{15}ON_3$ , m.p. 221·5°, not derived from tetrahydroacetophenone; semicarbazones  $C_8H_{13}ON_3$ , m.p. 225–226°, 236°, 221°, and 224–226°, respectively.

IV. The  $C_6H_6$  content of lignite gas benzene is much lower than hitherto assumed. The  $C_{10}H_8$  fraction of the generator tar oil contains 8%  $C_{10}H_8$  and only 2·5% of hydrogenated  $C_{10}H_8$ , all of which are unsaturated. The free occurrence of *m*-substituted derivatives of  $C_6H_6$  and  $C_{10}H_8$  is attributed to their formation from the resins of bitumen-A and -B.

[With W. PASCH.]  $C_6H_6$  is determined in lignite gas benzene by means of  $NH_3 + Ni(CN)_2$  (A., 1903, i, 469). In the higher fractions PhMe is determined by direct mononitration and, after removal of the bulk of the unsaturated compounds by means of  $H_2SO_4$  of gradually increased concentration, *m*-xylene, mesitylene, and  $\psi$ -cumene are separated as 2 : 4 : 6-trinitro-*m*-xylene, m.p. 180–181°, trinitromesitylene, m.p. 230–233°, and 3 : 5-dinitro- $\psi$ -cumene, m.p. 171–172°.  $C_{10}H_8$  is identified as the picrate, but hydrogenated  $C_{10}H_8$  does not appear to be present. Dehydrogenation of partly hydrogenated  $C_{10}H_8$  of generator tar oil is more readily effected with  $Ni-Al_2O_3$  at 360–380° than with S. The  $C_{10}H_8$  homologue  $C_{13}H_{14}$  (picrate, m.p. 123–124°) is not 1-isopropyl-naphthalene, which is synthetically prepared. 1- $\alpha$ -Hydroxyisopropyl-naphthalene, m.p. 96°, prepared from 1-naphthyl methyl ketone or Et 1-naphthoate and  $MgMeI$ , is dehydrated by  $Ac_2O$  and reduced by Na and EtOH to a mixture of 1-isopropyl-di- and -tetra-hydro-naphthalenes, dehydrogenated to 1-isopropyl-naphthalene, b.p. 132–134°/10 mm. (picrate, m.p. 85–86°).

H. WREN.

**Phenols in the tar of Saar coal.** J. PAIRA (Ann. Office Nat. Combust. liq., 1930, 5, 651–676; Chem. Zentr., 1931, i, 1701).—The fractions, after removal of neutral oil, were subjected to fractionation at 10 mm. between 80° and 130°. The lower fractions contained PhOH and its mono- and di-methyl derivatives; the fraction of b.p. 176–181° contained 70% of PhOH, possibly owing to superheating when the tar was produced. Tar from a Salerni furnace contained only 1% of PhOH. The fraction of m.p. 180–210° contained *o*-, *m*-, and *p*-cresol, 1 : 3 : 4- and 1 : 3 : 5-xenol.

A. A. ELDRIDGE.

**Asphalt from the cracking processes.** G. EGLOFF and J. C. MORRELL (Ind. Eng. Chem., 1931, 23, 679–680).—The production of asphalts from liquid residue derived from cracking operations is shown to be possible, from which it appears that polymerisation as well as decomposition takes place during cracking. The properties of the asphalts obtained in this way from a number of crude oil residues are tabulated. These asphalts meet the A.S.T.M. (D102–24r and D103–24r) speci-

cations except as regards solubility in  $CCl_4$ . The possibility of producing satisfactory asphalts from kerosene by cracking is indicated.

T. A. SMITH.

**Light oil from water-gas tar.** N. A. ORLOV (J. Appl. Chem., Russia, 1930, 3, 585–587).—The characteristics of the oil are recorded; it contains 1·5–2% of styrene (separated as dibromide) and 4·7% of indene (separated by Weissgerber's method).

CHEMICAL ABSTRACTS.

**Composition of neutral oils obtained from the primary tars of Rumanian lignite, and volumetric determination of saturated and unsaturated aromatic hydrocarbons in this oil.** N. DĂNĂILĂ and T. D. IONESCU (Bul. Chim. Pura appl., Bukarest, 1931, 31, 229–273; Chem. Zentr., 1931, i, 1546–1547).—The neutral oil fractions from the primary tars obtained up to 500° are richer in unsaturated hydrocarbons than are the corresponding petroleum fractions. The  $C_6H_6$  + PhMe + xylene fraction is < 1% of the original tar. The oils are dextrorotatory.

A. A. ELDRIDGE.

**Design of liquid-phase cracking equipment.** P. RYABUUKH (Azerbaid. Neft. Choz., 1930, No. 12, 76–82).—The theoretical considerations involved are discussed.

CHEMICAL ABSTRACTS.

**Cracking of Rumanian gas oil.** C. H. ALBERDING and W. A. KEIGHTLEY (Petroleum, 1931, 27, 407–409).—By cracking Rumanian gas oil (*d* 0·86) in a Dubbs cracking plant under 227 lb./sq. in. (about 16 atm.) pressure and redistilling the pressure distillate after washing with alkali and acid, a 60–65% yield of petroleum spirit (*d* 0·73–0·74) of high antiknock val. was obtained without recirculation. The residue from redistillation could be recycled. Various oils or low-temp. tars could be treated in the experimental plant used, which is described.

H. E. BLAYDEN.

**Refining of cracked spirit by physical methods.** R. A. WISCHIN (Petroleum, 1931, 27, 410–413).—Absorption, adsorption, and oxidation methods of refining petroleum spirit from cracking processes are briefly reviewed. Light spirit from brown-coal tar was fractionated, washed with acid and alkali, and then fractionated over glass balls or over ferruginous bauxite with simultaneous treatment by ozonised air, ultra-violet light, or a high-frequency electric discharge. It was also fractionated over granulated Sn, washed, and refractionated. The last-mentioned four methods removed the greatest amounts of gum-forming substances.

H. E. BLAYDEN.

**Contact filtration of bright [mineral oil] stocks.** V. MATUSEVICH (Azerbaid. Neft. Choz., 1930, No. 12, 64–75).—A mixture of long residuum from Dossor and Makat crude oil was treated with acid and, in two stages, with clay. Optimal quantities and conditions were determined.

CHEMICAL ABSTRACTS.

**Synthetic lubricating oils. Relation between chemical constitution and physical properties.** F. W. SULLIVAN, JUN., V. VOORHEES, A. W. NEELEY, and R. V. SHANKLAND (Ind. Eng. Chem., 1931, 23, 604–611).—A review of much of the literature dealing with the production of lubricating oils by polymerising olefines in the presence of  $AlCl_3$ . In general, it is shown that the

temp.-viscosity coeff. decreases with increasing mol. wt. of the raw material. The oils obtained by polymerising cyclic olefines show the characteristics of naphthenic oils to a high degree. The effect of time, temp., and amount of  $\text{AlCl}_3$  on the polymerisation yield and viscosity was investigated; 30 hr. at  $54.5-57.2^\circ$  and 3% of  $\text{AlCl}_3$  were found the most desirable. A commercial process has been developed.

T. A. SMITH.

**Viscosity-temperature relationship of lubricating oils.** R. G. SLOANE and C. WINNING (Ind. Eng. Chem., 1931, 23, 673-674).—Mathematical expressions for the variation of viscosity with temp. are either limited in the temp. range to which they apply or are too involved for graphical representation. The expression  $(\log \eta_k - A)(t - B) = C$  is simpler, but contains three consts. The const.  $B$  was found to show least variation and to introduce no very serious errors when its val. is taken as  $-135$ . The expression for the kinematic viscosity  $\log \eta_k = c/(t + 135) + A$  lends itself readily to graphical representation.

T. A. SMITH.

**Physical properties and constitution of mineral lubricating oils.** II. S. KYROPOULOS (Z. physikal. Chem., 1931, 154, 358-363; cf. B., 1929, 931).—A reply to criticisms by Bielenberg (B., 1930, 849).

F. L. USHER.

**Physical properties and constitution of mineral lubricating oils.** W. BIELENBERG (Z. physikal. Chem., 1931, 154, 364-369; cf. preceding abstract).—Polemical against Kyropoulos.

F. L. USHER.

**Composition and crystal form of the petroleum waxes.** S. W. FERRIS, M. C. COWLES, JUN., and W. M. HENDERSON (Ind. Eng. Chem., 1931, 23, 681-688).—Petroleum wax was freed from oil, fractionally distilled *in vacuo*, and the fractions themselves were fractionally crystallised from ethylene dichloride. The products consisted partly of plate-shaped crystals, partly of needles, and partly of crystals having no very definite shape. The first have the highest m.p. for a given b.p., and the second the lowest. Only the first type exists in the lower-boiling fractions. Evidence is produced which indicates that the cryst. form is characteristic of different hydrocarbon series and independent of the solvent. If crystallisation of both types is simultaneous, the needles tend to impress their form upon the plates. Photomicrographs are given. C. IRWIN.

**Refractometric investigation of paraffin products.** M. FREUND (Petroleum, 1931, 27, 409-410).—The paraffin content of artificial mixtures of paraffin and filtrate oil may be calc. fairly accurately from the  $n$  val. of the paraffin, the oil, and of the mixture by application of a simple mixture formula. By determining  $n$  for the final products of petroleum refining, refractometric examination of the petroleum and calculation of the paraffin content offers a method of process control quicker than the tedious analytical method.

H. E. BLAYDEN.

**Determination of water in power alcohol.** K. R. DIETRICH and C. CONRAD (Z. angew. Chem., 1931, 44, 532-534).—Available methods are surveyed, and a method based on absorption by acid of the  $\text{NH}_3$  evolved

when the liquid (50 g.) is allowed to drop slowly on to a 2-fold excess of  $\text{Mg}_3\text{N}_2$ , and titration of the excess of acid, is described. The  $\text{Mg}_3\text{N}_2$  reacts very readily, and only traces of  $\text{H}_2\text{O}$  may be determined; anhyd.  $\text{EtOH}$  does not react, but with  $\text{MeOH}$  at concentrations higher than about 60%  $\text{NH}_2\text{Me}$  is evolved. Accurate results are obtainable and the method is applicable to  $\text{Et}_2\text{O}$ ,  $\text{COMe}_2$ , and various types of motor spirit. Ca and Al nitrides are not satisfactory. H. F. GILLBE.

**Oil-well cement.**—See IX. **Dust removal from gases.**—See XI. **Hydrogenation of oils.**—See XII. **Fruit-tree emulsions.** **PhOH as germicide.**—See XVI. **Bagasse utilisation.**—See XVII.

#### PATENTS.

**Apparatus for preliminary treatment of fuel during its supply to furnaces.** AMER. ENG. CO. (B.P. 348,106, 3.1.30. U.S., 18.11.29).—The fuel is passed through a drying chamber, through which are circulated furnace gases of suitable temp., and thence to a vertical retort, externally heated by high-temp. furnace gases, wherein it is distilled with recovery of the volatile by-products. The treated fuel is fed directly from the bottom of the retort, *e.g.*, by means of a reciprocating ram and an underfeed stoker, to the combustion chamber of the furnace. A. B. MANNING.

**Apparatus for preheating materials containing coal and oil.** J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 348,189, 10.2.30).—The material is passed through a preheating coil contained in a combustion chamber, wherein a suitable fuel, *e.g.*, coal gas, natural gas, or vaporised fuel oil, is burned in stages in such a manner as to heat the coil uniformly. Thus a mixture of air and fuel, containing a great excess of either, is supplied to one end of the combustion chamber and the rest of the fuel or air necessary for complete combustion is admitted at suitable points along the chamber. A. B. MANNING.

**Distillation of coal.** F. PUENING (B.P. 348,361, 30.4.30. Ger., 30.4.29. Addn. to B.P. 312,238; B., 1930, 936).—The retort is modified by providing between the coking chambers and the heating gas ducts a gastight head-plate, which rests on water-cooled beams, supports the walls of the coking chambers, and carries the ducts for the entrance and exit of the heating gases. The head-plate is provided with expansion folds to ensure flexibility. The coal is charged into the chambers from the inner side of the annular arrangement by means of a machine which rotates with the inner part of the shell. Provision is made for levelling the charges in the chambers and returning any excess coal to the charging machine. A. B. MANNING.

**Cooling the hot coke discharged from coke ovens.** N.V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co. G.M.B.H. (B.P. 348,207, 14.2.30. Ger., 14.2.29).—The hot coke is charged into a shaft wherein it is cooled with the simultaneous production of water-gas by introducing steam or  $\text{H}_2\text{O}$  countercurrent to the coke. The small coke is separated from the larger as it leaves the oven and is charged into a chamber forming an extension of the upper part of the shaft, which is externally heated to a temp. sufficiently high to ensure

the practically complete conversion therein of the  $\text{CO}_2$  in the water-gas into  $\text{CO}$ . A. B. MANNING.

**Manufacture of products from coal and the like solid carbonaceous materials, asphalts, various petroleum products, tars, phenols, cresols, and the like by the action of hydrogen at elevated temperatures and pressures.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 348,243, 20.2.30).—The process is carried out in the presence of a catalyst prepared by treating metal oxides or hydrated metal oxides, *e.g.*, Fe or Co oxides, with  $\text{H}_2\text{S}$  prior to hydrogenation. The oxides are heated to 200–300° during at least part of the treatment with  $\text{H}_2\text{S}$ . A. B. MANNING.

**Manufacture of products from coal, tars, oils, and the like carbonaceous materials.** II. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 348,721, 21.2.30).—Catalysts which are used in the destructive hydrogenation of carbonaceous materials, *e.g.*, Fe, Co, Ni, Mo, etc., or their oxides, supported on carriers such as absorptive C or pumice, are reactivated by being heated in the presence of air or other oxidising gases. A. B. MANNING.

**Preparation of catalysts for use in the manufacture, by destructive hydrogenation, of hydrocarbons from carbonaceous material.** I. G. FARBENIND. A.-G. (B.P. 348,690, 15.2.30. Addn. to B.P. 247,584; B., 1927, 595).—The catalysts are prepared by treating oxides of metals of groups II and VI, *e.g.*, oxides of Mo, W, Cr, or U, mixed with oxides of Zn, Mg, or Al, etc., with volatile S, Se, or Te compounds, in the presence of  $\text{H}_2$  at raised temp. (Cf. B.P. 273,228; B., 1927, 695.) A. B. MANNING.

**Treatment of coals, tars, mineral oils, and the like, by hydrogenation under high pressure and at high temperature.** J. E. LÖFFLER (B.P. 348,449, 3.7.30).—The reaction components, the reaction vessel, and the reaction products are heated, or cooled, by means of high-pressure steam. The steam is preferably circulated in closed circuits, *e.g.*, by means of pumps, the heating or cooling action being controlled by varying the vol. in circulation or the temp. The circuit may include a heat accumulator. A. B. MANNING.

**Manufacture of hydrocarbons from coal, tars, mineral oils, and the like.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,252, 26.2.30).—In the destructive hydrogenation of carbonaceous materials under pressure, the desired reaction temp. is maintained by introducing O-containing compounds of C, *e.g.*,  $\text{CO}_2$  or phenols, which under the working conditions are reduced to hydrocarbons with evolution of heat, at one or more points in the course of the hydrogenation where an undesired fall of temp. would otherwise occur. A. B. MANNING.

**Destructive hydrogenation of high-boiling hydrocarbons with the formation therefrom of hydrocarbons of lower b.p.** GAS LIGHT & COKE CO., R. H. GRIFFITH, and R. N. B. D. BRUCE (B.P. 348,803, 29.3.30).—High-boiling hydrocarbons rich in open-chain compounds are blended with high-boiling closed-chain compounds and the mixture is subjected to destructive hydrogenation under pressure and at raised temp. The

effect of such blending is to stabilise the reaction temp. The process is preferably carried out in the presence of a suitable catalyst, *e.g.*, Mo promoted by Si, B, Li, Ca, or P. A. B. MANNING.

**Manufacture of carburetted water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of J. A. PERRY (B.P. 348,501, 20.10.30. U.S., 20.11.29).—The carburetting oil is sprayed on to the fuel bed, the heating of the top of which is increased during both the "blow" and the "run," by providing a pier of refractory material extending up through the centre of the bed. With this device an oil producing a relatively large quantity of coke on gasification can be used. A. B. MANNING.

**Production of combustible gas mixtures which include constituents supplied from a distance.** A. C. BECKER and W. BERTELSMANN (B.P. 349,453, 19.2.30. Ger., 9.4.29).—Coal gas is treated by fractional compression and low-temp. cooling, and the hydrocarbon fraction formed as a by-product is passed under pressure into long-distance piping. At the point of distribution it is blended with gas mixtures, *e.g.*,  $\text{H}_2$ , water-gas, etc., substantially free from  $\text{CO}$ , in such proportions that, after treating with catalysts in order to remove any  $\text{CO}$ , the final gas mixture is substantially identical with ordinary town gas. II. S. GARLICK.

**Recovery of carbon disulphide [from gas mixtures].** IMPERIAL CHEM. INDUSTRIES, LTD., and J. McAULAY (B.P. 349,061, 19.12.29).—A mixture of (*e.g.*, equal vols. of) a stripping oil (creosote oil) and "heavy liquid paraffin" is used for absorbing  $\text{CS}_2$ , *e.g.*, from the exit gases of the process described in B.P. 331,734 (B., 1930, 862). The  $\text{CS}_2$  is recovered from the solution by steam-distillation. L. A. COLES.

**Treatment of distillate petroleum products.** STANDARD OIL CO. OF NEW YORK (B.P. 349,427, 21.11.29. U.S., 23.4.29).—Decolorisation of petroleum oils, *e.g.*, gasoline and kerosene, and the prevention of the formation of colour, odour, and gums are effected by the addition of > 0.1% of a 10% solution of a polyhydroxybenzene compound, *e.g.*, pyrogallol, in an oil-miscible solvent, *e.g.*, an aliphatic alcohol. [Stat. ref.]

H. S. GARLICK.  
**Heat-treatment of gasoline.** (SIR) H. W. A. DETERDING (B.P. 349,812, 27.6.30. Holl., 16.8.29).—A gasoline fraction obtained by straight distillation without substantial cracking is separated into 2 fractions and the heavier fraction is subjected to heat treatment under cracking conditions, preferably under pressure, and with or without a catalyst. The treated fraction is united with the lighter fraction or some corresponding fraction from another source. H. S. GARLICK.

**Production of motor driving spirits.** W. W. TRIGGS. From GES. F. TEERVERWERTUNG M.B.H. (B.P. 349,655, 6.3.30).—Benzines from the low-temp. distillation of coal etc. are hydrogenated at 300–400°/100–200 atm. in the presence of an active C catalyst, under such conditions that min. cracking occurs. [Stat. ref.]

H. S. GARLICK.  
**Separation of olefines by distillation.** N.V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of R. M. DEANESLY (B.P. 348,817, 5.4.30. U.S., 5.6.29).—Mixtures of olefines and paraffins, the components of which



have little difference in v.p., *e.g.*, mixtures of butane and butylene, are separated by fractional distillation under suitable pressure in the presence of liquid anhyd.  $\text{NH}_3$  or  $\text{NH}_2\text{Me}$ . By suitably controlling the distillation the bulk of the paraffin appears in the distillate, whilst the olefine remains in the residue. A. B. MANNING.

**Burners for pulverised fuel.** H. WADE. From BÜTTNER-WERKE A.-G. (B.P. 350,051, 13.3.30).

**Oxy-acetylene and similar heating burners.** ALLEN-LIVERSIDGE, LTD., and A. STEPHENSON (B.P. 349,433, 26.11.29).

**[Feeding device for] acetylene generators.** C. S. MILNE (B.P. 250,166, 30.4.30).

**Coke-oven doors.** R. WILHELM (R. WILHELM, MASCHINENFABR.) (B.P. 350,368, 3.11.30. Ger., 2.11.29).

**Doors for coke ovens or the like.** SOC. GÉN. DE FOURN. À COKE SYSTÈMES LECOCQ (B.P. 350,340, 9.9.30. Belg., 27.9.29). H. KOPPERS A.-G. (B.P. 349,623, 1.3.30. Ger., 2.3.29).

**Doors of carbonising chambers, such as coke ovens and the like.** WOODALL-DUCKHAM (1920), LTD., and A. T. KENT (B.P. 349,737, 24.4.30).

**Filters [for oil]. Settling apparatus [for  $\text{NH}_4$  salts].—See I.  $\text{H}_2\text{SO}_4$  from  $\text{NH}_3$  saturators. Bicarbonates.—See VII. Inhibitor for protecting metals.—See X. Firedamp-proof lamp.—See XI. Rubber compounding.—See XIV. Artificial turf.—See XVI. Testing for CO in air. Disinfectant.—See XXIII.**

### III.—ORGANIC INTERMEDIATES.

**Determination of phenols. [Phenol from] ammoniacal liquor. Phenols in coal tar.**—See II. Abs. EtOH.—See XVIII. **Detection of chloramine-T. Test for  $\text{CH}_2\text{O}$  in milk.**—See XIX.

#### PATENTS.

**Hydration of acetylene to acetaldehyde.** R. RILEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,883, 31.1.30).—In order to maintain the activity of the  $\text{HgSO}_4\text{--Fe}_2(\text{SO}_4)_3$  catalyst, part (*e.g.*,  $\frac{1}{3}$ ) of the liquor is withdrawn (preferably continuously) and  $\text{FeSO}_4$  oxidised with  $\text{O}_2$  gas before returning to the circuit. The concentration of  $\text{Fe}_2(\text{SO}_4)_3$  may thus be maintained sufficiently high to avoid reduction of  $\text{HgSO}_4$  to Hg. C. HOLLINS.

**Manufacture of acetic acid from acetaldehyde.** HOLZVERKÖHLUNGS-IND. A.-G. (B.P. 347,323, 3.2.30. Ger., 26.2.29).— $\text{MeCHO}$  and at least twice the theoretical quantity of  $\text{O}_2$  (with or without  $\text{N}_2$ ) are introduced into a mixing vessel at  $60\text{--}75^\circ$ , whereby dangerous concentrations of peracetic acid are avoided. The waste gases are washed successively with a portion of the liquid reaction product, with reaction product freed from  $\text{MeCHO}$ , and with  $\text{H}_2\text{O}$ . The yield of 96% AcOH is 98% on the unrecovered aldehyde. C. HOLLINS.

**Manufacture of alkoxyaldehydes.** DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 347,943 and Addn. B.P. 348,001, [A] 12.6.30, [B] 15.9.30. Ger., [A] 20.7.29, [B] 25.11.29).—(A) The alkoxyacetals

formed by addition of 3 mols. of an alcohol to an unsaturated aldehyde are partly hydrolysed by acid ( $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ , alkyl- $\text{HSO}_4$ ,  $\text{H}_3\text{PO}_4$ , oxalic acid, toluene-sulphonic acid) at  $20\text{--}25^\circ$ , with production of alkoxyaldehydes. The formation and partial hydrolysis may be effected without isolation.  $\beta$ -Ethoxybutaldehyde, b.p.  $137\text{--}138^\circ/720$  mm. or  $40^\circ/12$  mm., and the  $\beta$ -methoxy-compound are described. (B) The alkoxyaldehyde is separated rapidly and continuously by distillation as formed. C. HOLLINS.

**Purification of methanol [methyl alcohol] and other alcohols of b.p. below  $100^\circ$ .** IMPERIAL CHEM. INDUSTRIES, LTD., and J. W. ARMIT (B.P. 346,658, 13.12.29).—The alcohol is treated with halogen equiv. to the unsaturated compounds present, distilled through hot aq. NaOH to remove halogen acid, and fractionated. C. HOLLINS.

**Manufacture of polymerised hydrocarbons and application thereof [as insulating materials].** STANDARD TELEPHONES & CABLES, LTD., and W. E. HUGH (B.P. 345,939, 16.12.29).—A mixture of a styrene and a diolefine with conjugated double linkings is polymerised, *e.g.*, by heating at  $100^\circ$ , with or without catalyst, antioxidant, swelling agent, rubber, wax, etc.; 60% of styrene and 40% of  $\beta\gamma$ -dimethylbutadiene, with 5–15% of mineral wax, are polymerised in an autoclave to give an insulating material for cables. C. HOLLINS.

**Manufacture of acrolein [acraldehyde].** SCHERING-KAHLBAUM A.-G. (B.P. 346,221, 10.6.30. Ger., 15.6.29).—Glycerol vapour is led over a salt of a tribasic (or higher) acid (Li, Cu, Bi, Ag, or Fe phosphates, Mg borate, etc.), preferably on a carrier (pumice), at  $400\text{--}500^\circ$ . C. HOLLINS.

**Manufacture of odoriferous substances [cyclic acetals].** I. G. FARBERIND. A.-G. (B.P. 346,115, 28.2.30. Ger., 28.2.29).—The cyclic acetals obtained by condensing a dihydric alcohol with a phenylated aliphatic aldehyde have value in perfumery. Examples are: phenylacetaldehyde with glycol (product has b.p.  $115\text{--}120^\circ/12$  mm. and rose odour), with propylene glycol (b.p.  $100^\circ/5$  mm., rose),  $\alpha\beta$ -butylene glycol (b.p.  $107\text{--}110^\circ$ , hyacinth),  $\alpha\gamma$ -butylene glycol (b.p.  $133\text{--}135^\circ/14$  mm., hyacinth),  $\beta\delta$ -isohexylene glycol (b.p.  $110\text{--}115^\circ/5$  mm., mignonette); hydratropaldehyde with glycol (b.p.  $106\text{--}108^\circ/5$  mm., earth and mushroom), with  $\beta\delta$ -isohexylene glycol (b.p.  $115\text{--}120^\circ/5$  mm., herbaceous and mignonette);  $\beta$ -phenyl-*n*-propaldehyde with glycol (b.p.  $115\text{--}120^\circ/5\text{--}6$  mm., flowery and fruity),  $\beta\delta$ -isohexylene glycol (b.p.  $130^\circ/5$  mm., flowery and fruity); cinnamaldehyde with glycol (b.p.  $140\text{--}145^\circ/8$  mm., cinnamon), or  $\beta\delta$ -isohexylene glycol (b.p.  $155\text{--}160^\circ/5$  mm., cinnamic). C. HOLLINS.

**Treatment [separation] of mixtures of amines.** H. S. ADAMS and L. MEUSER, ASSRS. to NAUGATUCK CHEM. CO. (U.S.P. 1,782,112 18.11.30. Appl., 4.8.25).— $\text{CS}_2$  is added to the mixed amines and the whole oxidised with acid  $\text{H}_2\text{O}_2$ ; primary amine (preferably present only in traces) is converted into carbimide, secondary into thiuram disulphide, and tertiary is obtained as salt. The process is applied, *e.g.*, to mixed methylamines and to the by-product from the manufacture of triethyltrimethylenetriamine. C. HOLLINS.

**Manufacture of tetra-alkylated thiuram disulphides.** H. S. ADAMS and L. MEUSER, Assrs. to NAUGATUCK CHEM. CO. (U.S.P. 1,782,111, 18.11.30. Appl., 4.8.25).—In the oxidation of dialkylamine dialkyldithiocarbamates with  $\text{H}_2\text{O}_2$ ,  $\text{CS}_2$  equiv. to the amine which would be liberated is added so that all the amine is converted into thiuram disulphide. C. HOLLINS.

**Manufacture of pyridine derivatives.** E. KOENIGS and H. GREINER (B.P. 346,246, 23.7.30. Ger., 23.7.29).—Pyridine is converted by  $\text{SOCl}_2$  in 5 hr. at  $100^\circ$  or in 2–3 days at  $15^\circ$  into *N*-4-pyridylpyridinium chloride hydrochloride,  $\text{C}_5\text{H}_5\text{N}(\text{Cl})\cdot\text{C}_5\text{H}_4\text{N}\cdot\text{HCl}$ , which is dimorphic, m.p.  $171\text{--}172^\circ$  (stable) and  $151\text{--}152^\circ$ . Fission by KOH at  $200^\circ$  or conc. aq.  $\text{NH}_3$  at  $150\text{--}160^\circ$  gives 4-aminopyridine and glutacondialdehyde (which is resinified); aniline gives aminopyridine and glutacondialdehyde dianil. C. HOLLINS.

**Manufacture of piperidine.** SCHERING-KAHLBAUM A.-G. (B.P. 346,222, 10.6.30. Ger., 21.6.29).—The catalytic hydrogenation of pyridine, using active Ni on  $\text{SiO}_2$  gel, is effected at or below  $160^\circ/100\text{--}150$  atm. The H may be supplied by tetrahydronaphthalene etc. C. HOLLINS.

**Manufacture of substitution products of carbazolesulphonic acids.** I. G. FARBENIND. A.-G. (B.P. 347,193, 23.1.30. Ger., 23.1.29).—The  $\text{NH}_2$  group in aminocarbazolesulphonic acids is diazotised and exchanged for other groups. 4:2:6:8- and 2:4:6:8-Hydroxycarbazolettrisulphonic acids, 6-iodo-4-hydroxycarbazole-2:8-disulphonic acid, 2:2':6:6':8:8'-hexasulpho-4:4'-dicarbazolyl disulphide, 4-chloro-, 4-bromo-, 4-iodo-, and 4-cyano-carbazole-2:6:8-trisulphonic acids, are described. C. HOLLINS.

**Decreasing the inflammability of organic nitro-compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 347,101, 17.12.29).—An acid amide, e.g., urea, is incorporated with Na *p*-nitrophenoxide, 2:4-dinitrophenoxide, etc., especially for use in wood preservation. 1–2 pts. of urea are added to 1 pt. of nitrophenol etc. C. HOLLINS.

**Manufacture of substituted phenolcarboxylic acids.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 347,407, 12.3.30).—*o*-4-Xylenol or 2-chloro-*p*-cresol are carboxylated by Kolbe's method, giving acids of m.p.  $200^\circ$  and  $210\text{--}212^\circ$ , respectively. C. HOLLINS.

**Manufacture of condensation products containing halogen [chloromethyl compounds].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 347,887, 3.5.30).—Aq.  $\text{CH}_2\text{O}$  saturated with HCl reacts with PhOH to give at  $40\text{--}45^\circ$  a viscous resin, or at  $5\text{--}10^\circ$  6:8-bischloromethylbenz-1:3-dioxan,  $\text{CH}_2\text{C}(\text{CH}_2\text{Cl})\cdot\text{C}\cdot\text{O}\text{---}\text{CH}$   
 $\text{C}(\text{CH}_2\text{Cl})\cdot\text{CH}\cdot\text{C}\cdot\text{CH}_2\cdot\text{O}$ , m.p.  $117^\circ$ ; *p*-cresol at  $20^\circ$  gives 3:5-bischloromethyl-*p*-cresol, m.p.  $86^\circ$ , technical cresols at  $40^\circ$  a resin. C. HOLLINS.

**Manufacture of aromatic condensation products [chloromethyl derivatives of aryl alkyl ethers or thioethers].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 347,892, 7.5.30).—Aryl alkyl ethers or thioethers (the aryl being Ph, alkylated or halogenated

Ph, or nitrophenyl) are treated with  $\text{CH}_2\text{O}$  and HCl so as to introduce 1 or 2 chloromethyl groups in *p*- and/or *o*-positions to the ether group. 2:4-Bischloromethylanisole, m.p.  $65^\circ$ , 3-chloro-2(?):4-bischloromethylanisole, m.p.  $47\text{--}48^\circ$ , b.p.  $172\text{--}174^\circ/10$  mm. (from *m*-chloroanisole), 2-methoxy-5-methylbenzyl chloride, m.p.  $9^\circ$ , b.p.  $118\text{--}120^\circ/15$  mm. (from *p*-tolyl methyl ether), 2:5-bischloromethylquinol dimethyl ether, m.p.  $165^\circ$ , 4(?)methoxy-3-nitrobenzyl chloride, m.p.  $87^\circ$ , b.p.  $175^\circ/5$  mm. (from *o*-nitroanisole), 2-methylthiol-5-methylbenzyl chloride, m.p.  $31^\circ$ , b.p.  $132\text{--}134^\circ$  (from *p*-tolyl methyl sulphide), and 2:4-bischloromethylthiophenol methyl ether, m.p.  $46^\circ$ , are described. C. HOLLINS.

**Manufacture of aromatic methyleneamino compounds [4-hydroxy-3-carboxybenzylated arylamines].** M. KAHN, W. SCHIESS, and L. ZEH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,781,982, 18.11.30. Appl., 23.6.27. Ger., 19.8.26).—The chloromethyl derivatives obtained from salicylic or cresotic acids by action of HCl and  $\text{CH}_2\text{O}$  are condensed with arylamines, pyridines, etc. to give coupling components for azo dyes. Products from 5-chloromethylsalicylic acid and metanilic acid, from 5-chloromethyl-*o*-cresotic acid and  $\gamma$ -acid (specially claimed) or methylaniline or pyridine (a quaternary salt) are described. C. HOLLINS.

**Manufacture of monoacyl[*m*- and *p*-]diamines of the benzene series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 347,774, 7.2.30).—Arylsulphonyl derivatives of *m*- and *p*-nitroanilines (which may carry halogen, alkyl, or alkoxyl as substituents) are reduced and acylated (the reduction products being sol. in alkali), and the arylsulphonyl group is removed by hydrolysis with  $\text{H}_2\text{SO}_4$ . The following are described: 6-benzamidocresidine, m.p.  $185^\circ$  (benzenesulphonyl derivative, m.p.  $168\text{--}171^\circ$ ), from 6-nitro-*N*-benzenesulphonylcresidine, m.p.  $207\text{--}208^\circ$ ; corresponding ethyl ether, m.p.  $158^\circ$  (benzenesulphonyl derivative, m.p.  $148^\circ$ ), Bu ether, m.p.  $120^\circ$  (benzenesulphonyl derivative, m.p.  $134^\circ$ ); 6-*op*-dichlorobenzamidocresidine, m.p.  $160^\circ$  (benzenesulphonyl derivative, m.p.  $203^\circ$ ); 6-anisamidocresidine, m.p.  $212^\circ$ ; 6-phenylacetamidocresidine, m.p.  $158^\circ$  (benzenesulphonyl derivative, m.p.  $183^\circ$ ); 6-*o*-chlorophenoxyacetamidocresidine, m.p.  $135^\circ$  (benzenesulphonyl derivative, m.p.  $196^\circ$ ); 6-cinnamamidocresidine (benzenesulphonyl derivative, m.p.  $198^\circ$ ); 4-chloro-5-benzamido-*o*-anisidine, m.p.  $174^\circ$  (toluene-*p*-sulphonyl derivative, m.p.  $175^\circ$ ); 5-benzamido-*o*-toluidine, m.p.  $154^\circ$  (toluene-*p*-sulphonyl derivative, m.p.  $195^\circ$ ); 6-benzamido-*m*-4-xylylidine, m.p.  $176^\circ$ ; 4-benzamido-*o*-anisidine, m.p.  $139^\circ$  (toluene-*p*-sulphonyl derivative, m.p.  $183^\circ$ ); 4-benzamido-2:5-dimethoxyaniline, m.p.  $168^\circ$  (benzenesulphonyl derivative, m.p.  $175^\circ$ ); diethoxy-compound, m.p.  $101^\circ$ ; 2-methoxy-5-ethoxy-compound, m.p.  $121^\circ$ . C. HOLLINS.

**Manufacture of halogenated aminoaryl[ene]thiazole compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 347,141, 22.1.30. Cf. B.P. 345,735; B., 1931, 667).—A 2-aminoarylenethiazole is chlorinated or brominated with substantially the theoretical amount of halogen. The thiazole may be produced by chlorinating an arylthiocarbamide, and brominated without

isolation. The production of 6-chloro- (m.p. 198—200°), 6-bromo- (m.p. 210—212°), 5 : 6-dichloro-4-methyl- (m.p. 250—255°), 6-chloro-4-methyl-, 6-bromo-4-methyl- (m.p. 215—218°), 5-bromo-6-ethoxy- (m.p. 200—205°) derivatives of 2-aminobenzthiazole, 5-chloro-2-amino- $\beta$ -naphththiazole, m.p. 247°, and 6-bromo-2-*p*-bromo-anilinobenzthiazole, m.p. 215—217°, is described.

C. HOLLINS.

**Oxidation of organic substances [with selenium dioxide etc.].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 347,743, 31.1.30).—Aromatic compounds containing Me or CH<sub>3</sub> groups are oxidised to aldehydes, ketones, or acids by heating with O compounds of Se, e.g., SeO<sub>2</sub> or H<sub>2</sub>SeO<sub>4</sub>. Ring systems, e.g., benzanthrone, susceptible to other oxidants are unattacked. 2-Methylbenzanthrone with SeO<sub>2</sub> in H<sub>2</sub>O at 230—240°, or with cone. H<sub>2</sub>SeO<sub>4</sub> in boiling AcOH, gives 2-aldehydobenzanthrone; with SeO<sub>2</sub> in boiling PhNO<sub>2</sub> the 2-carboxylic acid is formed. The methyl-naphthanthraquinone from 1-methylnaphthalene and phthalic anhydride gives with aq. SeO<sub>2</sub> at 240° either the acid or the aldehyde. 4-Benzylbenzanthrone similarly yields 4-benzoylbenzanthrone, and 1 : 5-dibenzoyl-2 : 6-dimethylnaphthalene is oxidised to the 2 : 6-dicarboxylic acid.

C. HOLLINS.

**Manufacture of chloro-derivatives of the anthraquinone-acridone series.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 347,095, 20.11.29).—1-Anilino-2-methyl- or *o*-toluidino-anthraquinone, which may contain Cl, is chlorinated in absence of acid-binders and H<sub>2</sub>O until a hexachlorophthaloylacridone is obtainable on hydrolysis. The hydrolysis product may be partly dehalogenated by reduction, e.g., by vatting and re-oxidising. The less sol. leuco-compounds may be separately oxidised. Rapid chlorination of 1-anilino-2-methylantraquinone at 170°, followed by hydrolysis, vatting at 55°, and re-oxidation leads to a pure red vat dye. [Stat. ref.]

C. HOLLINS.

**Hydrogenation of phenols etc.** Separation of olefines.—See II. Al salts.—See VII. Application of moulds. BuOH and COMe<sub>2</sub> by fermentation. Acids by fermentation.—See XVIII.

#### IV.—DYESTUFFS.

##### PATENTS.

**Manufacture of condensation products [acid wool dyes] of the benzanthrone series.** I. G. FARBERIND. A.-G. (B.P. 346,896, 7.2.30. Ger., 7.2.29. Addn. to B.P. 248,791; B., 1927, 550).—A 2-aryloxy-3-naphthoic acid, ester, or amide is converted by acid condensing agents (NaAlCl<sub>4</sub>) into benzanthrone derivatives. 5-Hydroxybenzanthrone-4-carboxylic acid, m.p. 285°, from 2-benzoyloxy-3-naphthoic acid or its Et ester, and 8-chloro-5-hydroxybenzanthrone-4-carboxylic acid, m.p. 250—252°, from the 2-*m*-chlorobenzoyloxy-compound, are chromable wool dyes.

C. HOLLINS.

**Preparation of [vat] dyes of the anthraquinone series.** IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and W. W. TATUM (B.P. 345,982, 30.9.29).—Aminoanthraquinones are condensed with halogenated pyrimidines, and, if desired, aroylated. Examples are : 2 : 4-dichloropyrimidine with 1 : 4-diaminoanthraquinone

(blue-violet; benzoylated, blue-red), 1-aminoanthraquinone (orange), 1 : 4-aminohydroxyanthraquinone (red-violet), and 1 : 5-diaminoanthraquinone (orange-red).

C. HOLLINS.

**Production of condensation products [vat dyes of the anthraquinone series] containing nitrogen.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 346,677, 8.10.29).—An *o*-aminoanthraquinonecarboxylic ester or amide is condensed with polynuclear (at least 4-ringed) carbocyclic or with heterocyclic compounds having reactive halogen etc., preferably in a solvent (C<sub>10</sub>H<sub>8</sub> or PhNO<sub>2</sub>) in presence of acid-binder and a Cu catalyst; or an *o*-halogenoanthraquinonecarboxylic ester or amide is condensed with a corresponding amine. The products are finally cyclised to acridones. Examples are : Me or Et 1-aminoanthraquinone-2-carboxylate with dibromo-1 : 2 : 6 : 7-dibenzpyrene-3 : 8-quinone (red-violet; acridonated, red-brown), with 8-chlorobenzanthrone (fused with KOEt, violet-grey to black), or with 10-chlorobenzanthrone (acridonated, brown); Et 1-chloroanthraquinone-2-carboxylate with amino-*allo*-*ms*-naphthadanthrone (acridonated, red-violet) or with amino-*ms*-benzodanthrone (acridonated, brown-green).

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone [carbazole-acridone] series.** I. G. FARBERIND. A.-G. (B.P. 348,849, 22.4.30. Ger., 22.4.29).—Anthraquinones, pyranthrones, dibenzanthrones, or other anthraquinone derivatives, carrying reactive halogen, are condensed with an NH<sub>2</sub> derivative of a carbazole of the anthraquinone series containing at least one acridone ring (see B.P. 323,543; B., 1930, 365). Dichloro-2-amino-3 : 4 : 6 : 7-dipthaloylcarbazole-8 : 9-acridone, obtained from 2 : 4 : 8-trichloro-6 : 7-phthaloylacridone by condensation with 1-amino-4-benzamidoanthraquinone, carbazolisation, and hydrolysis, is condensed with 1-chloroanthraquinone (grey vat dye) or 4-chloro-1-benzamidoanthraquinone (grey to black); dichloro-6 : 7-phthaloyl-3 : 4-(3'-amino-phthaloyl)carbazole-8 : 9-acridone, obtained similarly from 1-amino-5-benzamidoanthraquinone, is condensed with 5-chloro-1-benzamidoanthraquinone (red-grey), dibrominated pyranthrone (black-brown), or dibrominated anthanthrone (deep black).

C. HOLLINS.

**Manufacture of halogenoanthraquinone- $\beta$ (N)-benzacridones [halogenated 6 : 7-phthaloylacridones].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 347,722, 31.10.29).—6 : 7-Phthaloylacridones, which may already contain Cl or Br, are improved in strength and purity of shade by further chlorination or bromination in ClSO<sub>3</sub>H preferably in presence of Fe or S at 20—40°; ClSO<sub>3</sub>H itself may be used as chlorinating agent at 60° in presence of I. Amongst the examples are : 6 : 7-phthaloylacridone with Cl or Br and I at 60° for 2—3 hr. (powerful red vat dye); 2 : 4-dichloro-6 : 7-phthaloylacridone with Cl and Fe at 20—40° (yellowish-red); 6 : 7-phthaloyl-2-methylacridone with Cl and S (bluish-red); bisacridone from 1 : 5-dichloroanthraquinone and anthranilic acid, with Cl and FeCl<sub>3</sub> at 20° (violet-red). H<sub>2</sub>SO<sub>4</sub> or oleum may be used in place of ClSO<sub>3</sub>H.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone-acridone series.** From I. G. FARBERIND. A.-G. (B.P.

346,849, 23.10.29).—6 : 7-Phthaloylacridones and their halogen derivatives are treated with  $\text{PCl}_5$ ,  $\text{POCl}_3$ , etc., preferably in a solvent (trichlorobenzene), and then halogenated; if sol. products are obtained (e.g., at lower temp.) hydrogen halide is removed by means of  $\text{PhOH}$ ,  $\text{SO}_2$ , etc. Trichlorinated 6 : 7-phthaloylacridone with  $\text{PCl}_5$  in trichlorobenzene at  $200^\circ$  gives on chlorination at  $175^\circ$  a pure red vat dye. [Stat. ref.] C. HOLLINS.

**Manufacture of [mono]azo dyes [for wool, pigments, etc.].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 347,288, 27.1.30).—An arylamine, especially an *o*-aminophenol, is diazotised and coupled with an aminonaphtholsulphonamide. Examples are: aniline-*o*-sulphonic acid  $\rightarrow$  di(methylanilide) of acetyl-H-acid (red), or dianilide of H-acid (wine-red); *o*-aminophenol-4-sulphonic acid  $\rightarrow$  dianilide of H-acid (red-violet, green-blue on chroming); *m*-nitroaniline-6-sulphonic acid  $\rightarrow$  ethylanilide of  $\gamma$ -acid (brick-red); aniline-2 : 5-disulphonic acid  $\rightarrow$  methylanilide of  $\gamma$ -acid (red); 2-amino-4'-hydroxy-3'-carboxydiphenylsulphone-4-sulphonic acid  $\rightarrow$  methylanilide of  $\gamma$ -acid (blue-red, bluer on chroming). C. HOLLINS.

**Manufacture of azo dyes containing chromium.** I. G. FARBENIND. A.-G. (B.P. 347,290, 27.1.30. Addn. to B.P. 306,843; B., 1930, 811).—A pyrazolone derived from an aminoarylsulphonylsalicylic acid is treated with a Cr compound and then coupled with a diazotised arylamine other than an *o*-aminophenol. Examples are: 2-chloro-*p*-toluidine  $\rightarrow$  Cr compound of 1-[5-sulpho-2-(4'-hydroxy-3'-carboxyphenylsulphonyl)phenyl]-3-methyl-5-pyrazolone (greenish-yellow on wool); *o*-toluidine  $\rightarrow$  Cr compound of 1-[5-sulpho-2-(2'-hydroxy-3'-carboxy-5'-methylphenylsulphonyl)phenyl]-3-methyl-5-pyrazolone (yellow). C. HOLLINS.

**Manufacture of [chrome-yellow] azo dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,930, 25.9.29).—3-Methyl-5-pyrazolones derived from amino-salicylic acids are coupled with diazotised non-hydroxylated aminobenzenesulphonamides or aminobenzamides free from further sulphonic or carboxylic groups. Examples are: aniline-*m*-sulphonamide or *m*-aminobenzamide  $\rightarrow$  1-(2-hydroxy-5-sulpho-3-carboxyphenyl)-3-methyl-5-pyrazolone. [Stat. ref.] C. HOLLINS.

**Manufacture of [gallocyanine] dyes.** DURAND & HUGENIN A.-G. (B.P. 346,243, 19.7.30. Addn. to B.P. 301,329; B., 1930, 502).—The simple *m*-substituted gallocyanines used as starting materials in the process of the prior patent are condensed in acid ( $\text{HCl}$ ) with phenols, and, if desired, sulphonated, to give printing colours stable to discharge pastes. The gallocyanine from 6-nitrosodimethyl-*m*-toluidine and gallamide is condensed with resorcinol; that from 6-nitrosodimethyl-*m*-toluidine and gallic acid, with Schäffer acid. C. HOLLINS.

**Chlorinated anthraquinoneacridones.**—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Production of esparto fibre.** C. DE NEYMAN (Chim. et Ind., 1931, 25, 1078—1080).—The raw material is freed from encrusting substances by successive treatments with aq. suspensions of  $\text{CaO}$  either at room temp.

or at  $100^\circ$ . The amounts of  $\text{CaO}$  to be added in the first and succeeding baths and the duration of the treatments vary according to the temp. Either fresh or sea-water may be used, and if fuel is scarce, as in some tropical regions, an alternative maceration treatment with a more conc.  $\text{CaO}$  suspension is employed and the vats are heated by the sun.  $\text{Na}_2\text{CO}_3$  is not so efficient as  $\text{CaO}$ , but  $\text{NaOH}$  liquor is more satisfactory. Soda liquors such as are used in obtaining cellulose in papermaking have also been tried, both alone and with the addition of  $\text{CaO}$  after each boil. The latter method gives a product of better quality. Suitable apparatus for the boiling is described, and when water is scarce countercurrent washing should be employed. Beating or other mechanical treatment is necessary in order to free the fibres. B. P. RIDGE.

**Determination of the moisture content of fabrics by cobalt chloride test-paper.** K. YAMADA, H. OTSUKA, T. NAKAMURA, and F. TATEBE (J. Soc. Chem. Ind., Japan, 1931, 34, 135—136 B).—The duration of the colour change of a  $\text{CoCl}_2$  test-paper, at const.  $\text{H}_2\text{O}$  v.p., is proportional to the quantity of the salt present. Since the velocity of evaporation of moisture from a fabric is proportional to the  $\text{H}_2\text{O}$  content, the duration of the colour change may be employed to determine the latter; the error rarely exceeds 3%. H. F. GILLBE.

**Manufacture of sulphite pulp from young wood.** C. G. SCHWALBE and K. BERNDT (Papier-Fabr., 1931, 29, 145—148).—Sulphite pulps have been prepared from young fir trees (6 yrs.) of 4—5 cm. diam., cut during the "thinning" of forests. Digestion at  $70$ — $140^\circ$  for 18 hr. with liquor containing 4.2%  $\text{SO}_2$  and 0.94%  $\text{CaO}$  yields 45.9% of a pulp containing 89.6%  $\alpha$ -cellulose and 0.36% fats and resins, with a Cu number of 2.86. 2% of active Cl is required for bleaching. Beating is slow, but sheets of high strength are produced. T. T. POTTS.

**Cooking sulphite pulp with strong cooking acids.** E. HÄGGLUND (Zellstoff u. Papier, 1931, 11, 338—341).—Yields 5—10% higher may be obtained by using Ca or Mg bisulphite liquors containing 8%  $\text{SO}_2$  instead of the usual 4.3% and concentrations of  $\text{CaO}$  or  $\text{MgO}$  above 0.9%. The  $[\text{HSO}_3^-]$  has a great effect on the quality of the pulp and on its lignin content, and for pulps of the same yield the lignin content is smaller, i.e., the purer is the pulp, the higher is this concentration. Dissolution of lignin is more complete with  $\text{Ca}(\text{HSO}_3)_2$  than with  $\text{Mg}(\text{HSO}_3)_2$ , and white pulps cannot be obtained if the base content of the liquor is too low. For solutions of equivalent base concentration better cooking is effected, but poorer yields of sugar are given, by the Ca than by the Mg salt, but since the latter is much more sol. than the former at the temp. of decomp., this difference may be evened out by using more conc.  $\text{Mg}(\text{HSO}_3)_2$  solutions. B. P. RIDGE.

**Measurement of bleachability and cooking degree of sulphite pulp.** VON POSSANNER and R. A. KRAUSS (Papier-Fabr., 1931, 29, 317—322, 335—339, 348—357).—The principal known methods of testing bleachability of pulps and of determining cellulose and inercrants are considered in detail, comparative analyses being given. Tests on 35 pulps show the Willstätter

method for determining lignin to give consistently lower results than the Becker method. Lignin determinations are considered too lengthy for control purposes. Indirect methods depending on Cl consumption are critically examined, the methods of Sieber and of Roe being found the most reliable. The  $\text{KMnO}_4$  absorption method of Joachim is found to be simple and reliable,  $\text{KMnO}_4$  consumption being proportional to lignin content when exceeding 3%. Methods employing malachite-green or phloroglucinol are of no value. An extensive bibliography is given.

T. T. POTTS.

**Developments in the production of yellow and bleached straw pulp.** R. RUNKEL (Zellstoff u. Papier, 1931, 11, 342—345).—Use of dil. (1.5%) NaOH at 25° for 72 hr. gives a higher yield of pulp and a better removal of lignin than is obtained with CaO, soda ash and sulphite, or more conc. NaOH at 100° or 150°. Milder and continuous maceration processes are rendered possible by use of the new bar mill instead of the edge mill, and bleaching is then carried out by a combined Cl-HOCl process. Economical working depends on the demand for alkali and the cost of Cl, but even if the cost of chemicals is higher than for the older processes the greatly increased yield effects an economy.

B. P. RIDGE.

**Relation between chlorine consumption and copper number [of wood pulps].** E. FABRIZI (Papier-Fabr., 1931, 29, 361—367).—During the bleaching of wood pulp, Cu number decreases with the initially rapid consumption of Cl, rising then with further Cl consumption after a point which differs for varying types of pulp, the ratio Cu/Cl becoming const. This relationship may be used to determine the optimum point on a bleach consumption/time curve.

T. T. POTTS.

**Determination of  $\alpha$ -cellulose content and copper number of paper.** J. O. BURTON and R. H. RASCH (Bur. Stand. J. Res., 1931, 6, 603—619).—Determination of  $\alpha$ -cellulose (portion insol. in NaOH of mercerising concentration under standard conditions) by modification of Jentgen's method (B., 1911, 125), and of the Cu number (no. of g. of Cu in  $\text{Cu}_2\text{O}$  precipitated from alkaline solution of  $\text{Cu}(\text{OH})_2$  under specified conditions by 100 g. of material) by modification of Braidy's method (Rev. gén. Mat. Col., 1921, 25, 35), may be applied to paper testing providing the sample is reduced to cotton-like form by mechanical grinding (process described), which avoids heating or bruising the fibre, and providing allowance is made for the amount of sizing and loading materials present. Various factors affecting the accuracy of the results have been investigated, and the processes described in detail give results reproducible to 0.3 and 1.0%, respectively.

J. W. BAKER.

**Bursting-strength tests in evaluation of paper and corrugated board.** G. CLEMENS (Papier-Fabr., 1931, 29, 97—101, 129—135, 148—155, 167—171, 181—187, 198—203).—The investigations described arise out of the necessity for standardisation of corrugated board containers. The Schopper-Dalén bursting tester is critically examined. Dalén's assumption that paper forms a truly spherical surface in the bursting tester is found to be correct.

T. T. POTTS.

**Durability of paper.** KORN (Papier-Fabr., 1931, 29, 155—156).—A review of recent British and American official publications regarding durability standards for paper.

T. T. POTTS.

**Recovery of cotton from tyre scrap.**—See XIV.  
**Fibre in podophyllum rhizome.**—See XX.

## PATENTS.

**Production of filaments, yarns, threads, etc. from [waste] natural silk.** BRIT. CELANESE, LTD. (B.P. 349,220, 4.4.30. U.S., 19.4.29).—The silk, preferably degummed, is dissolved in acid, e.g.,  $\text{H}_2\text{SO}_4$  of 25—75% concentration, syrupy  $\text{H}_3\text{PO}_4$ , conc. HCl, etc., at from  $-10^\circ$  to  $10^\circ$  and spun into a large vol. of an alcohol either alone or in admixture with  $\text{H}_2\text{O}$ , NaOH, AcONa, or the like also at about  $0^\circ$ .

D. J. NORMAN.

**Manufacture of cellulose esters.** SOC. CHEM. IND. IN BASLE (B.P. 349,322, 2.6.30. Switz., 1.6.29).—Ripened or unripened cellulose xanthate is treated for a short time at raised temp. with an org. acid anhydride optionally in the presence of diluents and/or catalysts. The esterification is easily controlled and the resulting products are free from S and show the dyeing properties of ordinary cellulose esters. E.g., cotton fabric may be printed with viscose solution, dried, and treated for  $\frac{1}{2}$ —2 hr. at  $140^\circ$  with  $\text{Ac}_2\text{O}$  to give a fabric suitable for effect dyeing. Alternatively, cellulose xanthate filaments may be spun from viscose solution, e.g., using as coagulant a solution of an alkali salt acidified with AcOH, and subsequently esterified in filament form in the absence of solvents for cellulose xanthate or other esters.

D. J. NORMAN.

**Manufacture of esters of cellulose or of its transformation products or of other carbohydrates.** 1. G. FARBEIND. A.-G. (B.P. 348,960, 21.7.30. Ger., 19.7.29. Addn. to B.P. 301,036; B., 1930, 504).—Esterification is carried out in the presence of liquid  $\text{SO}_2$  under increased pressure produced by means of  $\text{N}_2$  or compressed air.

F. R. ENNOS.

**Manufacture of cellulose acetates.** CELLULOSE ACETATE SILK CO., LTD., P. C. CHAUMETON, and G. II. WAKEFIELD (B.P. 348,292, 19.3.30).—Cellulose material is pretreated below  $20^\circ$  with a lower fatty acid (AcOH) and a relatively small quantity of  $\text{SO}_2$  (not more than 5% of the total amount of the acetylating bath), with addition of conc.  $\text{H}_2\text{SO}_4$  and/or AcOH, if desired, and is afterwards acetylated in the usual manner. Alternatively, the  $\text{SO}_2$  may be added to the complete acetylating bath before addition of the cellulosic material; the products are acetates of relatively high viscosity.

F. R. ENNOS.

**Manufacture of artificial fibres and films from cellulose esters.** 1. G. FARBEIND. A.-G. (B.P. 348,931 and Addn. B.P. 348,959, [A] 23.6.30, [B] 18.7.30. Ger., [A] 11.7.29, [B] 18.7.29).—(A) The reaction mixture containing cellulose acetate dissolved in liquid  $\text{SO}_2$  obtained by the process of B.P. 301,036 (B., 1930, 504) is dry-spun (e.g., into air) or wet-spun (e.g., into  $\text{H}_2\text{O}$  or a salt solution) without preliminary isolation of the cellulose ester. In the dry-spinning process the catalyst and any free

AcOH present in the reaction mixture are just neutralised by addition of conc.  $\text{Na}_2\text{CO}_3$  solution, the  $\text{H}_2\text{O}$  and salts thus formed being removed by freezing and filtration. This procedure, however, is unnecessary in wet spinning. (B) The reaction mixture containing, *e.g.*, liquid  $\text{SO}_2$  55%, cellulose acetate 22%, AcOH 16%,  $\text{H}_2\text{O}$  5–6%, and catalyst 1–2%, is dry-spun into air and the resulting somewhat swollen filament, the composition of which is approx. cellulose acetate 50%, AcOH 33%,  $\text{H}_2\text{O}$  12%, and catalyst 5%, is completely coagulated in, *e.g.*,  $\text{H}_2\text{O}$  or a salt solution at 20–70°.

D. J. NORMAN.

#### Manufacture of artificial silk from viscose.

BREDA-VISADA, LTD., and R. O. JONES (B.P. 348,743, 27.2.30).—Viscose silk of dull lustre is obtained by emulsifying with the viscose solution, before spinning, up to 1% of a non-glyceride ester of animal or vegetable origin which is resistant to hydrolysis by alkalis, *e.g.*, sperm oil, lanoline, carnauba wax, beeswax, etc.

D. J. NORMAN.

#### Manufacture of artificial silk.

S. WILD (B.P. 348,168, 3.1.30. Switz., 9.1.29).—The spinning solution is maintained at a const. temp. in the region of the nozzle by means of a surrounding jacket or heat exchanger, into which liquid is introduced at an initial temp. not more than 2° below that at which the spinning solution is to be kept. After leaving the heat exchanger, the liquid is cooled below and subsequently heated to the required initial temp. for re-entry.

F. R. ENNOS.

#### Manufacture of artificial silk and the like.

BORZYKOWSKI (B.P. 348,644, 11.2.30. Ger., 1.3.29).—While being brought from the spinning bobbin or spun cake to a wound form suitable for the textile industry, the filaments are cleaned, preferably by hairy material such as skin, shaved by means of an adjustable slit, and oiled or finished.

F. R. ENNOS.

#### Spinning of acid solutions of silk fibroin.

I. G. FARBENIND A.-G. (B.P. 349,387, 20.8.30. Ger., 31.8.29).—Stronger and softer filaments are obtained by the process of B.P. 339,089 (B., 1931, 153) if two coagulating baths are used. The first bath contains a conc. aq. solution of alkali salts of mineral acids (*e.g.*, mixed KCl and NaCl) with addition of 16–33 vol.-% of a saturated solution of alkali formate or acetate and about 15 g. of  $\text{CH}_2\text{O}$  per litre. The second consists of a saturated solution of alkali (or  $\text{NH}_4$ ) formate or acetate with addition of about 12.5% of Na lactate solution (64%). The coagulated filament is then passed through air for several metres without tension and stretched to 5–8 times its length. The finished silk is washed on the spool, soaped, and dried. The addition of 0.02–0.1% of an essential oil, *e.g.*, turpentine, to the spinning solution minimises the tendency of the spun filaments to stick together.

D. J. NORMAN.

#### Production of artificial filaments, threads, ribbons, etc.

H. DREYFUS (B.P. 346,673, 10.10.29).—High-melting org. compounds, preferably  $\text{H}_2\text{O}$ -insol. and in fine division (0.0001–0.0005 mm.), are incorporated in the spinning solution to give artificial (especially acetate) silks with subdued lustre. Particularly suitable are diacetyl-benzidine or -tolidine, dibenzoyl-

benzidine, di- $\beta$ -naphthyloxamide, diphenyloxamide, succinic  $\alpha$ -naphthylamide, thiocarbonylbenzidine, carbonylbenzidine, 4:4'-dicarbamidodiphenyl, 4:4'-bis-phenylcarbamidodiphenyl, etc.

C. HOLLINS.

#### Direct manufacture and employment of artificial filaments.

B. BORZYKOWSKI (B.P. 348,759, 10.3.30. Ger., 24.6.29. Addn. to B.P. 347,241; B., 1931, 626).—The washed and, if desired, after-treated cake is placed with its lower edge on a plane soft-faced support; a tensioning device, inserted in the cake so as to project above its upper edge, is fixed to the plane support so that loosening of the thread layers on unwinding or lateral displacement of the cake is prevented.

F. R. ENNOS.

#### Treatment of cellulosic materials.

DISTILLERS CO., LTD., W. P. JOSHUA, and P. EAGLESFIELD (B.P. 348,740, 26.2.30).—The liquor resulting from the acid hydrolysis of cellulose is passed while still hot and under pressure (12–15 atm.) through a coil where it is heated at 160–180° for 6–10 min. to convert unfermentable polysaccharides into monoses. The hot liquor is then passed through a defecating tower containing, *e.g.*, Lahn phosphate or bone charcoal to remove fermentation inhibitors and replace the acid used in the hydrolysis by  $\text{H}_3\text{PO}_4$ . The liquor must at all stages show an acid reaction and, if the defecating material is of an alkaline nature, this must be washed with acid before use.

D. J. NORMAN.

#### Saccharification of carbohydrates.

H. DREYFUS (B.P. 349,032, 19.2.30).—Cellulosic material, *e.g.*, sawdust, is impregnated with 10–20 times its wt. of 0.25–1% (preferably 0.25–0.5%)  $\text{H}_2\text{SO}_4$ , pressed until it retains <1% of  $\text{H}_2\text{SO}_4$  on the wt. of carbohydrates present, and heated under a pressure of 4–9 (preferably 6–7) atm. at 145–155° for 8–15 hr.

D. J. NORMAN.

**Filters.**—See I. Diazotisable fibres.—See VI. Non-splintering glass.—See VIII. Roofing elements. **Floor covering.**—See IX. **Insulation for cables.**—See XI. **Adhesive plaster.**—See XX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### Theories of lustre [in fabrics].

J. M. PRESTON (J. Soc. Dyers and Col., 1931, 47, 136–143).—A review of theories of lustre and methods of measurement, with particular reference to the lustre of fabrics of different constructions and colour. The dyeing of cellulose acetate silk, dulled by a treatment with boiling soap, to a deep shade of black restores its lustre; this does not occur when dulling is obtained by abrasion of the surface of the silk fibres. Lustre is associated with the longitudinal appearance of a fibre rather than with its cross-section, although the latter has a secondary effect. Reflected light from dull, non-desulphurised viscose silk has a distinct bluish tinge due to greater diffusion by refraction of blue than of red light. A method has been developed for recording simultaneously on a photographic film mounted cylindrically the light reflexions in different directions from fabrics.

A. J. HALL.

**Laundering of fabrics.** J. T. HOLDEN (J. Soc. Dyers and Col., 1931, 47, 143–144).—Fabrics and garments are sorted according to size and colour and

then washed in three stages (low-temp. washing, boiling, and rinsing), of which full details are given. Lack of balance in the construction of a fabric gives rise to creases during laundering, and white lines appear if the fabric is poorly penetrated by dyes. "Marking-off" of vat dyes (particularly the yellows) is a common fault and is probably due to reduction. A. J. HALL.

**Bleaching of pulps.**—See V.

#### PATENTS.

**Liquids or plastic preparations for textiles and leather.** H. T. BÖHME A.-G. (B.P. 349,586, 9.12.29. Ger., 29.12.28).—Aq. dispersions of the alkyl or cyclo-alkyl esters of the aromatic sulphonic acids derived from monohydric alcohols are used as wetting or dispersing agents. E. LEWKOWITSCH.

**Production of bleaching agents [for flour etc.].** S. P. SCHOTZ (B.P. 345,985, 28.11.29).—Bleaching agents are produced by the interaction of an acid halide and an aldehyde, and are applied in admixture with powdered anhyd. oxides, hydroxides, carbonates, etc. of Li, Na, K, NH<sub>4</sub>, Ca, Mg, Zn, Th, Ce, or tungstates, vanadates, molybdates, or titanates. Examples are: PhCHO, BzCl, NaOH, and MgCO<sub>3</sub>, with or without Na<sub>3</sub>VO<sub>4</sub>; *o*-chlorobenzaldehyde, BzCl, etc.; PhCHO, AcCl, Na<sub>2</sub>CO<sub>3</sub>, KHCO<sub>3</sub>, MgCO<sub>3</sub>, Ce<sub>2</sub>(CO<sub>3</sub>)<sub>3</sub>. C. HOLLINS.

**Coloration of materials made of or containing organic derivatives [esters or ethers] of cellulose.** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 346,751, 9.1.30).—Pyrazolone dyes, preferably unsulphonated, are stable to reducing discharges and are added to the discharge paste for coloured discharge printing. Examples are: aniline, *o*- or *p*-anisidine, or amino-acetanilide → 1-phenyl-3-methyl-5-pyrazolone.

C. HOLLINS.

**Vat assistant for use in dyeing.** J. G. KERN and C. J. SALA, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,782,122, 18.11.30. Appl., 4.8.27).—"Tri-ethanolamine" or other hydroxylated alkylamine is added to a vat-dye paste to improve the exhaust, depth and brightness of shade, and levelling power.

C. HOLLINS.

**Manufacture of azo dyes on the fibre [ice colours].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 347,113, 20.1.30).—An unsulphonated diazo-compound is coupled on the fibre with an arylamide of 5-hydroxy- $\alpha$ -naphthacarbazole-4-carboxylic acid (annexed formula). Examples are: *o*-toluidide with diazotised 5-nitro-*o*-anisidine (blue-black) or 5-chloro-*o*-toluidine (violet-brown); *p*-anisidine with diazotised 5-nitro-*o*-anisidine (blue-black) or 5-chloro-*o*-toluidine (green-black);  $\beta$ -naphthylamide with diazotised 5-nitro-*o*-anisidine (red-black) or 5-chloro-*o*-toluidine (brown); 5-methoxy-*o*-toluidide with diazotised 5-nitro-*o*-anisidine or *o*-toluidine (black).

C. HOLLINS.

**Manufacture of diazotisable fibres.** C. GRÄNACHER (B.P. 347,117 and 347,263, [A] 21.1.30, [B] 23.1.30. Switz., [A] 17.4.29. [A] Addn. to B.P. 346,385; B., 1931, 672).—Cotton fibres, regenerated or partly acetyl-

ated cellulose, etc. are heated at 90–100° with (A)  $\alpha$  homologue of nitrobenzyl halide, e.g., nitromethylbenzyl chloride, or a chloronitrobenzene, e.g., 1-chloro-2:4-dinitrobenzene, or (B) an *o*-dinitrobenzene, e.g., 4-bromo- or 4:5-dichloro-1:2-dinitrobenzene, in presence of acid-binders (Li<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>) other than a caustic alkali; the NO<sub>2</sub> groups are then reduced.

C. HOLLINS.

**Treatment [printing] of [cellulose ester or ether] textiles etc.** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 346,694, 2.1.30).—*p*-Dihydroxy-compounds, e.g., quinol, toluquinol, and their mono-ethers and halogen derivatives, are added to printing pastes, especially in discharge printing, as aids to penetration.

C. HOLLINS.

**[Production of crêpe effects in] textile materials.** BRIT. CELANESE, LTD., and W. A. DICKIE (B.P. 348,589, 13.11.29).—Threads of degummed silk or of org. derivatives of cellulose are impregnated with materials containing casein, rubber, etc., which are then treated with CH<sub>2</sub>O, tannin, etc. to prevent softening during the swelling process. The coated threads, after highly twisting, are incorporated into fabrics which are treated with H<sub>2</sub>O, aq. EtOH, or salt solutions to cause swelling of the coating without rendering it relatively soft or yielding; the coating is subsequently removed in the swelling bath, if necessary after adjustment of its temp. and/or composition.

F. R. ENNOS.

**Protection of wool, fur, hair, etc. against attack by textile pests [mothproofing agents].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,039, 9.1.30).—H<sub>2</sub>O-sol. thiuronium salts obtained, e.g., from phenyl- or tolyl-thiocarbamide and ethylene dibromide or benzyl or 2:6-dichlorobenzyl chloride, are used. C. HOLLINS.

**Rotary machines for washing, decolorising, disinfecting, etc. [fabrics].** E. S. EYMERIC (B.P. 349,674, 10.3.30. Tunis, 3.4.29).

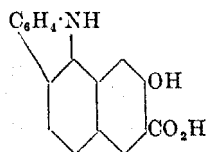
**Cellulose esters.** See V. **Bleaching powder.** Al salts.—See VII. **Laundry soaps.**—See XII. **Moulded articles.**—See XIII. **Rubber-fabric finishes.**—See XIV.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Characteristics of a [calcium-]vanadium catalyst and a new catalyst for sulphuric acid [manufacture].** W. W. SCOTT and E. B. LAYFIELD (Ind. Eng. Chem., 1931, 23, 617–620).—Conversions of SO<sub>2</sub> into SO<sub>3</sub> with the Holmes-Elder Ca-V catalyst (B., 1930, 659) with varying rates of flow were determined in the laboratory. A similar catalyst prepared by adding K vanadate and Na silicate to 5% BaCl<sub>2</sub> solution was similarly tested. It was found to operate slightly better at 450° than at 500° and better than the Ca-promoted catalyst under corresponding conditions; 1 lb. of catalyst with a flow of 17–35 cu. ft. of 8% SO<sub>2</sub> per hr. gives a conversion of 98.0–98.8%. Otherwise 1 lb. of V will produce 5–10 lb. of H<sub>2</sub>SO<sub>4</sub> per hr. at 98% or 13 lb. at 97%.

C. IRWIN.

**Dehydration of salts of phosphoric acid.** S. S. DRAGUNOV (Udobr. Urozhai, 1930, 2, 409–416).—The percentage of pyro- and meta-phosphates, and the speed





of transformation at various temp., are determined from graphs for the acid and for the Na, K, and Ca salts.  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$  is more sol. in citrate solution than  $\text{CaHPO}_4$ . Phosphate should not be dried above  $100^\circ$  for more than 30–50 min. Insol.  $\text{Ca}(\text{PO}_3)_2$  can be re-hydrated at high pressure. CHEMICAL ABSTRACTS.

**Causticisation of sodium carbonate by ferric oxide.** XV. Thermal change in a mixture of sodium carbonate and ferric oxide in an atmosphere of carbon dioxide. M. MATSUI and K. BITO (J. Soc. Chem. Ind., Japan, 1931, 34, 149–150 B; cf. A., 1928, 243).—Experiments with 2–3 mg. of the reactants gave for the dissociation temp.  $855$ – $856^\circ$ . H. F. GILLBE.

**Determination of free alkali and carbonate in alkaline hypochlorites.** KERNY (J. Pharm. Chim., 1931, [viii], 13, 570–573).—Neutral  $\text{H}_2\text{O}_2$  (B.P.; 5 c.c.) is agitated with 10 c.c. of Dakin's solution or eau de Javelle diluted with  $\text{H}_2\text{O}$  (1 : 10) until effervescence has ceased; 50 c.c. of boiled, distilled  $\text{H}_2\text{O}$  and 3 drops of phenolphthalein solution are added and the mixture is titrated with  $0.1N\text{-H}_2\text{SO}_4$  2–3 drops at a time, and with const. shaking. The carbonate is determined on the same solution and in a similar manner after the addition of methyl-orange. E. H. SHARPLES.

**Determination of total nitrogen in commercial calcium cyanamide.** R. POHLAND (Z. angew. Chem., 1931, 44, 386–388).—Total N in the impurities in commercial  $\text{CaCN}_2$  may be determined by the Kjeldahl method, heating for more than 1 hr. being unnecessary under the conditions described. H. F. GILLBE.

**Ammonium carbonate treatment of polyhalite.** J. R. HILL and J. R. ADAMS (Ind. Eng. Chem., 1931, 23, 658–661).—In Texas and New Mexico large deposits of polyhalite,  $2\text{CaSO}_4 \cdot \text{MgSO}_4 \cdot \text{K}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ , occur. As it is only very slowly sol. in  $\text{H}_2\text{O}$  it has been proposed to extract the K by leaching with  $(\text{NH}_4)_2\text{CO}_3$  solution. This alone was found to ppt. less than half the Mg, but with the addition of an equal wt. of aq.  $\text{NH}_3$  precipitation of Mg as also of Ca is practically complete. The insol. salt,  $\text{MgCO}_3 \cdot (\text{NH}_4)_2\text{CO}_3 \cdot 4\text{H}_2\text{O}$ , is formed. The solution can readily be brought to saturation with respect to  $\text{K}_2\text{SO}_4$  by shaking for 1.5 hr. at room temp. The liquor forms mixed crystals of  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  on evaporation, and the  $\text{NH}_3$  content of the filter cake can readily be recovered by steaming. The saturated mother-liquor contains 5.43 g.  $\text{K}_2\text{SO}_4$  per 100 c.c. Much higher concentrations can be obtained by treatment with  $(\text{NH}_4)_2\text{CO}_3$  alone, but the product contains considerable quantities of  $\text{MgSO}_4$ . C. IRWIN.

**Determination of phosphoric acid in phosphate rock.** E. N. ISAKOV and V. A. KAZARINEVA (Udobr. Urozhai, 1930, 2, 416–420).—The material (15–20 g.) passed through a 0.5-mm. sieve is dried for 3.5 hr. at  $105^\circ$ ; 4 g. are dissolved in 40 c.c. of aqua regia during 30 min. After dilution to 0.0001 Denigès' colorimetric method is employed. The max. error is 2%.

## CHEMICAL ABSTRACTS.

**Oxidation of ferrous sulphate.** W. A. DAMON (67th Annual Rep. on Alkali etc. Works, 1930, 21–27).—When hot conc.  $\text{FeSO}_4$  solution is oxidised with  $\text{HNO}_3$  in presence of  $\text{H}_2\text{SO}_4$  the primary gaseous product is  $\text{NO}$ , and  $\text{NO}_2$  is formed only if  $\text{HNO}_3$  is present in excess.

The evolution of gas is at first slow, but when about two thirds of the total gases have been evolved rises suddenly and rapidly. It is doubtful whether this is entirely due to the solubility of  $\text{NO}$  in  $\text{FeSO}_4$ . If cryst.  $\text{FeSO}_4$  be added to the mixed acids the reaction is essentially similar, but the rate of gas evolution is determined by the rate of crystal addition. The best method of recovering  $\text{NO}$  if a chamber  $\text{H}_2\text{SO}_4$  plant is available is by its absorption in  $\text{H}_2\text{SO}_4$ . Alternatively, passage of the exit gases through scrubbers packed with cryst.  $\text{FeSO}_4$  leads to a recovery of 16–27% of  $\text{HNO}_3$ , as determined in the laboratory. The actual oxidising agent is probably  $\text{N}_2\text{O}_3$  as the reaction proceeds readily at atm. temp. The residual gases may then be absorbed in  $\text{H}_2\text{O}$ . C. IRWIN.

**Densities of molten cryolite and of molten mixtures of cryolite and barium fluoride.** N. KAMEYAMA and A. NAKA (J. Soc. Chem. Ind., Japan, 1931, 34, 140–142 B).—Contrary to earlier reports, the sp. gr. of cryolite increases continuously with rise of temp. The sp. gr. of mixtures of cryolite and  $\text{BaF}_2$  have been determined from  $950^\circ$  to  $1122^\circ$ . At  $1050^\circ$  cryolite has  $d$  2.03, and a mixture containing 75.1 mol.-% of  $\text{BaF}_2$  has  $d$  3.74. The sp. gr. required in the electrolytic refining of Al by the three-layer method may thus be obtained by use of a suitable mixture of these compounds. H. F. GILLBE.

**Utilisation of secondary reactions in igneous electrolysis.** L. ANDRIEUX (Chim. et Ind., 1931, 25, 1047–1057).—A discussion. The prep. of  $\text{TiB}_2$ ,  $\text{TaB}_2$ ,  $\text{CbB}_2$ , and borides of the rare-earth metals by the electrolysis of fused materials is described.

C. W. GIBBY.

**Technology of solid carbon dioxide.** G. T. REICH (Chem. Met. Eng., 1931, 38, 271–274).—When liquid  $\text{CO}_2$  at 1100 lb. per sq. in. pressure is subjected to a sudden release of pressure it is partly solidified. The  $\text{CO}_2$  snow is compressed into blocks at 800 lb. per sq. in. The yield is 50%. In the Dry Ice and Frick machines the chamber for collecting the snow acts also as the chamber for compressing it. In the Carba process the snow with much adhering liquid is formed first, and on lowering the pressure the whole solidifies to a block. The economics of the process and the uses of solid  $\text{CO}_2$  are discussed. D. K. MOORE.

**Oxidation of phosphorus by water and the preparation of phosphoric acid.** L. HACKSPILL (Chim. et Ind., 1931, 25, 1058–1063).—An account of the methods available for the manufacture of  $\text{H}_3\text{PO}_4$ .

C. W. GIBBY.

**S compounds.**—See I.  $\text{NH}_3$  from gasworks.—See II. Metal powders from salts.—See X. Oxidation in soils.—See XVI.  $\text{PH}_3$  in spring waters.—See XXIII.

## PATENTS.

**Production of sulphuric acid from waste gases [from ammonia saturators] containing hydrogen sulphide and carbonic acid.** W. W. GROVES. From Dr. C. OTTO & Co. G.M.B.H. (B.P. 349,238, 14.4.30).—The gases are introduced into a furnace, heated to  $700^\circ$ , into which air is admitted to convert the  $\text{H}_2\text{S}$  into  $\text{SO}_2$ . The furnace is of the double-tube type, one portion being

subjected to external heating, the other embedded in a bad heat-conducting material, *e.g.*, bauxite. The gases are dried, first by indirect cooling and then by  $\text{H}_2\text{SO}_4$ , and subsequently enter a contact furnace, the  $\text{SO}_3$  produced being absorbed by the  $\text{H}_2\text{SO}_4$  used in the drying process. W. J. WRIGHT.

**Recovery of gas mixtures rich in sulphur dioxide [for the sulphuric acid contact process].** M. SCHROEDER (B.P. 349,287, 14.5.30. Ger., 15.5.29).—Gas mixtures containing  $\text{SO}_2$ , after purification, compression, and cooling, are treated under pressure with  $\text{H}_2\text{O}$  so as to give a relatively conc. solution of  $\text{SO}_2$ . The solution is passed down a tower in countercurrent to air, which may be heated, at atm. pressure or under, a gas mixture having a higher % of  $\text{SO}_2$  than the original one being thus obtained. W. J. WRIGHT.

**Purification of sulphuric acid.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 348,866, 6.5.30).—To remove nitric constituents from  $\text{H}_2\text{SO}_4$  above 60% concentration,  $\text{SO}_2$  dissolved in  $\text{H}_2\text{SO}_4$  or water is added to the impure acid and the mixture is treated with a current of air or flue gas at 100–200°. About 1.5 mols. of  $\text{SO}_2$  should be added for each mol. of  $\text{HNO}_3$ . H. ROYAL-DAWSON.

**Manufacture of sodium and ammonium bicarbonates.** A. MENTZEL (B.P. 348,482, 21.8.30. Ger., 23.8.29).—The  $\text{CO}_2$  required in the  $\text{NH}_3$ -soda process is derived from gases obtained in the low-temp. carbonisation of coal or lignite, these being first purified from tar and, after the precipitation, being utilised as fuel. W. J. WRIGHT.

**Production of alkali carbonates.** WINTERSHALL A.-G. (formerly KALI-IND. A.-G.), C. T. THORSSELL, and A. KRISTENSSON (B.P. 348,825, 8.4.30. Ger., 16.4.29. Addn. to B.P. 300,629; B., 1929, 850).—Crude sylvinic salts are substituted for the KCl formerly employed, so that a mixture of solid K and Na carbonates is obtained, these being converted into the bicarbonates and separated. W. J. WRIGHT.

**Fixation of ammonia-nitrogen.** N. CARO and A. R. FRANK (B.P. 349,001, 29.1.30. Addn. to B.P. 347,641; B., 1931, 630).—Wet humic acid, or vegetable matter containing or capable of forming it, is heated under pressure to 200° to render it more dense, and after filtering is treated at 50–300° in a wet condition with  $\text{NH}_3$  and gases containing  $\text{O}_2$  under pressure. Steam or  $\text{CO}_2$  may be introduced. The speed of oxidation may be regulated by adding positive catalysts, *e.g.*, alkaline substances or heavy metal salts, or negative catalysts, *e.g.*, oxides or carbonates. W. J. WRIGHT.

**Treatment of phosphate rock and the like.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 349,877, 23.9.30. Nor., 28.9.29. Addn. to B.P. 339,340; B., 1931, 201).—Part of the mother-liquor from the crystallisation in the prior process is used for washing and cooling the escaping gases and is then added to the  $\text{HNO}_3$  used for dissolving fresh phosphates; another part, cooled to –20°, is added to the solution prior to or during crystallisation. The quantities of the materials and the concentration of the  $\text{HNO}_3$  are so adjusted that the mother-liquor yields immediately a dry product on neutralisation with  $\text{NH}_3$ . L. A. COLES.

**Production of potassium monophosphate.** KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 349,409, 30.9.30. Ger., 25.10.29. Addn. to B.P. 327,885; B., 1930, 861).—KCl and  $\text{H}_3\text{PO}_4$  (1 : 1 mol.) are heated, without reduction of the  $\text{H}_2\text{O}$  content, with the mother-liquor of the process, which is saturated with  $\text{KH}_2\text{PO}_4$ ,  $\text{H}_3\text{PO}_4$ , until the Cl is completely expelled; on cooling the solution,  $\text{KH}_2\text{PO}_4$  crystallises and is removed. L. A. COLES.

**Production of diammonium phosphate.** KUNST-DÜNGER PATENT VERWERTUNGS A.-G. (B.P. 348,970, 12.8.30. Swed., 23.8.29).— $\text{NH}_3$  is absorbed by a boiling solution of  $\text{H}_3\text{PO}_4$  or  $\text{NH}_4\text{H}_2\text{PO}_4$ , loss of  $\text{NH}_3$  being avoided by passing the mixture of steam and escaping  $\text{NH}_3$  into dil.  $\text{H}_3\text{PO}_4$ , preferably heated to the b.p. W. J. WRIGHT.

**Manufacture of sodium hyposulphite and pure zinc oxide or carbonate.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,776, 17.3.30).—The solution obtained by treating a suspension of Zn dust with 1 equiv. of  $\text{SO}_2$  is treated with Zn dust, ZnO, or  $\text{ZnCO}_3$ , and is then filtered and treated with NaOH or  $\text{Na}_2\text{CO}_3$  to ppt. ZnO or  $\text{ZnCO}_3$ , which is washed and dried. L. A. COLES.

**Composition for skating surfaces.** M. H. GURTH (B.P. 348,178, 6.2.30).—A fused mixture of cryst.  $\text{Na}_2\text{S}_2\text{O}_3$  (100 pts.), up to 10 pts. of Na lactate, and (if desired) 2–20 pts. of cryst.  $\text{Na}_2\text{SO}_4$ ,  $\text{Na}_2\text{CO}_3$ , and/or  $\text{Na}_2\text{HPO}_4$  is used. L. A. COLES.

**Manufacture of double [nitrate] salts.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,301, 24.5.30).—Salts of the type  $\text{M}'(\text{OH})\text{NO}_3$ ,  $\text{M}'\text{NO}_3$  are obtained by fusing the constituents salts and allowing the product to cool slowly. Instead of  $\text{M}'(\text{OH})\text{NO}_3$  equimol. quantities of  $\text{M}'(\text{OH})_2$  and  $\text{M}'(\text{NO}_3)_2$  may be used. In the above formulæ  $\text{M}' \equiv \text{Ca}$ ,  $\text{Sr}$ ,  $\text{Ba}$ , or  $\text{Mg}$  and  $\text{M}' \equiv \text{K}$ ,  $\text{NH}_4$ , or  $\text{CO}(\text{NH}_2)_2$ . A. R. POWELL.

**Production of stable bleaching powder.** I. G. FARBENIND. A.-G. (B.P. 348,992, 25.9.30. Ger., 26.10.29).—Finely-divided bleaching powder is brought for a few sec. in contact with a current of air at above 150° (preferably 160–180°) flowing in the same direction. L. A. COLES.

**Production of storage-proof bleaching powder.** A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 349,358, 15.7.30).—Bleaching powder containing at least 36% available Cl is mixed with 0.5–1.0 pt. of CaO per 1 pt. of total  $\text{H}_2\text{O}$  and, after the CaO has slaked, the mixture is treated with a current of hot air to reduce the  $\text{H}_2\text{O}$  content to about 1%. L. A. COLES.

**Manufacture of aluminium compounds and phosphoric acid.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,363, 1.5.30).—Material containing Al compounds and  $\text{H}_3\text{PO}_4$  is leached at 60–100° by HCl in countercurrent, the solution being evaporated, cooled, treated with gaseous HCl to ppt.  $\text{AlCl}_3$ , and filtered. The filtrate is heated or treated with a current of gas or air to remove the HCl and recover  $\text{H}_3\text{PO}_4$ . W. J. WRIGHT.

**Manufacture of aluminium (A) complex compounds, (B) salts of organic acids, and (C) solid water-soluble salts of oxalic acid.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,419, 348,790,

and 348,789, 24.3.30).—(A) Alkali aluminates alone or together with K, Na, Ba, Ca, or Mg carbonates are dissolved in aq. solutions of aliphatic dicarboxylic or hydroxycarboxylic acids containing  $< C_6$  per mol. Complex salts of two or more of these elements with oxalic, tartaric, and lactic acid are described; formulae of the cryst. salts are given in many cases. (B) Granulated Al is activated by immersion in a solution of  $HgCl_2$  and is then dissolved by boiling with a solution of an aliphatic di- or hydroxy-carboxylic acid, *e.g.*, lactic, oxalic, tartaric, or glycolic acid. (C) Solutions of neutral or basic Al oxalates are dehydrated at above  $100^\circ$ , preferably in the form of thin films or as spray, in a heated gas stream or in vac. Application of such Al salts in the dyeing industry and for pharmaceutical purposes is indicated. A. R. POWELL.

**Manufacture of titanium preparations.** I. G. FARBENIND. A.-G. (B.P. 348,724, 21.2.30. Addn. to B.P. 346,009; B., 1931, 645).—Part of the excess  $H_2SO_4$  in the solution is removed as  $CaSO_4$  by addition of  $CaCO_3$ , and the Ti in the filtrate is then precipitated by hydrolysis as basic sulphate. A. R. POWELL.

**Manufacture of antimony oxides.** DEUTS. SCHMELZ-U. RAFFINIERWERKE A.-G., and H. W. GRIMM (B.P. 348,138, 4.2.30).—Powdered Sb or Sb ore is introduced into a combustion furnace into a current of gas or vapour, and the latter is burned with addition of  $O_2$  or air. The material may also be introduced into the flame of burning coal dust or oil fuel, so that it is held in suspension by the flame. The oxide which fuses and flows away may be mixed with further quantities of Sb ore. W. J. WRIGHT.

**Manufacture of satisfactory emanating preparations.** DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 348,705, 18.2.30. Ger., 6.3.29).—Cone. preparations containing Ra, meso-Th, etc. dispersed on  $Fe(OH)_3$  ( $Fe : Ra = 16 : 1$ ) are prepared by precipitating the  $Fe(OH)_3$  in the presence of Ra salts, and washing the ppt. with  $(NH_4)_2CO_3$  solution to prevent dissolution of the Ra. L. A. COLES.

**Apparatus for decomposing salts or the like.** H. B. BISHOP (B.P. 349,065, 11.1.30. U.S., 25.9.29).—The apparatus comprises a reaction chamber with two sets of stirring arms, a spraying device, fed by means of a siphon, and a dust catcher through which the gaseous products of decomp. pass. In operation, the salt is heated below its m.p. and volatilised, and the acid is sprayed in during agitation. W. J. WRIGHT.

**Preventing the caking of salts.** PATENTVERWERTUNGS A.-G. ALPINA (B.P. 348,060, 28.1.30. Ger., 28.1.29).—Fertilising salts in a finely-divided state are subjected to pressure in an extrusion press, with heating or cooling, the extruded material being reduced to granules of the desired size, either by the jerky action of the press or by cutting. W. J. WRIGHT.

**Production of hydrogen by the action of steam on metals.** T. LICHTENBERGER and L. KAISER (B.P. 348,018, 30.1.30. Ger., 10.7.29).—In a continuous process, the molten metal and a molten salt mixture are contained in a conical trough, in which is fixed a gas-collecting bell, permeable to H at its tapered lower

portion only. The metal oxides, formed by introducing steam into the metal, rise to the surface and are there reduced by water-gas generated by the action of steam on a layer of coal, the reduced metal falling to the base of the trough. The H produced by oxidation of the metal thus passes directly into the bell without contamination by the oxides. Residual gases from the reduction of the oxides are utilised to heat the trough.

W. J. WRIGHT.

**Electrolytic manufacture of chlorine.** WESTVACO CHLORINE PRODUCTS, INC., Assees. of F. S. LOW (B.P. 348,792, 24.3.30. U.S., 27.1.30).—HCl (20% solution) is continuously supplied to the cathode chamber of an electrolytic cell and maintained therein at  $45-100^\circ$ . Leaving the anode chamber as 10% solution, it passes through a blow box, in which the Cl is expelled by air into a tower, where it is regenerated by upward-flowing HCl solution, being finally pumped back to the supply tank. Graphite anodes and a cathode of Cu coated with spongy Cu give the best results. Cl of 99.9% concentration is obtained. W. J. WRIGHT.

**Settling apparatus [for  $NH_4$  salts].** Treating gases with liquids.—See I.  $CS_2$  from gas mixtures.—See II. Fertilisers.—See XVI. Regeneration of sulphites from fermentation process.—See XVIII. Testing for CO in air.—See XXIII.

## VIII.—GLASS; CERAMICS.

**Insulation of glass-furnace regenerators.** R. M. LANGLEY (J. Amer. Ceram. Soc., 1931, 14, 376—381).—The economy to be effected by insulation is greater than that calc., because air leakage is also reduced and more accurate combustion control is made possible.

J. A. SUGDEN.

**Hydraulic classifiers as an aid to batch grinding; some possible economies in grinding pottery material.** L. ANDREWS (Trans. Ceram. Soc., 1931, 30, 98—111).—During an 8-hr. grind on a pan mill, the material was examined at 2-hr. intervals, and the relative proportions of different grain-sizes, the relative quantities of  $-0.01$  mm. material produced, and the relative increase in the surface of the material for each successive period were determined. A simple hydraulic classifier for use with existing pan mills or cylinders is described, and its advantages are pointed out. A rapid laboratory elutriator for continuous works use is also described. F. SALT.

**Grading and sampling of Missouri burley and diaspore clays.** C. R. FORBES (J. Amer. Ceram. Soc., 1931, 14, 382—388).—Confusion in the present terminology of Missouri high- $Al_2O_3$  clays is pointed out, and a uniform practice of sampling and analysis and a sliding scale of prices, based on the  $Al_2O_3$  content, are recommended. J. A. SUGDEN.

**Florescence. III. Effect of firing conditions on the soluble salt content of clayware.** F. L. BRADY and E. H. COLEMAN (Trans. Ceram. Soc., 1931, 30, 169—184).—Two brick-making clays were fired to various temp. between  $400^\circ$  and  $1100^\circ$  in atm. of air, air plus 1.5% of  $SO_2$ , and these with the addition of steam. Little variation in the sulphate content of a calcareous clay free from pyrites fired in air was noted,

but there was a well-marked max. at about 700° when SO<sub>2</sub> was present, and firing to the highest temp. did not remove the sulphate. A pyritic clay fired in air showed a max. sulphate content at 700°, which was increased when SO<sub>2</sub> was added. Firing above this temp. caused a marked reduction in sulphate content.

F. SALT.

**Calcium fluoride porcelain.** A. DAMIENS (Compt. rend., 1931, 192, 1235—1237).—A mixture of 75 pts. of natural fluor, ground to 120-mesh, with 25 pts. of carefully washed CaF<sub>2</sub> precipitated from very dil. solutions of KF and CaCl<sub>2</sub>, is worked up with 20% of H<sub>2</sub>O, then pressed, and dried at 100°, forming a compact mass which can be worked or turned into tubes, crucibles, etc. These are then baked at 800—1300°, care being taken to prevent contact with furnace gases. The product is semi-transparent, impermeable to H<sub>2</sub>O, and capable of withstanding rapid changes of temp. Different portions can be united by aid of the original mixture and reheating.

C. A. SILBERRAD.

**Metal constituents in bright-gold [lustre used in ceramic decoration].** I. Heat-resisting metals.

II. Fastening metal, bismuth. III. Metals other than bismuth and rhodium. A. NAKATSUCHI (J. Soc. Chem. Ind., Japan, 1931, 34, 164—165 B, 165—166 B, 166 B).

—I. Ordinary bright-gold lustre when fired on ceramic ware above 500° leaves a deposit of metal which shows many grain boundaries and cracks. Rh, Al, Th, and Sn compounds in the lustre prevent this graining effect. There is an optimum concentration of Rh above which the fired lustre acquires a dark colour, but just below which a very bright smooth surface is obtained. The Rh in the metal film fired at 768° is present as finely-divided Rh<sub>2</sub>O<sub>3</sub>.

II. Bi compounds in Au lustre act as fluxes for fastening the Au film on the ware, but do not restrain grain growth, which, however, is retarded by addition of Rh. An excess of Bi in the lustre inhibits the beneficial action of Rh; optimum brilliancy is obtained only with certain definite proportions of Au, Bi, and Rh and with slow heating to 760° followed by slow cooling.

III. Addition of Cr, Al, Fe, and Ru resins to Au lustres containing Bi and Rh improves the brilliancy and still further restrains grain growth. More than 1.27% Cr in the metal deposit darkens the film.

A. R. POWELL.

**Testing the effect of slags on refractory bricks.**

J. SCHAEFER and F. BAUMHAUER (Feuerfest, 1931, 7, 33—36).—Three types of bricks, one of SiO<sub>2</sub> and two of magnesite, were tested for slag-resistance in a new type of furnace. At 1600° the SiO<sub>2</sub> brick was badly corroded by open-hearth furnace slag; with the magnesite bricks little fusion took place between the slag and the refractory, but the slag penetrated the structure of the brick, forming two layers. The chemical composition, sp. gr., and thickness of these layers were determined. From these data the degree of corrodibility is presented numerically, but the appearance of the brick and of its fracture after testing give sufficient indication to the skilled observer.

F. SALT.

**Refractories for furnaces.**—See XI.

## PATENTS.

**Non-splintering glass.** C. H. FIELD and D. HASLETT (B.P. 348,723, 21.2.30).—An adhesive for uniting two glass sheets with or without an interposed cellulose ester sheet contains a cellulose ester, lactic acid, and (preferably) Et lactate.

L. A. COLES.

**Manufacture of splinterless glass.** I. G. FARBER-IND. A.-G. (B.P. 349,283, 13.5.30. Ger., 13.5.29).—The strengthening layer comprises a previously prepared vinyl ester (e.g., acetate) final polymerisation product, incorporated, if desired, with a softening agent.

L. A. COLES.

**Manufacture of graphitic refractories.** R. M. DOIDGE, and MORGAN CRUCIBLE CO., LTD. (B.P. 348,149, 7.2.30).—Mixtures comprising Si (or a Si alloy) and more than 1 equiv. of graphite, together with SiC, amorphous C, tar, clay, binders, fluxes, etc., as desired, are moulded and fired at 1200—1500°.

L. A. COLES.

**Manufacture of bonded abrasive articles.** CARBORUNDUM CO., LTD., Assees. of R. C. BENNER (B.P. 349,602, 25.2.30. U.S., 14.10.29).—A dispersing agent (e.g., bentonite) and H<sub>2</sub>O are kneaded successively at about 50° into rubber swollen with, e.g., benzene, and the mixture is heated at 82° to effect inversion of phase; abrasive fillers, H<sub>2</sub>O, Pb(OAc)<sub>2</sub>, and S are then added and the mixture is vulcanised.

L. A. COLES.

**Grinding or polishing of sheet glass.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY, Assees. of P. TOMMY-MARTIN and D. A. LOUPE (B.P. 349,928, 1.2.30. U.S., 4.2.29).

**Centrifugal machines [for kaolin etc.].**—See I. **Abrasive wheels.**—See XIII.

## IX.—BUILDING MATERIALS.

**Reactions in rotary cement kilns.** T. HASHIMOTO and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1931, 34, 130—132 B).—Examination of samples of cement taken at intervals along kilns of 200 ft. length reveals the existence of 4 reaction zones, viz., those of preheating, calcining, sintering, and cooling. The length of each zone and the progress of the reaction vary with the method of firing. The reaction between Al<sub>2</sub>O<sub>3</sub> and CaO commences only when all the CaCO<sub>3</sub> has decomposed; it appears, however, that after the decomp. has commenced part of the CaO is free and part combined. The free CaO is removed very rapidly on passing beyond the limit of the calcining zone.

H. F. GILLBE.

**Hydrated lime and diatomaceous earth (diatomite) in oil-well cement.** A. REID (J. Inst. Petroleum Tech., 1931, 17, 211—224).—A cement grout with a 40—50% H<sub>2</sub>O—cement ratio can be easily handled by a pump, but the large excess of H<sub>2</sub>O above that required for proper hydration reduces the ultimate strength of the cement. Experiments made to reduce the viscosity of the cement grout by adding CaO and diatomaceous earth showed that these substances in all proportions appeared rather to increase the viscosity, but for structural work they might be used to make the material more workable. Addition of CaO slightly increases the setting time and tensile strength up to 7 days.

J. A. SUGDEN.

**Relations between burning practice and the main components of the raw meal [for cement]—lime, silica, and alumina.** O. FREY (Cement, 1931, 4, 392—397).—Results of careful sampling and examination of a raw stratified deposit determine the curve of uniformity of the raw materials, which is a valuable factor in selecting the nature of the kiln lining. The most important factor is the clinkering temp., which may be low (1300—1350°), normal (1350—1450°), or high (1450—1550°), the last indicating high SiO<sub>2</sub> or high Al<sub>2</sub>O<sub>3</sub> contents. Taken in conjunction with the fusing effect of the fuel ash the character of the lining may be selected, as the best quality of firebrick is unsuitable above 1400°, a high-Al<sub>2</sub>O<sub>3</sub> brick or clinker concrete being generally more suitable. C. A. KING.

**Mixed Portland cements.** VIII. S. NAGAI (J. Soc. Chem. Ind., Japan, 1931, 34, 162—164 B).—The free CaO in hydrated Portland cement products is increased by heating and on subsequent exposure to the air the CaO is converted first into Ca(OH)<sub>2</sub> and then into CaCO<sub>3</sub>; the resulting expansion causes disintegration of the cement or concrete. On the other hand, mixed cements of the "soliditite" and "neo-soliditite" types show a smaller content of free CaO after heating than when freshly mixed, and hence do not disintegrate.

A. R. POWELL.

**Hardening of Portland cement.** F. F. TIPPMMANN (Zement, 1930, 19, 1225—1234; Chem. Zentr., 1931, i, 1658—1659).—Hydrolysis of Ca silicate to SiO<sub>2</sub> gel and Ca(OH)<sub>2</sub> is followed, in hardening, by interaction of the products. A micro-procedure for observing the process in mortar is described and the effect of gypsum is discussed.

A. A. ELDRIDGE.

**Hydraulic lime in concrete.** G. W. HUTCHINSON (Eng. News-Rec., 1931, 106, 974—976).—Measurements of the compressive strengths of concrete mixtures obtained by mixing Portland cement with hydraulic CaO indicate the possible usefulness of the latter material for making concrete more watertight, durable, and uniform.

O. J. WALKER.

**Titanium mineral in ultrabasic titaniferous slags.** C. W. CARSTENS (Z. Krist., 1931, 77, 504—505).—Analysis and X-ray examination show the brownish-yellow skeleton crystals occurring in Al slags and Al cement clinker to be perovskite. There is no trace of a TiO (cf. A., 1929, 19).

C. A. SILBERRAD.

**Refractory coatings.** L. LITINSKY (Feuerfest, 1931, 7, 65—80).—The thermal expansion of a refractory cement or coating must be closely adapted to that of the brickwork. Brief descriptions are given of the many commercial cements etc. and of the different types of sprayers. An appendix gives about 170 references to the literature.

F. SALT.

**Effect of building materials on paint.**—See XIII.

## PATENTS.

**Concentration of [raw cement] sludge material.** F. KRUPP GRUSONWERK A.-G. (B.P. 348,062, 29.1.30. Ger., 21.12.29).—The sludge is drawn in thin layers from a recess in the bottom of the exhaust gas duct of the calcining furnace by rotating discs set in the direction of the gas stream and dipping into the recess.

The partly dried sludge is scraped from the discs and falls to the bottom of the recess, whence it is conveyed to the furnace.

L. A. COLES.

**Anhydrite or gypsum cementing compositions.** P. P. BUDNIKOFF (B.P. 348,766, 12.3.30).—Cements are prepared by adding natural or artificial anhydrite, or hydrated CaSO<sub>4</sub> to dolomite which has been baked at 600—900°, or to mixtures of MgO, MgCO<sub>3</sub>, and CaCO<sub>3</sub>. Sulphates of heavy or alkali metals may be added during the crushing process.

C. A. KING.

**Production of building material.** EUREKA GES. F. LEICHTBAU U. ISOLIERPLATTEN, and C. E. DUNZ (B.P. 349,629, 20.10.30).—Vegetable fillers are worked up with a suspension of a hydraulic binder (Portland cement, plaster of Paris, etc.) in agar-agar solution to a plastic mass which is moulded hot and allowed to set.

L. A. COLES.

**Manufacture of imitation marble.** S. GAUDIO (B.P. 349,485, 27.1.30).—Slabs etc. prepared by the setting of plastic mixtures comprising "Keene's superfine white cement," marble or alabaster powder, CaO, alum, and H<sub>2</sub>O, with the addition, if desired, of colours, are immersed in H<sub>2</sub>O on the surface of which a linseed oil-turpentine colour solution is floated and stirred to give a stripe effect to the slabs. After removal and drying, the slabs are steeped successively in oil, in liquid bronze, and in lacquer, and are finally wax-polished.

L. A. COLES.

**Metal [copper]-clad roofing elements.** INTERNAT. COPPERCLAD CO., ASSEES. OF S. LEVY (B.P. 349,690, 22.3.30. U.S., 22.3.29).—Felted fibrous material impregnated with a waterproofing compound is coated on the under surface with asphalt into which, e.g., crushed slate is pressed, and on the upper surface with electroconductive material (bronze powder) which is covered electrolytically with Cu over the parts to be exposed to the air.

L. A. COLES.

**Felt-base floor covering.** ARMSTRONG CORK CO., ASSEES. OF E. J. PIEPER (B.P. 349,792, 12.6.30. U.S., 17.6.29).—A felt base impregnated with bituminous material is provided with successive coatings of a casein size, an alkyd resin (e.g., "Glyptal"), and a decorative oil- or nitrocellulose-base paint.

L. A. COLES.

**Mixing machines [for concrete].** J. S. WITHERS. FROM KOEHRING CO. (B.P. 348,837 and 349,723, 14.4.30).

**Production of laminated wooden boards.** B. F. MISKIN (B.P. 350,169, 1.5.30 and 31.1.31).

**Feeding rotary kilns. Liquid heating and storage. Mixing of moulding substances.**—See I. Nitro-compounds of decreased inflammability.—See III. Floor coverings etc.—See XIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Yield point and initial stages of plastic strain in mild steel subjected to uniform and non-uniform stress distribution.** G. COOK (Phil. Trans., 1931, A 230, 103—147).—The relation between the stress at the yield point in simple tension and in the distribution produced by torsion, flexure, and internal pressure in a

hollow cylinder was determined. All the results were consistent with the supposition that the initial dislocation resulting in elastic breakdown takes place at a critical value of the shear stress at a certain depth in the material, *e.g.*, the surface layer possesses a higher elastic limit than the interior. C. A. KING.

**Analysis of the coating of galvanised sheet steel.** F. W. SCOTT (Chemist Analyst, 1931, 20, No. 2, 4—5).—The coating is removed by 3.3*N*-H<sub>2</sub>SO<sub>4</sub> in contact with pure Zn, and the loss in wt. determined. The solution is titrated for Fe<sup>++</sup> with KMnO<sub>4</sub>, and Pb is determined as PbSO<sub>4</sub>. Cd in the filtrate is determined as CdS. Sn is determined in another sample by precipitation as Sn(OH)<sub>2</sub> and ignition to SnO<sub>2</sub>. Zn is determined by difference. CHEMICAL ABSTRACTS.

**Determination of iron in the coating of galvanised steel.** B. E. COHN (Chemist Analyst, 1931, 20, No. 2, 4).—The solution (*cf.* preceding abstract) is treated with HgCl<sub>2</sub> before titration with KMnO<sub>4</sub>.

CHEMICAL ABSTRACTS.

**Products of corrosion of steel [in oxygenated water].** H. O. FORREST, B. E. ROETHELI, and R. H. BROWN (Ind. Eng. Chem., 1931, 23, 650—653).—A 0.01*M*-solution of Fe<sup>++</sup> on agitation with O<sub>2</sub> precipitates Fe(OH)<sub>3</sub> at up to *p*<sub>H</sub> 8.5. At *p*<sub>H</sub> 11 the ppt. consists of Fe(OH)<sub>2</sub>. At intermediate points interaction produces a magnetic oxide. The corrosion rate of cleaned low-C steel cylinders in oxygenated H<sub>2</sub>O with and without rotation was measured. With initial rotation an impervious film of Fe(OH)<sub>3</sub> was formed and the corrosion rate was low. In the second, stationary, period local concentrations allowed the formation of Fe(OH)<sub>2</sub> in addition, and as the resulting magnetic oxide was not coherent corrosion increased. When rotation recommenced corrosion decreased again.

C. IRWIN.

**Resistance of "Silumin" to corrosion by acid and alkaline solutions.** O. SPENGLER and J. WIGAND (Z. Ver. deut. Zucker-Ind., 1931, 81, 260—266).—Laboratory experiments showed distilled H<sub>2</sub>O, 40% sugar solution, first-carbonatation juice, and molasses to have little effect at 30°, though the loss in wt. in contact with 0.1*N*-H<sub>2</sub>SO<sub>4</sub> and 0.1*N*-HCl was appreciable at this temp. after 32 days. At 80°, however, there was distinct evidence of attack after 21 days in the case of the 40% sugar solution (*p*<sub>H</sub> 3.2), of alkaline juice, of molasses, and of 0.1*N*-H<sub>2</sub>SO<sub>4</sub>. 0.1*N*-HCl caused a marked amount of attack at this higher temp.

J. P. OGILVIE.

**Influence of siliceous matter on the reduction of magnetic sands.** K. IWASE and M. FUKUSHIMA (J. Study Met., 1930, 7, 524—534).—The effect at 900—1150° was studied. At lower temp. the effect is small, but at higher temp. FeO and other substances combine with the SiO<sub>2</sub> forming semi-fused substances, and the rate of reduction is considerably retarded.

CHEMICAL ABSTRACTS.

**Influence of colloidal ferric hydroxide on the properties of moulding sand.** E. TAKAHASHI (J. Study Met., 1930, 7, 552—562).—The addition of colloidal Fe(OH)<sub>3</sub> increases either the plasticity or the gas-permeability according to the ramming conditions.

The increase in gas-permeability appears to be due to the coagulation of clayey particles.

CHEMICAL ABSTRACTS.

**Microchemical and luminescence-analytical examination of metal surfaces.** A. KUTZELNIGG (Mikrochem., 1931, 9, 360—366).—The presence of protective films of lacquer or varnish on metals can be recognised by their characteristic fluorescence on exposure to ultra-violet light; films of ZnO and basic Zn compounds under these conditions also fluoresce. Sulphide films may be detected by the azide-iodide reaction, and Cu<sub>2</sub>O and CuO films by their reaction with dil. H<sub>2</sub>SO<sub>4</sub>, the former turning black due to conversion into CuSO<sub>4</sub> and Cu, and the latter dissolving completely. Metal coatings are best identified by the streak test followed by treatment of the streak with a reagent giving a characteristic test for the metal; certain tests for Au, Ag, Cu, Sb, Sn, Pb, Ni, Cd, Fe, Zn, and Al are briefly described. A. R. POWELL.

**Experimental flotation of oxidised silver ores.** H. S. GIESER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1931, No. 401, 9 pp.).—A discussion of the org. S compounds available and their effects.

CHEMICAL ABSTRACTS.

**Micro-determination of platinum in alloys.** R. STREBINGER and H. HOLZER (Mikrochem., 1931, 9, 401—421).—The alloy is dissolved in aqua regia and the solution evaporated to dryness on the water-bath with a few drops of H<sub>2</sub>O<sub>2</sub>. The residue is dissolved in HCl and the solution evaporated with a few mg. of KCl. The dry mass is extracted with a 1:1 mixture of EtOH and Et<sub>2</sub>O free from aldehydes and the K<sub>2</sub>PtCl<sub>6</sub> collected in a micro-Neubauer crucible, washed with the extraction solution, dried, and ignited at 1200° in an electric furnace. The KCl is then extracted with hot H<sub>2</sub>O and the residual Pt ignited and weighed. Au, Pd, Rh, and base metals do not interfere; Ir quantitatively accompanies the Pt. For the colorimetric determination of Pt the K<sub>2</sub>PtCl<sub>6</sub> ppt. is washed with EtOH saturated with KCl, dried in a current of cold air, and dissolved in hot H<sub>2</sub>O. The solution is treated with 2 drops of HCl and a few mg. of KI and the colour of the resulting red solution of K<sub>2</sub>PtI<sub>6</sub> compared with that of a standard. A. R. POWELL.

**Throwing power of electroplating solutions.** S. KANEKO (J. Soc. Chem. Ind., Japan, 1931, 34, 135 B).—An equation is given relating the quantity of metal deposited with the distance between the electrodes *l*, the current density *I*, the current efficiency *ε*, the potential drop, *v*, at the cathode, and the resistance of the electrolyte *ρ*. The uniformity of a deposit is increased by increasing *l*, decreasing *ρ*, or increasing the value of *∂v/∂l*. H. F. GILLBE.

**Preparation of metal powders by electrolysis of fused salts. III. Tantalum.** F. H. DRIGGS and W. C. LILLIENDAHL (Ind. Eng. Chem., 1931, 23, 634—637; *cf.* B., 1931, 118).—Ta may be prepared by the electrolysis of a mixture of 700 g. KCl, 280 g. KF, 100 g. K<sub>2</sub>TaF<sub>7</sub>, and 100 g. Ta<sub>2</sub>O<sub>5</sub> in a Ni crucible with a current density of 90 amp./sq. dm. The KCl causes the metal to have a coarse grain and markedly increases the current efficiency. The cathode deposit

of metal and solidified salt is treated with  $H_2O$  to remove the latter and the metal ground and boiled with a  $H_2SO_4$ - $HNO_3$  mixture to remove impurities. The metal (of about 99.9% purity) is then pressed into bars and degassed by heat treatment *in vacuo*. If this is omitted it is brittle. It is also possible to plate Fe, Ni, or Mo cathodes with tantalum under suitable conditions, and such plating retains its lustre in the presence of all corrosive reagents except HF. C. IRWIN.

**Densities of molten cryolite.**—See VII. **Bright-gold lustre.**—See VIII.

#### PATENTS.

**[Flotation] concentration of mineral.** A. C. DAMAN (B.P. 349,108, 20.1.30. U.S., 3.6.29).—Ore delivered to a flotation apparatus is ground so that only a portion is freed from gangue, the tailings being separated and returned for regrinding. The pulp is delivered over an impeller to give intimate contact with air or gas introduced under pressure, and the cover is provided with openings to permit heavy sand and minerals to return from the flotation chamber into the region of the impeller. C. A. KING.

**Operation of blast furnaces.** E. BAUMGARTNER, Assee. of H. SCHMOLKA (B.P. 349,015, 11.2.30. Ger., 11.2.29).—The charge is made up with a flux, *e.g.*, limestone or dolomite, which has been calcined without losing its shape or mechanical strength. Such method may expel only 65–85% of the  $CO_2$  content, and the product should be charged in a freshly burned condition. C. A. KING.

**Furnaces [for pre-reduction etc. of oxide ores].** A. STANSFIELD (B.P. 346,874, 27.1.30).—A rectangular stack is provided with vertical partitions between which and the walls are supported hearths which slope alternately from front and back. Comminuted material, *e.g.*, ore, slides down the hearths in succession, being controlled by reciprocating rakes. Apertures are formed in the partitions and connecting passages in the side walls through which gases (usually of a heating and reducing nature) flow upwards in transverse zig-zags over the ore, though some hearths may be provided with muffle roofs so that the gases evolved from the ore may be collected separately. At the bottom the ore may be discharged to a cooler or to an electric furnace which fuses the material and provides the pre-treating gases as described in U.S.P. 1,748,805 (B., 1930, 672). B. M. VENABLES.

**Single or multi-stage retort furnace [for ore reduction].** A. MUSSO and W. P. DEPPE (B.P. 348,012, 26.11.29).—A rotary cylindrical furnace is heated by radiation from surface-combustion elements outside the shell. It is provided with fixed ends projecting inside, and with sealing means between the fixed and moving parts outside, the cylindrical part of the shell. An axial hollow shaft or girder serves to support a number of pipes through which gaseous products are withdrawn from different zones of the furnace, which may be operated under vac. Internal transverse partitions permit slight differences in vac. between different zones. B. M. VENABLES.

**Production of molten and refined metals from their crude ores.** A. E. WHITE. From DOHERTY RES. CO. (B.P. 349,625, 1.3.30).—A column of electrically conducting material is forced upwardly through a feed barrel to the centre of the hearth of a furnace, and the top of the column is used as an electrode to strike an arc with one or more electrodes through the walls of the furnace. The degree of heating is determined by the rate of feed of the column, which may also be caused to give contact with the second electrode to provide resistance-heating. C. A. KING.

**Furnaces for melting metals and the heat treatment of metal objects.** WELLMAN SMITH OWEN ENG. CORP., LTD., and H. W. SOWARD (B.P. 349,745, 1.5.30).—A furnace of the reversing regenerative type is provided with a central gas port with an air port on either side so directed that the air stream mixes with the gas. A third air port above the gas outlet has means for directing the air current either downwards into the flow from the gas port or away from this stream, *e.g.*, along the furnace arch. C. A. KING.

**Furnaces for annealing metals under a protective dome.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 348,859, 29.4.30. Ger., 1.5.29).—The bottom of a circular pit annealing furnace is provided with a centrally disposed depression and radial channels to allow circulation of heat. A draw rod passes through the dome and base plate on which the material is stacked. C. A. KING.

**Heat-treating continuous metal strips.** W. W. TRIGGS. From DURHAM DUPLEX RAZOR CO. (B.P. 348,050, 3.1.30).—Material such as safety razor-blade strip is heated by being continuously drawn through a furnace, then cooled and hardened throughout by being sandwiched between liquid-cooled tanks which are reciprocated transversely, the temper of the centre portion alone being drawn by contact with a heated fin. [Stat. ref.] B. M. VENABLES.

**Melting of metals in induction furnaces without iron cores.** M. TAMA, and HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 348,655, 15.2.30).—The induction coil is mounted so that it can be lowered in relation to the crucible. During melting the whole charge is surrounded by the coil, which is lowered when the charge is fluid, and a number of tappings are provided to use only the portion of the coil around the crucible. C. A. KING.

**Electric shaft furnace for treating metal scrap or ores.** J. HALÁSZ (B.P. 349,449, 17.2.30).—One electrode is built in the bottom of a shaft furnace, the other being an extension of the upper part of the shaft when scrap is treated. For smelting ore the upper tubular electrode is of smaller diam. than that of the shaft. A resistance layer of C, CaO, or slag with high m.p. is arranged at the bottom of the furnace. C. A. KING.

**Refining of metals in electric inductor furnaces.** G. H. CLAMER (B.P. 349,446, 13.2.30. U.S., 20.2.29).—A melted charge is introduced into the furnace, to the coils of which current of comparatively low frequency is applied so that the heat available is sufficient



only to maintain the molten condition, but by the known pinch effect the metal is caused to circulate. Additional heat is supplied by independent means, e.g., by arc discharge. A jet of air or other gas is directed on to the crown produced on the surface of the metal.

C. A. KING.

**Electrically heated salt-bath furnace [for metal articles].** A. G. E. HULTGREN (B.P. 348,907, 4.6.30).—The shape and arrangement of the furnace are such that the current passing between co-ordinated electrodes arranged in pairs does not traverse the space to be occupied by the immersed articles. The faces of the pair of electrodes turned toward each other should not be closer than half the width of the crucible, and may approach each other towards the bottom of the furnace.

C. A. KING.

**Reduction of iron ore.** A. MUSSO and W. P. DEPPE (B.P. 349,477, 21.11.29).—Fe ore is reduced by the fixed C of coal by heating the mixture below 950° in a gastight retort. The chamber is exhausted constantly by means of pumps and the final mixture of gangue and Fe, both in an unfused condition, is separated mechanically.

C. A. KING.

**Production of spongy iron.** F. KRUPP A.-G., FRIEDRICH-ALFRED-HÜTTE (B.P. 348,033, 5.2.30. Ger., 8.2.29).—A mixture of finely-divided Fe ore and sufficient (2%)  $\text{MgCl}_2$  or  $\text{Na}_2\text{SiO}_3$  solution to form a damp mass is rotated in a drum so as to break the mass up in balls, which are heated until they harden, screened into various sizes, and reduced in graded layers by means of CO or water-gas.

A. R. POWELL.

**Manufacture of iron having special magnetic properties.** J. Y. JOHNSON. From I. G. FARBERND. A.-G. (B.P. 347,304, 30.1.30).—Fe powder obtained by the decomposition of  $\text{Fe}(\text{CO})_5$  and containing approx. equal percentages of C and O is heated at 650° in  $\text{H}_2$  to remove C and O, and the sintered product is heated and compressed into ingots at 1100°, which are rolled in a series of steps with intermediate annealing in  $\text{H}_2$  at 800–850°. The resulting thin sheets have high initial and high max. permeabilities, a small coercive force, and a small hysteresis loss.

A. R. POWELL.

**Magnetic [iron-nickel] alloys.** CALLENDER'S CABLE & CONSTRUCTION CO., LTD., and S. BECKINSALE (B.P. 347,305, 30.1.30).—Alloys containing 65–80% Ni, 15–33% Fe, 0.05–10% Co, 0.05–15% Si, and 0.05–10% Mo are claimed. For the manufacture of wire the preferred composition is 75% Ni, 23.4% Fe, 0.5% Co, 0.1% Si, and 1% Mo, and of alloys for dust cores 72% Ni, 16% Fe, 7% Si, 2% Co, and 3% Mo. The alloys after working are annealed in a non-oxidising atm. at 875–925°, then slowly cooled to 625°, and quenched.

A. R. POWELL.

**Austenitic non-corrodible steels.** F. ATKINSON and T. HAGON (B.P. 348,586, 2.11.29).—The intercryst. disintegration of austenitic steels containing 10–25% Cr and 5–12% Ni is prevented by adding 0.5–6% Si and one or more of the metals, W, Mo, Cu, Ti, or V to spheroidise the carbides. The steels are annealed at 900° and quenched, then reheated at 500–900°. [Stat. ref.]

A. R. POWELL.

**Manufacture of stainless irons and steels.** F. ATKINSON and T. HAGON (B.P. 349,009, 11.1.30).—The alloys consist of Fe with 14–20% Cr, 0.1–2.5% Ni, 0.08–0.25% C, and 0.75–2% Cu; they are softer than Ni-steel and free from tendency to hot-shortness.

A. R. POWELL.

**[Inhibitor] for protecting [ferrous] metals from the attack of acids.** V. BERTLEFF (B.P. 348,596, 5.2.30. Addn. to B.P. 293,701; B., 1929, 176).—The inhibitor comprises the product obtained by sulphonating with conc.  $\text{H}_2\text{SO}_4$  or oleum a tar-oil base having a b.p. above 240°. Liquorice extract, saponin, or glycyrrhizic acid is also added to the pickling bath as foam-producing agent.

A. R. POWELL.

**Manufacture of steel alloys.** A. E. G. T. VON VEGESACK, and UDDEHOLMS AKTIEBOLAG (B.P. 349,326, 6.6.30. Addn. to B.P. 268,616; B., 1927, 448).—The steel contains 1.1–1.3% C, 10–16% Cr, and 0.75–1.3% Mn.

A. R. POWELL.

**Elimination of iron from inorganic materials.** VEREIN. STAHLWERKE A.-G. (B.P. 349,033, 15.2.30. Ger., 15.3.29. Addn. to B.P. 341,060; B., 1931, 296).—After reduction of the Fe compounds to metal, Cl is passed over the red-hot material to volatilise the Fe as  $\text{FeCl}_3$ .

A. R. POWELL.

**Heat treatment of copper ores etc. [for recovery of copper by flotation].** MINERALS SEPARATION, LTD., and T. J. TAPLIN (B.P. 348,024, 31.1.30).—Sulphide or mixed sulphide and oxidised ores of Cu are subjected to a sulphating roast at below 650° and the product is mixed with 2% of charcoal and 0.5% NaCl for 1–2 hr. at 650° in a rotating furnace free from oxidising gases. The Cu is thus reduced to metal and  $\text{Cu}_2\text{S}$ , which are recovered by oil-flotation. The process is applicable to the recovery of Cu from burnt pyrites.

A. R. POWELL.

**Improvement of copper-titanium alloys.** M. and H. LISSAUER, B. GRIESMANN, and W. KROLL (B.P. 349,142, 28.2.30. Ger., 28.2.29).—Cu alloys with a max. of 4% Ti and up to 10% of one or more of the elements Ni, Cr, Mn, Fe, Co, or Mo, or up to 5% of S, Mg, or Al, are quenched from 650–1000° and aged at 250–600°.

A. R. POWELL.

**[Copper-zinc-tin] alloy.** H. KANZ (B.P. 349,484, 25.1.30. Switz., 1.2.29).—An alloy having anti-frictional properties which are improved on heat treatment and containing 62% Cu, 30% Zn, 4% Sn, 2.5% Pb, and 1.5% Ni is claimed.

H. ROYAL-DAWSON.

**Smelting of material containing tin oxide.** "BERZELIUS" METALLHÜTTEN GES.M.B.H., M. G. FREISE, and H. MASCHMEYER (B.P. 349,313, 29.5.30).—The material is heated in a rotary furnace with finely-divided coke or coal and fluxes to form a pasty mass in which the globules of reduced Sn are suspended. The mass is broken up and the Sn recovered by chemical or electrochemical means or by gravity concentration. Alternatively, the charge is melted down in a shaft or reverberatory furnace to recover the Sn.

A. R. POWELL.

**Recovery of tin from scrap tinfoil etc.** J. W. HINCHLEY (B.P. 346,674, 14.1.30).—The detinning liquor comprises an alkaline solution of Na plumbite

containing Pb, NaOH, and  $H_2O$  in the ratio 1 : 23 : 19. The solution is made by dissolving  $Pb_2$  sponge in NaOH. solution which is agitated by a current of air or by dissolving  $Pb(OH)_2$  [from  $Pb(OAc)_2$  and NaOH] in NaOH. The Sn in the scrap replaces the Pb in solution and is subsequently recovered by precipitation with  $Ca(OH)_2$ , the resulting NaOH solution being used to redissolve the Pb sponge formed in the detinning operation and thus regenerate the detinning solution. A. R. POWELL.

**Refining of metals [lead] and alloys [lead-tin] of low m.p.** A. HENDERSON (B.P. 347,222, 24.1.30).—Crude Pb from the blast furnace is freed from Cu, Sb, etc. by filtering the molten metal at just above the m.p. If the amount of Sb present exceeds that required to combine with the Cu, further quantities of Cu are added or after the first filtration the metal may be heated with Al or Zn and again filtered. The process is applicable to the purification of Sn-Pb alloys containing Cu or Sb. A. R. POWELL.

**Preparation of solder.** J. D. FRY (B.P. 348,113, 6.2. and 23.10.30).—Molten solder is allowed to fall in a series of drops from a jet on to a cold, moving, metallic plate, or into a liquid, or into two liquids, of which one floats on the other, so that it solidifies in pellets with flattened bottoms. A. R. POWELL.

**[Zinc] alloy, particularly for bearing surfaces.** H. C. HALL (B.P. 349,378, 5.6.30).—An alloy of Zn with 3–8 (5)% Al, 0.1–1.8 (1.1)% Cu, 0.2% Ni, 0.05% Mn, and 0.05% Fe is claimed. A. R. POWELL.

**[Tungsten and/or molybdenum] alloys.** W. MÜLLER and OLGA, PRINZESSIN ZUR LIPPE (B.P. 348,641, 10.2.30).—A mixture of 55–70% Mo and/or W, 2% C, and 26–40% of one or more of the metals Fe, Mn, Co, Ni, or Cr (of which at least 20% is Cr) is melted for a short time with up to 3% of known hardening agents. The alloys are suitable for cutting and abrading tools. A. R. POWELL.

**Electrolytic cleaning of [iron or steel] wires, bands, etc. [prior to galvanising].** F. A. HERRMANN (B.P. 348,205, 14.2.30).—The wire (etc.) is passed continuously through a solution of  $H_2SO_4$  containing 35–250 g./litre wherein it is cleaned anodically with a c.d. exceeding 20 amp./sq. dm., so that the metal becomes passive and the  $O_2$  bubbles cause the scale to become detached. A. R. POWELL.

**Electroplating process [for nickel-chromium on iron or steel articles].** METALS PROTECTION CORP. (B.P. 349,747, 3.5.30. U.S., 10.5.29).—See U.S.P. 1,774,269; B., 1931, 303.

**Furnaces for wire heating.** H. B. BLYTHE (B.P. 349,671, 10.3.30).

**Casting of metals [of high m.p.].** C. PIEL (B.P. 349,863, 3.9.30. Ger., 7.9.29).

**Welding [machine].** E. G. BUDD MANUFG. CO., Assees. of A. F. HANSON (B.P. 350,292, 14.7.30. U.S., 8.8.29).

**Separator [for foundry sand].** Mixing of moulding substances. Hardness tester.—See I. Roofing elements.—See IX. Rubberising surfaces.—See XIV.

## XI.—ELECTROTECHNICS.

**Welded zig-zag windings and refractory shapes for high-temperature [electric] furnaces.** L. NAVIAS (J. Amer. Ceram. Soc., 1931, 14, 365–375).—New methods of construction of the furnace tubes both for external and internal windings are described. Mo windings on alundum were used up to  $1650^\circ$  and W windings on MgO tubes up to  $2000^\circ$ , the windings being protected from oxidation by  $H_2$ . W is too brittle to be spirally wound; a zig-zag winding with welded joints should be employed. J. A. SUGDEN.

**Dust removal from industrial gases by the Cottrell process of electrical precipitation.** C. W. HEDBERG (Proc. Eng. Soc. W. Penn., 1931, 47, 63–106).—Cottrell plants for detarring coke-oven gas consist of a number of 6- or 8-in. pipes, 9 ft. long, in each of which hangs a weighted rod. The gas passes down outside the pipes and up through them. The power consumption is 5–8 kw.-hr. per  $10^6$  cu. ft. of gas. They are installed after the primary coolers. Precipitators for the removal of "fly-ash" from powdered-fuel steam plants are usually placed between the fan and the stack. Large units are employed of two sections in series each with 8–15 ducts with wire discharge electrodes separated by reinforced concrete slabs. The power consumption is about 5 kw.-hr. per  $10^6$  cu. ft. of gas cleaned; a single precipitator can deal with the gases corresponding to a steam output of 500,000 lb./hr. The primary cleaning of blast-furnace gas leaves a dust content of 0.1–0.5 grain per cu. ft. Such gas can be used as steel mill fuel if the content is reduced to 0.01 grain per cu. ft., which is possible by the Cottrell process; it may then be used also for gas engines. The process is attractive in cases where water is scarce or difficulties occur with effluents. In working such plants it is desirable to add 25–30 lb. of coal to the furnace per ton of Fe, as the hydrocarbon vapours prevent "back ionisation." Single-unit precipitators of the powdered-fuel type are used, and the temp. should not much exceed  $280^\circ$  or insulator failures become frequent. Such hot dry-cleaning may save 5% of the total heat in the gas. Where cooling is permissible, as when the gas is required for gas engines, initial spray-cooling followed by electrical precipitation is a cheaper process. Plate-and-wire or pipe precipitators are then used. Another lay-out uses two precipitators in series, each preceded by a cooler. C. IRWIN.

**Refining of cracked spirit.**—See II. Igneous electrolysis.—See VII. Metal powders from salts. Electroplating solutions.—See X. Nutrients in soil.—See XVI.

### PATENTS.

**Heating grids for electric-resistance furnaces.** W. S. SMITH and C. V. H. GARNETT (B.P. 349,138, 28.2.30).—Bars triangular in cross-section are arranged so that two sides of the triangle face the area to be heated. J. S. G. THOMAS.

**Production of insulating materials.** ELECTRICAL RES. PRODUCTS, INC., and F. C. TOMLINS (B.P. 348,213, 14.2.30. Addn. to B.P. 307,966; B., 1929, 401).—Distilled  $H_2O$  is employed for the washing and heat-treatment processes already referred to. J. S. G. THOMAS.

**Insulating compounds.** ELECTRICAL RESEARCH PRODUCTS, INC., Assees. of A. R. KEMP (B.P. 348,192, 10.2.30. U.S., 2.3.29).—Compounds resulting from the exothermic reaction of rubber and/or balata with a sulphonic compound containing the group  $R\cdot SO_2\cdot X$ , in which R represents an org. radical or OH, and X represents OH or Cl, *e.g.*, *p*-phenolsulphonic acid, to which, if necessary, an antioxidant, *e.g.*, a mixture of acetaldol and  $\alpha$ -naphthylamine, may be added, are claimed. J. S. G. THOMAS.

**Electrical insulation [for cables etc.].** I. G. FARBENIND. A.-G. (B.P. 348,129, 2.1.30. Ger., 13.2.29).—Insulating material comprising a mixed fatty acid ester of cellulose, one radical of which is preferably Ac, is claimed. Thus, *e.g.*, a solution of cellulose acetate-butyrate in  $COMe_2$  is cast and cut into tapes. J. S. G. THOMAS.

**[Active mass for cathodes of] electric accumulators.** I. G. FARBENIND. A.-G. (B.P. 348,421, 5.6.30. Ger., 6.6.29).—A mixture of at least 20% of finely-powdered, highly active Fe with  $Cd(OH)_2$  and, if desired, about 7% of glycerin is used. J. S. G. THOMAS.

**Sealing compositions for electrical condensers.** BAKELITE CORP., Assees. of E. R. HANSON and M. E. DELANEY (B.P. 349,116, 14.2.30. U.S., 15.2.29).—Compositions composed of 3–9 pts. of a chlorinated  $C_{10}H_8$ , *e.g.*, tri- or tetra-chloronaphthalene, and 1 pt. of gilsonite are claimed. J. S. G. THOMAS.

**Electric-discharge lamps.** GEN. ELECTRIC CO., LTD., J. W. RYDE, and H. G. JENKINS (B.P. 349,261, 30.4.30).—The form, position, and/or intensity of the luminous column of an electric-discharge lamp is caused to vary continually by introducing into the lamp a continuous stream of an impurity, *e.g.*,  $C_{10}H_8$  vapour, which is cleaned up by the discharge. J. S. G. THOMAS.

**Metal-vapour [electric-discharge] lamps.** H. STAMMERREICH (B.P. 348,677, 11.2.30).—Lamps having at least one electrode composed of a liquid alloy, *e.g.*, Zn amalgam, are operated so that, during operation, separation of the constituents of the alloy is effected, and those constituents of the electrode not desired in the formation of the arc are removed, *e.g.*, by centrifugal force, from the arc space. J. S. G. THOMAS.

**Firedamp-proof electric incandescence lamp.** L. MELLERSH-JACKSON. From SIEMENS-SCHUCKERT-WERKE A.-G. (B.P. 348,670, 16.12.29).—The space between the lamp bulb and a surrounding glass globe is filled with a gas or vapour, *e.g.*,  $O_2$  or  $CCl_4$ , which, on fracture of the lamp during operation, accelerates destruction of the lamp filament and extinguishes the incandescence. [Stat. ref.] J. S. G. THOMAS.

**Cements for capping electric lamps etc.** GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 349,244, 15.4.30. Ger., 24.4.29).—A cement comprising one or more esters of high b.p., more especially phosphoric esters of homologous phenols, *e.g.*, phenyl and tolyl phosphates, in addition to the usual resins, solvents, and fillers is claimed. J. S. G. THOMAS.

**Manufacture of coloured electric-light bulbs.** IMPERIAL CHEM. INDUSTRIES, LTD., and E. A. BEVAN (B.P. 349,160, 6.3.30).—Coloured lacquers comprising synthetic resin compositions of "glyptal" and  $CH_2O$ -urea or -thiourea and dyes are applied to the bulbs by dipping, and are hardened by the normal heat of the lamp. S. S. WOOLF.

**Coating of [metallic] filamentary material [for use as electron emitters].** STANDARD TELEPHONES & CABLES, LTD. From WESTERN ELECTRIC CO., INC. (B.P. 348,176, 5.2.30).—The filament, *e.g.*, of Pt-Ni alloy, is continually passed through coating, baking, and flashing units, first in one direction and then in the other, so that successive coatings may be applied. F. R. ENNOS.

**Determining the intensity of colours.** J. RAZEK and P. J. MULDER (B.P. 349,565, 28.12.29).—Photoelectric currents produced by the incidence of successive parts of the spectrum of the radiation from a source are amplified by a thermionic device, including a network with a second thermionic device for compensating fluctuations of current supply to the amplifier. J. S. G. THOMAS.

**Manufacture of recording media for rendering visible signs or symbols in electrochemical facsimile and picture-recording apparatus.** V. BAUSCH, JUN., T. BAUSCH, V. BAUSCH, SEN., and F. BAUSCH (F. SCHÖLLER & BAUSCH) (B.P. 349,189, 15.3.30. Ger., 16.3.29).—Paper impregnated with the iodide of a multivalent element, *e.g.*,  $CdI_2$ , is used. J. S. G. THOMAS.

**Photoelectric devices.** KOLSTER-BRANDES, LTD. From KOLSTER RADIO CORP. (B.P. 350,075, 18.3.30).

**Thermionic cathodes for electric-discharge tubes.** BRIT. THOMSON-HOUSTON CO., LTD., and L. J. DAVIES (B.P. 348,679, 11.2.30).

**Introduction of gas into sealed vessels such as those of electric-discharge devices.** GEN. ELECTRIC CO., LTD., J. W. RYDE, and N. L. HARRIS (B.P. 350,069, 17.3.30).

**[Heating of solid cathodes in] mercury-vapour rectifiers.** GRAMOPHONE CO., LTD., and R. B. MORGAN (B.P. 350,070, 17.3.30).

**Manufacture of electron-emitting cathodes for multiple-electrode thermionic valves.** LOEWE AUDION GES.M.B.H., and B. WIENECKE (B.P. 349,667, 8.3.30. Ger., 8.3.29).

**Heat accumulators.**—See I. Insulating materials.—See III. Cl.—See VII. Roofing elements.—See IX. Production of molten and refined metals. Furnace for metal scrap etc. Salt-bath furnace. Magnetic Fe and Fe-Ni alloys. Cleaning of wires etc.—See X. Rubber for insulation.—See XIV. Tobacco.—See XX. Testing for CO in air. Treatment of water.—See XXIII.

## XII.—FATS; OILS; WAXES.

**Effect of light on the oxidation of fat.** C. H. LEA (Proc. Roy. Soc., 1931, 108 B, 175–189).—A new test for rancidity is described in which the oil or fat is heated, in an atm. of  $N_2$ , with a solvent mixture of

AcOH and  $\text{CHCl}_3$  in the presence of solid KI. After cooling, the reaction mixture is added to 5% KI solution and titrated with 0.002N- $\text{Na}_2\text{S}_2\text{O}_3$ . The result so obtained measures the active O originally present in the fat. A quantitative modification of the Kreis reaction is described also. Oxidation of fats is accelerated sensibly by weak artificial light, whilst exposure for a few min. to direct sunlight is sufficient to produce rancidity. The reaction on exposure to light is autocatalytic, subsequent oxidation of the fat being accelerated by a previous brief exposure. The differences in susceptibility between fats cannot be attributed to any of the non-fatty substances present, but are probably due to differences in the chemical nature of the fats. Bleaching of the yellow pigment of beef fat occurs at a comparatively early stage in the oxidation process. The intensity of the Kreis test does not run strictly parallel with the active O content. Increase in active O following exposure to light was associated with, at most, a very small increase in free acidity. W. O. KERMACK.

**Free fat in soap and its determination.** S. KAWAI and R. TONOIKE (J. Soc. Chem. Ind., Japan, 1931, 34, 153—154 B).—The free fat in toilet soaps (about 0.02—0.09%) is extracted with light petroleum from an aq. EtOH solution of the soap. The extract is saponified, freed from unsaponifiable matter, and recovered for weighing as fatty acids. The fatty acids from the free fat had neutralisation val. about 140 and I val. about 50—80, the corresponding figures for the combined fatty acids being 204—210 and 36—38.

E. LEWKOWITSCH.

**Determination of naphthasulphonic acid soaps in soap-mineral oil mixtures.** W. SCHAEFER (Chem. Umschau, 1931, 38, 131—132).—After saponification and extraction of the unsaponifiable matter (modified Spitz-Hönig method), the EtOH is distilled off from the soap solution, which is acidified with dil.  $\text{H}_2\text{SO}_4$  and extracted with light petroleum. The extract, after repeated washing with 50% MeOH, yields the fatty, naphthenic, and resin acids. The alcoholic washings are evaporated, dissolved in  $\text{H}_2\text{O}$ , acidified with conc. HCl, and extracted with  $\text{Et}_2\text{O}$ . The  $\text{Et}_2\text{O}$  solution is washed with 20%  $\text{Na}_2\text{SO}_4$  solution, dried, and on evaporation yields the naphthasulphonic acids as soaps. The soaps have a small acid val. (about 10), possibly due in part to traces of naphthenic acids.

E. LEWKOWITSCH.

**Germicidal assay of soaps.** H. C. HAMILTON and F. THISTLETHWAITE (J. Lab. Clin. Med., 1931, 16, 391—396).—In tests employing the skin of guinea pigs toilet and washing soaps were ineffective, whilst a soap base containing  $\text{HgI}_2$  (1%) was effective.

CHEMICAL ABSTRACTS.

**Influence of anti-oxidants on the rate of oxidation of linseed oil. II. Phenols and aromatic amines.** A. M. WAGNER and J. C. BRIER (Ind. Eng. Chem., 1931, 23, 662—666; cf. B., 1931, 401).—Pyrogallol (70),  $\alpha$ -naphthol (40), resorcinol (4), compared with quinol (100), represents the order of efficiency of the phenols in prolonging the induction period of the oxidation of linseed oil at 100°.  $\beta$ -Naphthol, diphenylamine,  $\alpha$ -naphthylamine, and phenyl- $\beta$ -naphthylamine have no anti-oxidant effect at 100°. *m*- and *p*-Phenylene-

diamines are slightly more effective at 100° than is quinol, and, unlike the latter, can interrupt oxidation that has already set in; they have no anti-oxidant action at 30°, and it appears that decomposition products of these diamines are the actual retarding agents at the higher temp.

E. LEWKOWITSCH.

**Substitution of steam for carbon dioxide in the hydrogenation of oils.** V. YASHCHENKO (Masloboino Zhir. Delo, 1929, No. 5, 23—24).—Before removing the catalyst, steam at 120—140° is passed through the vessel.

CHEMICAL ABSTRACTS.

**New compounds produced during the hydrogenation of fish oils.** S. UENO and R. YAMASAKI (J. Soc. Chem. Ind., Japan, 1931, 34, 151—152 B; cf. B., 1931, 499).— $\text{C}_{10}$ ,  $\text{C}_{12}$ ,  $\text{C}_{14}$ , and  $\text{C}_{16}$ -aldehydes, apparently of the isoaliphatic series (which are the cause of the odour of hardened fish oils) were isolated from the unsaponifiable fraction of the malodorous volatile products of hydrogenation.

E. LEWKOWITSCH.

**The sulphur monochloride reaction of fatty oils. V. Reaction velocity and viscosity of sulphochlorinated oils.** E. H. HARVEY and H. A. SCHUETTE (Ind. Eng. Chem., 1931, 23, 675—676).—Neither the velocity of the thermal reaction of fatty oils treated with  $\text{S}_2\text{Cl}_2$  (6.7%; cf. B., 1928, 935) nor the viscosities of the products bear any consistent relation to the I val. of the original oils. The rate of temp. rise is exceptionally great for menhaden oil. The viscosities of the products plotted against the viscosities of the original oils give a smooth curve.

E. LEWKOWITSCH.

**Composition of the oil from *Soja hospida*.** A. HEIDUSCHKA and H. EGER (Chem. Umschau, 1931, 38, 129—130).—Oils extracted from the same sample of soya beans by light petroleum in the laboratory and in the factory did not differ essentially. The fatty acid composition (%) of the oil (I val. 132.6), determined by the Pb salt- $\text{Et}_2\text{O}$  separation and bromination experiments, was: palmitic 13.31,  $\alpha$ -linolenic 2.7,  $\alpha$ -linoleic 19.51,  $\beta$ -linoleic 33.58, oleic 24.18. No stearic acid could be detected.

E. LEWKOWITSCH.

**Jack pine.**—See XIII. Fruit-tree emulsions.—See XVI.

PATENTS.

**Sulphonation of higher fatty acids and fatty acid derivatives.** N.V. CHEM. FABR. "SERVO," and M. D. ROZENBROEK (B.P. 347,592 and 349,527, 21.1.30. Holl., 22.1.29).—(A) A higher proportion of  $\text{H}_2\text{SO}_4$  can be organically combined by sulphonating the higher fatty acids (above  $\text{C}_{12}$ ) etc. in the presence of aliphatic sulphohydroxy-acids, e.g., isethionic acid, their salts, or sulphuric esters. (Cf. B.P. 312,283; B., 1931, 71.) (B) The fatty acids are sulphonated in the presence of mixed anhydrides of AcOH (or its homologues) with inorg. oxy-acids (except  $\text{H}_2\text{SO}_4$ ), e.g.,  $\text{H}_3\text{BO}_3$ , oxy-acids of P (which may still retain one or more OH groups).

E. LEWKOWITSCH.

**Manufacture of soap.** HENKEL & Co. G.M.B.H. (B.P. 348,689, 14.2.30. Ger., 30.3.29. Cf. B.P. 301,020; B., 1929, 825).—Liquid potash soaps to which K acetate or lactate has been added remain liquid down to -8° and do not discolour with age. [Stat. ref.]

E. LEWKOWITSCH.

**Soaps for laundry and other purposes.** M. SIFF [ORIENT SOAP WORKS (LONDON)] and H. SWITHEBANK (B.P. 349,003, 15.4.31).—A blueing or whitening soap containing up to 2% of alkali-blue or sol.-blue and sufficient alkali, *e.g.*, Na silicate, is colourless, but yields a blue liquor on dissolution. E. LEWKOWITSCH.

**Thickening of vegetable [castor] oil.** W. W. TRIGGS. From M. MORGAN (B.P. 349,684, 18.3.30).—Castor oil (80 pts.) is thickened or solidified by heating to about 47.5° and stirring in dropwise 1 pt. of HNO<sub>3</sub> (d 1.4—1.42) and then raising the temp. to about 116° until "crackling" ceases. E. LEWKOWITSCH.

**Artificial turf.**—See XVI.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Relation between thixotropy and levelling characteristics of paint.** E. L. McMILLEN (Ind. Eng. Chem., 1931, 23, 676—679).—The Kampf rotation viscosimeter has been modified so that consistency measurements can be made at shearing stresses below 1 dyne/sq. cm. and rates of shear as low as 10<sup>-6</sup> sec.<sup>-1</sup> Waring's discussion (Soc. of Rheology, Dec., 1930) of levelling properties neglects the variation in yield val. after brushing-out due to the thixotropic nature of paint. The fluidity of all paints immediately after brushing-out or stirring is high and approx. of the same order, but decreases very rapidly (< 2 min.) after stirring has ceased. For good levelling, a paint must be formulated so that this regain of plasticity after brushing is slow enough to permit elimination of brush marks, but sufficiently rapid to prevent sag on vertical surfaces.

E. LEWKOWITSCH.

**Effect of building materials on paint films.** H. M. LLEWELLYN (Dept. Sci. Ind. Res., Building Res., 1930, Bull. No. 11, 4 pp.).—The chief factor which affects the soundness of a paint film on building materials is the moisture content of the material at the time of painting, as, in addition to the weaker bond on a damp surface, hydrostatic pressure may be set up in various ways behind the impermeable film. Sol. salts may cause similar effects by efflorescence, and drying oils, *e.g.*, linseed oil, are attacked by caustic alkalis to form sol. soaps. Oil paints are liable to failure by saponification when applied to damp surfaces containing free CaO and salts of Na and K, *e.g.*, Portland cement, asbestos cement, etc. Prussian blue, chrome, and many org. pigments are sensitive also to the same reaction.

C. A. KING.

**Viscosity research into protective [nitrocellulose] coatings.** A. W. VAN HEUCKEROOTH and J. R. STEWART (J. Rheology, 1931, 2, 223—229).—Various viscosity cups for the rapid testing of paints, lacquers, and paint oils are described and compared, the Pratt-Lambert type being recommended for the factory and the Gardner mobilometer for the laboratory. A chart is shown for the determination of the viscosity of a blend of nitrocelluloses of different known viscosities. The tensile strengths of nitrocellulose films prepared from AcOBu solutions increase in approx. proportion with the resistance to exposure (roof test) and with the viscosity (except for very high viscosities, when both these properties decrease).

J. GRANT.

**Resins of jack pine.** J. B. PHILLIPS (Pulp Paper Mag. Can., 1931, 31, 211—219).—The resins consist chiefly of resin acids and fatty and unsaponifiable matter. The fatty constituents consist largely of acids and glycerides of the unsaturated type (oleic, linoleic, and traces of linolenic acids). The fats of seasoned wood contain much oxidised fatty acid.

CHEMICAL ABSTRACTS.

**Dispersions of the quicksand type.**—See I.  
**Resins in podophyllum rhizome.**—See XX.

### PATENTS.

**Production of [primer] coatings.** BRIT. CELANESE, LTD. (B.P. 348,266, 5.3.30. U.S., 5.3.29).—The surface is "filled" with powder or pasty primers containing over 50% of a cellulose derivative and/or plasticiser, and is then given a coat of a cellulose composition.

E. LEWKOWITSCH.

**Preparation of [cellulosic] coating materials [floor coverings, plastic masses, etc.].** IMPERIAL CHEM. INDUSTRIES, LTD., and B. W. FOSTER (B.P. 348,156, 4.11.29. Cf. B.P. 331,837; B., 1930, 872).—H<sub>2</sub>O-wet cellulose esters or ethers (*e.g.*, nitrocellulose) are incorporated with a non-volatile combined gelatiniser and plasticiser (*e.g.*, Bu phthalate, camphor oils, etc. or *p*-toluenesulphonamide and castor oil) and, if desired, a softener, in quantities such that the wt. of the plasticiser and softener together does not greatly exceed the dry wt. of the ester or ether; the H<sub>2</sub>O which separates is poured off, and the remainder may be removed by heating. Inert fillers, pigments, etc. may also be added.

L. A. COLES.

**Synthetic resins and coating compositions prepared therefrom.** BAKELITE CORP., Assees. of V. H. TURKINGTON (B.P. 349,522, 27.2.30. U.S., 28.2.29).—The product obtained by reaction between a phenol and a fatty oil (*e.g.*, cresol 100 pts., tung oil 100 pts.) in the presence of hexamethylenetetramine (10—25 pts.) (with or without subsequent heating with, *e.g.*, paraformaldehyde) is sol. in turpentine and petroleum distillates, and with driers dries to a clear resistant film at room temp.

E. LEWKOWITSCH.

**Production of synthetic resins and coating compositions made therefrom.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,442, 23.1.30. U.S., 23.1.29).—Furylethylene is polymerised by heating for at least 3 hr. at 180° with an oxidising or acid catalyst, *e.g.*, Bz<sub>2</sub>O<sub>2</sub>, H<sub>2</sub>O<sub>2</sub>, KClO<sub>4</sub>, O<sub>3</sub>, linseed oil acids, etc., and solvents, *e.g.*, PhMe, tolyl phosphate; nitrocellulose and solvents therefor may be incorporated.

S. S. WOOLF.

**Coating and impregnating compositions.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. BAIRD (B.P. 349,464, 24.2.30).—Aq. emulsions of "glyptal-type" resins dissolved, if desired, in org. solvents are prepared prior to or simultaneously with their incorporation with aq. solutions or dispersions of rapid thermohardening resin, *e.g.*, PhOH- or urea-CH<sub>2</sub>O resins.

S. S. WOOLF.

**Application of synthetic resins to surfaces.** BRIT. THOMSON-HOUSTON Co., LTD. Assees. of J. G. E. WRIGHT (B.P. 349,399, 9.9.30. U.S., 10.9.29).—Surfaces

are coated with an "alkyd" resin mainly in the "B stage," together with a small amount in the "A stage," in the form of a suspension in an inert volatile liquid, *e.g.*,  $\text{CCl}_4$ , pigment being present if desired; the film is "cured" *in situ* at 125–180° for about 5 hr.

S. S. WOOLF.

**[Synthetic-resin] varnishes etc.** BAKELITE CORP., Assees. of H. L. BENDER (B.P. 349,599, 22.2.30. U.S., 23.2.29).—Resinoids, *e.g.*,  $\text{PhOH}$ - or  $\text{PhOH}$ -oil- $\text{CH}_2\text{O}$ , urea- or thiourea- $\text{CH}_2\text{O}$ , in the A condition are advanced towards the C condition, *e.g.*, by heating with a high-boiling A-resinoid solvent which is a non-solvent for C-resinoids, *e.g.*,  $\text{BuOH}$ , giving a colloidal dispersion of the advanced resinoid having low penetrative power for test paper. Alternatively, the A-resin is heated in the absence of solvent, the latter being incorporated at a later stage to break down the gel structure. Air-drying varnishes needing no further heat-curing are thus obtained.

S. S. WOOLF.

**Mouldable compositions.** BAKELITE CORP., Assees. of H. L. BENDER (B.P. 349,521, 27.2.30. U.S., 28.2.29).—Fibrous fillers or sheet materials are impregnated with a reactive phenolic resinoid comprising residual amounts of a  $\text{CH}_2$ -containing agent (*e.g.*,  $\text{CH}_2\text{O}$  etc.) and a non-phenolic ingredient to combine therewith, *e.g.*, urea.

S. S. WOOLF.

**Manufacture of plastic substances.** DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 348,593, 14.1.30. Ger., 2.3.29).—Durable elastic materials are prepared by condensing azelaic acid with glycerol by heating at about 160–180° with or without the aid of condensing agents.

E. LEWKOWITSCH.

**Manufacture of artificial [plastic] masses.** I. G. FARBENIND. A.-G. (B.P. 348,077, 6.2.30. Addn. to B.P. 304,612; B., 1930, 677).—Synthetic rubber, *e.g.*, from dimethylbutadiene, is used in conjunction with linoxyn (and nitrocellulose, plasticisers, fillers, resins, etc.) in linoleum cement.

E. LEWKOWITSCH.

**Softeners for plastic materials.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 349,100, 21.11.29).—Compositions comprising a vinyl ester polymeride as sole binding agent, an ester of phthalic acid, *e.g.*, the  $\text{Bu}_2$  or  $\text{Bu}$  cyclohexyl ester, as softener, solvents, and pigments are claimed.

S. S. WOOLF.

**Coating and plastic materials.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 349,562, 21.11.29).—Mixtures of vinyl ester polymerides and as softener therefor an aryl ether of a polyalcohol in which the ratio ether group : OH group is not less than 2 : 1, *e.g.*, di- or tri-benzylin, together with a suitable solvent mixture and, if desired, pigments, are claimed as non-brittle compositions.

S. S. WOOLF.

**Manufacture of resinous condensation products.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,420, 4.6.30. U.S., 4.6.29).—The product of the condensation of  $\text{NH}_4\text{CNS}$  with  $\text{CH}_2\text{O}$  in an acid, alkaline, or neutral medium is evaporated and hardened at 90–100°.

E. LEWKOWITSCH.

**Bonded materials.** BAKELITE CORP., Assees. of H. KLINE (B.P. 347,738, 28.1.30. U.S. 29.1.29).—Fibrous fillers and a reactive (phenolic) resinoid (with or without a suitable solvent) are consolidated by heating at 170°

under pressure (<500 lb./sq. in.) until the resin liquefies and then sets.

E. LEWKOWITSCH.

**Bonded articles [abrasive wheels] embodying synthetic resins.** BAKELITE CORP., Assees. of R. S. DANIELS (B.P. 347,577, 20.11.29. U.S., 24.11.28).—An abrasive (as filler) is incorporated and moulded with a glycerol (1 pt.)-phthalic anhydride (2 pts.) resinoid which has been prepared by heating to the point of incipient gelation.

E. LEWKOWITSCH.

**Production of variegations in plastic masses.** N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 349,670, 8.3.30. Holl., 19.11.29).

**Manufacture of [veined] ornamental articles from synthetic resin.** SYNTHAFORM FABR. F. FORMTEILE AUS SYNTHETISCHEN EDEIHARZEN G.M.B.H. (B.P. 349,753, 7.5.30. Ger., 17.12.29).

**Manufacture of [lacquered or painted] moulded articles.** R. WEITZEL (B.P. 348,216, 15.2.30. Fr., 22.2.29).

**Chloromethyl compounds.**—See III. **Azo pigments.**—See IV. **Coloured electric-light bulbs.**—See XI. **Pigments in rubber dispersions.**—See XIV. **[Painters'] size.**—See XVII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**X-Ray diffraction study of chicle.** C. W. STILLWELL (Ind. Eng. Chem., 1931, 23, 703–706).—The X-ray patterns for crude and refined chicle differ in that the former contains a few extra lines, indicating that in crude chicle the gutta is present as a cryst. constituent, but not in refined chicle. The operations of melting, filtration, and resolidification involved in the refining process presumably reduce the size of the gutta particles to such an extent that they no longer give a cryst. pattern. Crude chicle is a mixture of at least 3 cryst. constituents, *viz.*, the gutta, the resin, and the  $\text{C}_6\text{H}_6$ -insol. matter (mainly Ca oxalate) and at least one amorphous fraction which is sol. in  $\text{H}_2\text{O}$ . Chicle gutta is identical with the hydrocarbon of gutta-percha and balata. Unlike rubber and gutta-percha, the crystals in chicle do not assume a preferred orientation on stretching.

D. F. TWISS.

**Manganese in raw rubber.** R. O. BISHOP and K. C. SEKAR (Rubber Res. Inst. Malaya, 1931, 2, 239–245).— $\text{KMnO}_4$  is only a weak disinfectant, especially at low concentrations, and its presence in a certain proprietary fungicide is undesirable. If  $\text{KMnO}_4$  is added to latex sufficient Mn remains in the rubber after coagulation and washing to cause development of tackiness on accelerated ageing and to effect definite retardation of vulcanisation.

D. F. TWISS.

**Rate of deposition of [rubber] latex on porous moulds.** H. W. GREENUP (Ind. Eng. Chem., 1931, 23, 688–691).—Variation of pressure, rubber concentration, and temp. in the deposition of rubber from latex on alundum filtration thimbles of medium porosity is found to be of negligible effect relative to the influence of variation in  $p_H$ . At  $p_H$  6.1 aggregation of the rubber globules in the latex ensues with consequent marked increase in the rate of filtration and deposition.

D. F. TWISS.

**Occurrence of "air-cracking" in stretched rubber.** A. VAN ROSSEM and H. W. TALEN (Kautschuk, 1931, 7, 79—86, 115—117; also, in part, Proc. K. Akad. Wetensch. Amsterdam, 1931, 34, 450—453).—For examination of the relative tendency of rubber to develop cracks on exposure under different degrees of stretching it is convenient to use wedge-shaped pieces 1 mm. and 8 mm. thick, respectively, at the two ends, so that the necessary range of degrees of extension is obtained simultaneously in one sample. The largest cracks appear at 10—20% elongation, and occur not only on the side exposed to sunlight, but also on the shaded side; night exposure leads to a similar result. Instead of the evidently inappropriate, although customary term "sun-cracking," the name "air-cracking" is recommended. It is believed that minute proportions of  $O_3$  in the air are responsible. Mere formation of a surface skin of oxidised rubber is not responsible, because the same result is not produced by oxidation with air or  $O_3$  and subsequent stretching, but very dil.  $O_3$  gives the characteristic result with stretched rubber. It is possible that the cracking phenomenon is associated with the crystallisation of rubber in spite of the fact that X-rays reveal orientation only above 70% elongation. With increasing degree of vulcanisation the cracks become smaller and more numerous; compounding ingredients of various types, including accelerators and anti-oxidants, have no important influence, although softeners capable of "blooming" may retard the effect at very low concentrations of  $O_3$ . N oxides,  $SO_2$ , and Cl do not induce air-cracking, and the effect seems to be a characteristic of  $O_3$  and of rubber.

D. F. TWISS.

**Effect of storage on milled rubber.** C. M. CARSON (Ind. Eng. Chem., 1931, 23, 691—694).—Smoked sheet rubber after mastication and subsequent storage for 9 months shows a decided decrease in modulus, plasticity, and recovery. Masticated rubber "freezes" at below  $0^\circ$  and thaws at 15—25°. If placed under slight pressure for several months, "freezing" occurs even at room temp., and thawing results only at about  $50^\circ$ . The stored masticated rubber shows rougher extrusion than newly masticated material; it appears also to have lost some of its natural antioxidant and to contain a reduced proportion of fatty acid and  $COMe_2$ -sol. substances, but an increased proportion of constituents sol. in alcoholic KOH.

D. F. TWISS.

**Effect on vulcanised rubber compounds of immersion in boiling water.** K. J. SOULE (Ind. Eng. Chem., 1931, 23, 654—658).—The swelling of vulcanised rubber in boiling  $H_2O$  follows a similar course to that of swelling in a solvent, a rapid initial effect being observed which gradually decreases. In some samples absorption still continues after 10,000 hr. in boiling  $H_2O$ . Raw rubber absorbs  $H_2O$  to a much greater extent than does vulcanised rubber, but remains intact after 1000 hr. Some fillers reduce the absorptivity and others increase it; of 35 fillers C black was the most marked of the former class and ultramarine of the latter. Swelling in boiling  $H_2O$  does not vary in any simple manner with the hardness, but, in general, loss in resistance to cutting varies directly with the amount of  $H_2O$  absorbed.

D. F. TWISS.

**Recovery of rubber and cotton from unvulcanised tyre-ply scrap. Extraction process.** C. S. POWELL (Ind. Eng. Chem., 1931, 23, 701—703).—The acid process for the recovery of rubber from unvulcanised tyre-ply scrap not only destroys the cotton and removes a large proportion of the zinc oxide from the rubber, but also, since the advent of very active acidic accelerators, necessitates a pretreatment with caustic alkali to prevent vulcanisation during the acid treatment. Solvent processes have the advantage of recovering both rubber and cotton. A process is described in which the unvulcanised scrap is treated with  $C_6H_6$  for 2 hr. in a revolving cage lined with 30-mesh monel-wire screen. The solution is then drained away and the residual cotton washed by repeated agitation with fresh additions of solvent. The retained solvent is removed from the cotton by treating under reduced pressure with steam. The recovered cotton is clean and white, and contains < 1% of rubber. In addition to recovering the cotton, the cost of the solvent process is only 30% of that of the acid process. The rubber solution produced is most economically applied to the impregnation of fabric. It is important to avoid mixing scrap containing different org. accelerators.

D. F. TWISS.

**C black.**—See II. **Hevea buds.** **Spraying of rubber plants.**—See XVI.

## PATENTS.

**Manufacture of artificial rubber or rubber-like masses.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 349,499, 24.2.30).—In the polymerisation of emulsified butadiene and its homologues with or without olefinic benzene compounds, the presence of a small proportion of an org. compound containing Cl, e.g.,  $CCl_4$  or Na trichloroacetate, leads to an increased yield and a more plastic product.

D. F. TWISS.

**Manufacture of uniformly plastic rubber.** NAUGATUCK CHEM. CO., Assees. of W. A. GIBBONS (B.P. 349,280, 12.5.30. U.S., 24.5.29).—An artificial aq. dispersion of vulcanised rubber or reclaim is freed from metallic particles and other impurities, sprayed into a drying atm., and the dried material finally compacted in a press to a uniformly plastic mass.

D. F. TWISS.

**Dispersion of pigments in rubber dispersions.** GOODYEAR TIRE & RUBBER CO., Assees. of C. R. PARK (B.P. 348,174, 4.2.30. U.S., 14.5.29).—Pigments such as channel black or thermatomic black are exposed, during manufacture or subsequently, to the vapours of an oily material, e.g., pine oil, tung oil, or petroleum distillate. The ease of dispersion of the pigments in aq. or other suspensions of rubber, e.g., latex, is thereby enhanced.

D. F. TWISS.

**Rubber compounding.** BARRETT CO., Assees. of A. B. COWDERY and T. A. BULIFANT (B.P. 348,761, 10.3.30. U.S., 14.3.29).—Coal-tar oil ( $d^{38}_{20} 1.06$ —1.15, b.p. above  $200^\circ$ ) substantially free from cryst. material at  $25^\circ$  is used as a softening or dispersing agent in rubber compounding; it imparts high resistance to abrasion, great tensile strength, and good ageing qualities.

D. F. TWISS.



**Manufacture of vulcanised rubber.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 349,461, 22.2.30).—The condensation products of mercaptobenzthiazole, dithiocarbamates, and their analogues with reactive aralkyl halides, such as  $\text{CH}_2\text{PhCl}$  or nitrobenzyl chloride are applied as vulcanisation accelerators.

D. F. TWISS.

**Manufacture of rubber articles.** ANODE RUBBER CO., LTD., Assees. of E. B. NEWTON (B.P. 348,537, 12.2.30. U.S., 6.3.29).—Unvulcanised masticated rubber and unmasticated rubber obtained from latex may be caused to adhere firmly to one another, even before vulcanisation, by superposing the two layers after coagulation of the latex, but prior to its drying; the composite product is subsequently dried and vulcanised. A valve-patch of masticated rubber reinforced with fabric may thus be affixed to a coagulated wet motor tube formed by electrodeposition from latex.

D. F. TWISS.

**Manufacture of latex compositions.** GOODYEAR TIRE & RUBBER CO. (B.P. 349,273, 6.5.30. U.S., 27.7.29).—An adhesive cement for the manufacture of rubber articles is made by mixing rubber latex with small amounts of pine tar and of an oxidised pine oil.

D. F. TWISS.

**Production of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., and (A) W. G. GORHAM and G. W. TROBRIDGE, (B) E. W. MADGE (B.P. 349,062—3, 20.12.29).—Porous or micro-porous articles of vulcanised rubber such as battery separators and filtering diaphragms are produced by coagulating or gelling aq. dispersions of the material (A) in an assembly of moulds built up in nest form, and then vulcanising under such conditions that evaporation of the  $\text{H}_2\text{O}$  is prevented, or (B) upon suitable moulds or backing strata (provided with narrow grooves or reliefs which prevent distortion by syneresis and consequent shrinkage) and thereafter submitting them to heat-treatment.

D. F. TWISS.

**Production of rubber composition [for electrical insulation].** J. E. POLLAK. From W. B. WIEGAND (B.P. 348,299, 21.3.30).—The insulating properties of materials containing rubber are improved by the presence of up to 6% of C black of high adsorptive properties, introduced as such and not added in the form of reclaimed rubber. The mechanical qualities are also improved.

D. F. TWISS.

**Coating of solid surfaces with rubber or the like.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 348,303, 24.3. and 5.6.30).—Compositions suitable for bonding rubber to surfaces such as metal are produced by treating rubber, ketones (such as cyclohexanone), or mixtures thereof with  $\text{CH}_2\text{O}$  or its polymerides or derivatives, e.g., methylal, in the presence of a condensing agent such as  $\text{HCl}$ . The reaction may be effected in a suitable solvent. Gutta-percha or rubber may be introduced into the compositions subsequent to the chemical reaction.

D. F. TWISS.

**Production of finishes on rubber fabrics and rubber goods.** F. THOMPSON (B.P. 348,169, 9.1.30).—A frosted appearance or sheen is imparted to textile

fabrics and rubberised materials by applying globules (diam.  $> 1$  mm.) of light-reflecting or -refracting material, e.g., glass,  $\text{SiO}_2$ , or synthetic resin. [Stat. ref.]

D. F. TWISS.

**Preservation of rubber.** GOODYEAR TIRE & RUBBER CO., Assees. of A. M. CLIFFORD (B.P. 346,079, 4.2.30. U.S., 14.3.29).—Diaminotriarylmethanes, especially 4:4'-diaminotriphenylmethane, or the products obtained by heating these with amines (*o*-toluidine, *m*-phenylenediamine), are applied as anti-agers.

C. HOLLINS.

**Manufacture of rubber [with increased resistance to deterioration].** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,489, 8.9.30. U.S., 9.9.29).—Products of the formula  $\text{A}\cdot\text{R}\cdot\text{CO}\cdot\text{R}'\cdot\text{A}'$ , where A and A' represent  $\text{NH}_2$  groups and R and R' represent aryl radicals, e.g., 4:4'-tetramethyldiaminobenzophenone, are used as anti-oxidants.

D. F. TWISS.

**Manufacture of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,985, 12.9.30. U.S., 17.9.29).—Agents for retarding the deterioration of rubber are produced by allowing  $\text{C}_2\text{H}_2$  to react with amino-compounds such as  $\text{NH}_2\text{Ph}$ ; a catalyst such as  $\text{Cu}_2\text{Cl}_2$  or  $\text{HgCl}_2$  may be used, if desired.

D. F. TWISS.

**[Anti-ager] for manufacture of rubber and the like.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,371, 25.7.29).—The addition of  $\beta$ -naphthol to a rubber mixture does not affect the rate of vulcanisation, but the vulcanised product has enhanced resistance to deterioration and does not become substantially discoloured even on exposure to direct sunlight.

D. F. TWISS.

**Bonded abrasives.**—See VIII. **Insulating compounds.**—See XI. **Plastic masses.**—See XIII.

## XV.—LEATHER; GLUE.

**Hydration of animal skin by volume-change method.** IV. **Effect of various factors on the hydration of calfskin.** E. R. THEIS and F. T. BENTON (Ind. Eng. Chem., 1931, 23, 367—370; cf. B., 1930, 339).—It was found that the ability of calfskin to hydrate was reduced by post-mortem action, that the bacterial-enzymic lag period was reduced from 12 hr. in a fresh skin to  $\frac{3}{4}$  hr. after 48 hr., and that the hydration of calfskin in saturated  $\text{NaCl}$  solution was progressively diminished by post-mortem action. Hydration tests with different saturated solutions showed the following order of diminishing effect:  $\text{Ca}(\text{OH})_2$ ,  $\text{H}_2\text{O}$ ,  $\text{Ca}(\text{OH})_2$  + excess  $\text{CaO}$ , 0.05*N*- $\text{NaOH}$ ,  $\text{NaCl}$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{NaCl}$  + 5*M*- $\text{CaCl}_2$ ,  $\text{CaCl}_2$ ,  $\text{KCl}$ ,  $\text{MgSO}_4$ ,  $\text{LiCl}$ ,  $\text{NaCl}$  + 5%  $\text{NaCO}_3$ . Skins are found to be hydrated (and not dehydrated) by brine curing: it is the loosely-held  $\text{H}_2\text{O}$  in the skin which is diminished. A dry-cured calfskin was soaked in a 2%  $\text{NaCl}$  solution; max. hydration was attained in 15 hr., after which bacterial enzymic action set in. By soaking cured calfskins in  $\text{NaCl}$  solutions of concentration  $> 5\%$ , hydration is slower and degeneration retarded and delayed. Dry-cured calfskins should be soaked in a 10—15%  $\text{NaCl}$  solution for 24 hr. and then in  $\text{H}_2\text{O}$  for 24 hr., whereby degeneration is delayed until after 45 hr. Bacterial activity was shown to be delayed by all antiseptics except  $\text{C}_{10}\text{H}_8$ . Greater hydration was obtained by soaking calfskin in larger vols. of  $\text{H}_2\text{O}$ . The hydration of

calfskin was diminished by soaking in  $H_2O$  at higher temp. It is suggested that the hydration of skin could be measured by changes in its wt. while suspended in  $H_2O$ . D. WOODROFFE.

**Action of pancreatin on collagen in presence of lime and ammonium sulphate.** A. KÜNTZEL and O. DIETSCH (Collegium, 1931, 155—162).—The proteolytic effect of pancreatin on collagen is repressed by  $Ca(OH)_2$ . No proteolytic effect was observed after 6 hrs. treatment in saturated lime-water. The proteolytic effect of pancreatin on collagen was increased by increasing the amount of  $(NH_4)_2SO_4$  in the solution. The efficiency of pancreatin in bating will be greater, the greater is the amount of free  $CaO$  removed from the skins. A greater bating effect with pancreatin will be obtained on pelts which have been delimed than on those not delimed. The bating effect is not affected much by variations of  $p_H$  between 6 and 10.

D. WOODROFFE.

**Enzyme lime liquors [for hides and skins].** M. BERGMANN, M. LISSITZIN, and G. SCHUCK (Collegium, 1931, 132—136).—Hair-loosening tests were conducted with papain solutions at  $p_H$  5, 7, and 8, respectively. The hair on calfskin was loosened in the shortest time by 4 times its amount of a 0.2% papain solution in a citrate buffer solution at  $p_H$  5.0. Determination of the rates of dissolution of hide substance by phosphate and bisulphite buffers, by papain alone, and by each buffer, respectively, showed that the papain is activated by these buffers. The enzyme was inactivated by Zn salts (0.04%).

D. WOODROFFE.

**Sampling of leather for physical investigations.** C. H. SPIERS (J. Soc. Leather Trades' Chem., 1931, 15, 153—166, 206—226).—It is suggested that before proper methods for testing leather or studying the relation between various physical properties and other factors can be devised, the peculiarities of the leather as regards the properties under consideration must be systematically studied.

D. WOODROFFE.

**Iron and copper in vegetable tan liquors and tanning extracts, and absorption and deposition of iron and copper impurities during tannage.** M. P. BALFE and H. PHILLIPS (J. Soc. Leather Trades' Chem., 1931, 15, 226—235).—The amounts of Fe and Cu in tannin extracts are low. More than 0.01% Fe may occur in myrobalans, gambier, and sumac extracts and > 0.015% Cu is usually present in chestnut extracts, the latter due to the use of Cu autoclaves in their manufacture. Fe and Cu impurities in tan liquors are freely absorbed by hides and retained in the grain layers. Evidence was obtained that tan liquors are frequently contaminated by Fe during use. Serious Fe contamination is shown if the total sol. matter in tan liquors contains > 0.010% Fe, and may arise in the limeyard, or from the use of  $H_2SO_4$ . The Yoe colorimetric method of determining Fe and Cu is described and recommended.

D. WOODROFFE.

**Evaluation of vegetable tannin extracts.** W. VOGEL (Collegium, 1931, 191—200).—Tannin extracts should not be judged merely on their tannin contents, but sugars (because they form acid), proteins as bacterial food, inorganic salts (since they repress swelling), and

starchy, gummy, and resinous substances (which function as protective colloids peptising the tannins) should be determined in the non-tans. The insol. matter, which serves as filler for heavy leathers or may be stabilised for tanning purposes, was 0.6% by the official method of analysis, but 80% in a solution of an extract of  $d$  1.070. It attains a max. at this  $d$  with most extracts. The mineral salt content of an extract is indicated by the ash, which varies from 1.0% in an untreated quebracho extract to 5.7% in a sulphited quebracho extract. Tannin extracts can be divided into three groups according to their  $p_H$ : synthetic tans and sulphite-cellulose extracts, 2.0—3.4; pyrogallol tans, 3.2—4.0; pyrocatechol tans, 4.3—6.2. Analytical solutions of "Triumph" quebracho extract and valonia extract were not rendered turbid, as were untreated and "Crown" quebracho extracts, pine bark, chestnut, and oakwood extracts, on lowering their  $p_H$  by additions of acid. The determination of the flocculation point of more conc. solutions was difficult. The colour of solutions of the extract and that of leathers tanned with them should be measured. The increase in wt. of unchromed hide powder after treatment with different tannin extracts was (%): quebracho (Triumph), 90—98; quebracho (untreated), 85—95; oakwood, 80—90; chestnut, valonia, sulphited quebracho, 70—80.

D. WOODROFFE.

**Leather, sanitation, and colloid chemistry.** J. A. WILSON (J. Amer. Leather Chem. Assoc., 1931, 26, 297—312).—A lecture.

#### PATENTS.

**Manufacture of [gelatinous] plastic masses for printing rollers, hectographs, etc.** A.-G. F. CHEM. PROD. VORM. H. SCHEIDEMANDEL, and W. WACHTEL (B.P. 348,384, 14.5.30. Ger., 14.5.29).—Gelatin, preferably in pearl form, is soaked in cold glycerin previously diluted with the quantity of  $H_2O$  desired in the final product. Alternatively, the  $H_2O$  is added to the gelatin by soaking or is left in it by incomplete drying during its manufacture.

L. A. COLES.

**Preparations for leather.**—See VI.

#### XVI.—AGRICULTURE.

**Soils. X. Mode of fixation of ammonia in the soil.** J. CLARENS and H. MARGULIS (Bull. Soc. chim., 1931, [iv], 49, 508—514. Cf. B., 1928, 869; 1931, 690).—The fixation of  $NH_3$  by soil is not appreciably affected by  $CaCO_3$ .

R. CUTHILL.

**Sulphur oxidation in Alberta soils.** J. D. NEWTON (Sci. Agric., 1931, 11, 612—622).—In soils examined, Common Scab cannot be controlled by applications of S in economic quantities. Lawn grass is injured by amounts of S requisite to kill dandelions. Increased acidity in soil due to S treatment decreased the bacterial, but not the fungal, numbers.

A. G. POLLARD.

**Moorland cultivation and the agricultural use of moors in 1925—1929.** B. TACKE (Bied. Zentr., 1931, 60A, 129—145).—A comprehensive survey, with bibliography, of recent work concerning the general cultivation, manuring, cropping, etc. of moor soils.

A. G. POLLARD.

**Trustworthiness of the quinhydrone method for determining the  $p_H$  of soil.** G. HATOS (Mezög. Kutat., 1930, 3, 189—190; Chem. Zentr., 1931, i, 1664).—A freshly ignited Pt electrode which has been kept for 1—2 min. in distilled  $H_2O$  containing quinhydrone, when immersed in the mixture of soil suspension and quinhydrone, gives within 5 sec. a potential which is const. for at least 10—15 min.

A. A. ELDRIDGE.

**Potassium thiocyanate method for determining soil acidity.** Y. KAMOSHITA (J. Imp. Agric. Exp. Sta., Japan, 1930, 1, 85—88).—Treatment of soil with an alcoholic solution of KCNS produces a red colour if the soil is more acid than  $p_H$  6.88, and the depth of colour is proportional to the acidity of the soil. At the same  $p_H$  the colour increases with exchangeable acidity, since this is accompanied by increase in  $Al_2O_3$  and  $Fe_2O_3$  and decrease in  $CaO$  and  $MgO$  brought into exchange. Hence the exchangeable  $Fe^{+++}$  of soils is considered responsible for part of the exchange acidity.

CHEMICAL ABSTRACTS.

**Determination of the nutrient material in soil by electrodialysis.** J. DI GLERIA (Mezög. Kutat., 1930, 3, 185—188; Chem. Zentr., 1931, i, 1664).—The P and K values are greater than those obtained by Neubauer's method.  $SO_4^{--}$  and  $NO_3^-$  can be determined by electrodialysis.

A. A. ELDRIDGE.

**Micro-determination of silica in aqueous soil extracts.** A. NÉMEC, J. LANIK, and A. KOPPOVÁ (Z. anal. Chem., 1931, 83, 428—445).—The solution containing 0.05—0.7 mg.  $SiO_2$  per 100 c.c. is treated with 5 c.c. of 1% citric acid solution, 5 c.c. of 5%  $(NH_4)_2MoO_4$  solution containing 5% of  $H_2SO_4$ , and after 5 min. with 7 c.c. of 4%  $H_2SO_4$ , 2 c.c. of 1%  $NaVO_3$  solution, and 2 c.c. of  $Na_2S_2O_3$  solution. After 15 min. the liquid is neutralised with 35 c.c. of 10%  $Na_2CO_3$  solution containing one sixth its vol. of glycerol. Max. intensity of the blue colour develops after 2 hr.  $H_3PO_4$  gives a similar colour which rapidly fades and disappears entirely in 2 hr.; high results are obtained, however, when the solution contains  $> 0.01$  mg. Fe and  $> 0.1$  mg.  $P_2O_5$  per 100 c.c. Exact maintenance of the above conditions in standard and test solutions is necessary for correct results.

A. R. POWELL.

**Use and action of phosphates on chernozem.** M. A. EGOROV (Pochvoved., 1928, 23, No. 3—4, 5—28).—On the assumption that the Ca humates undergo double decomp. with sol. phosphates, soil was mixed with K, Na, or  $NH_4$  phosphate or hydrogen phosphate solutions and the mixture was filtered. The colour of the filtrate depends on both the cation and the anion.  $Na^+$  is the most active;  $H_2PO_4^-$  and  $HPO_4^{--}$  give a slight colour, whilst  $PO_4^{--}$  produces a deep colour. The intensity of the colour increased with increase in concentration and an increase in the time of shaking. The addition of  $CaCO_3$  gives a darker extract, but  $CaCO_3$  alone is inactive;  $FeCO_3$  behaves similarly. Dialysis experiments are described; a dark liquid was obtained when the soil was treated with  $NaH_2PO_4$  or (more slowly)  $Na_2HPO_4$ , the effect being attributed to formation of  $Ca(H_2PO_4)_2$  and  $CaHPO_4$ , respectively. Addition of  $FeSO_4$  or  $FeCl_3$  to extracts of  $Na_2HPO_4$  had no effect

on the mobility of the humus. The reactions in all cases depend on the formation of a sol. or insol. phosphate.

CHEMICAL ABSTRACTS.

**Calcium arsenate and unproductiveness in certain soils.** W. B. ALBERT and W. R. PADEN (Science, 1931, 73, 622).—The addition of large amounts of Ca arsenate to light sandy loam soils eventually interferes to a serious extent with the growth of cowpeas, oats, cotton, and various grasses. The As contents of seedlings grown on soils containing that element in varying amounts are recorded.

L. S. THEOBALD.

**Copper as an essential for plant growth.** A. L. SOMMER (Plant Physiol., 1931, 6, 339—345).—Tomato, flax, and sunflower plants made poor growth unless the nutrient contained small amounts of Cu.

A. G. POLLARD.

**Influence of the nutrient content of seeds on the determination of the nutrient content of soils ("b" in the effect law).** W. POHLENZ (Bot. Archiv., 1930, 31, 141—196).—In determining the growth curves of plants by Mitscherlich's method the P content of the seeds is of significance only when a nutrient-free medium (e.g., sand) is used, or in soils when the seeds are unusually large. In such cases the seed-P, being utilisable by the plants, should be included in the initial phosphate content ("b") of the soil. The S-form of the initial portion of plant-growth curves is confirmed. Beyond this initial stage the "effect factor" for P is approx. const. for all plants.

A. G. POLLARD.

**Determination of the lime and fertiliser requirement of soil.** J. CSIKY (Mezög. Kutat., 1930, 3, 250—261; Chem. Zentr., 1931, i, 1664).—Determinations of the  $p_H$  in aq. and in KCl suspension were made on Hungarian soils; in soils free from carbonate the hydrolytic acidity, and in those containing carbonate the  $CO_3^{--}$  content, were also determined.

A. A. ELDRIDGE.

**Absorption and utilisation of phosphoric acid by plants.** KLEBERGER (Ergeb. Agrik.-Chem., 1930, 2, 53—68; Bied. Zentr., 1931, 60A, 156—157).—The intake of P by rye seedlings was affected by top dressings of N, but not of K. In pot cultures the citrate-sol. P of Rhenania phosphate was superior to the citric acid-sol. P of basic slag. Superphosphate did not affect the acidity of acid or neutral soils. Manurial treatment definitely influenced the ash content, and in particular the P contents of the leaves, of potatoes. In field trials P manuring increased the sugar content and root:top ratio of sugar beet and also the starch content and tuber:haulm ratio of potatoes.

A. G. POLLARD.

**Effect of drying of soils on solubility of plant nutrients.** B. TACKE (Kulturtechniker, 1930, 6, 588—590; Bied. Zentr., 1931, 60A, 145—146).—In a criticism of Zunker's theory, the increased solubility of phosphates consequent on the drying of soils is ascribed to changes in the degree of dispersion, involving a reduction in adsorptive power.

A. G. POLLARD.

**Effect of nitrogen, potash, phosphate, and calcium on the tillering and yield of oats.** E. JUNG (Ernähr. Pflanze, 1931, 27, 225—228).—Applications of various fertilisers increased the tillering of oats in

comparison with unmanured controls, but differences resulting from the individual fertilisers were not very definite. The uniformity in the number of tillers per plant was greatest where a complete fertiliser or a potash fertiliser alone was used. The total yield of grain in fertilised soils was in the order: complete fertiliser > K only > P only > N only. The average yields of plants producing the same number of tillers followed the same order.

A. G. POLLARD.

**Comparative growth efficiency of two varieties of oats with increasing applications of nitrogen and varied soil-moisture content.** TORNAU and K. MEYER [with W. PRASSLER] (J. Landw., 1931, 79, 155—189).—A xerophytic variety of oats from soil treated with increasing proportions of N, and at varying moisture contents, was less injured by the heavier applications of N than a hygrophytic variety grown under similar conditions. The percentage utilisation of the added N increased with the soil-moisture content, the xerophyte utilising high N applications at low moisture contents better than the hygrophyte. With a sufficiency of moisture the intake and utilisation of N were higher in the hygrophyte. With increasing applications of N both the growth period and the max. yield increased. The dry-matter production of the hygrophyte exceeded that of the xerophyte only where the N supply was small. "Transpiration productivity" (dry matter produced per litre of  $H_2O$  transpired) was higher in the xerophyte under all conditions. Application of N fertilisers reduced transpiration. The N intake per unit  $H_2O$  absorbed was greater in the xerophyte, especially where the salt concentration was high.

A. G. POLLARD.

**Potash manuring of lucerne.** A. LOCHNER (Ernähr. Pflanze, 1931, 27, 222—225).—The efficient manuring of crop rotations including lucerne is discussed in the light of the heavy demand of this crop on the soil potash.

A. G. POLLARD.

**[Effect of] nitrogen fertilisers on cotton.** V. N. MANDRUGIN (Udobr. Urozhai, 1930, 2, 363—369).—The best results during the year of application were from  $(NH_4)_2SO_4$ , followed by  $KNO_3$ ,  $CaCN_2$ ,  $NH_4NO_3$ , and urea: and the best after-effects from  $NH_4NO_3$ , followed by  $(NH_4)_2SO_4$ ,  $KNO_3$ , urea, and  $CaCN_2$ . The highest total increase in yield for 2 yrs. was given by  $(NH_4)_2SO_4$ , followed by  $NH_4NO_3$ ,  $KNO_3$ ,  $CaCN_2$ , and urea. When the fertilisers were added during boll formation  $CaCN_2$  was preferable. Cotton takes up more N in the later than during the early stages of growth.

CHEMICAL ABSTRACTS.

**Relation of nitrate-nitrogen and nitrification to the growth of tobacco following timothy.** R. P. THOMAS (Wisconsin Agric. Exp. Sta. Res. Bull., 1930, No. 105, 28 pp.).—Poor growth and the prevalence of brown root rot in tobacco are associated with the presence in the soil of much cellulosic matter and a consequent low nitrate content in the early part of the season. Application of nitrate to soil after the appearance of root rot is of little value. For tobacco after timothy, applications of CaO, P, and a high proportion of nitrate or easily nitrifiable material are necessary.

A. G. POLLARD.

**Effects of certain fungicides on the viability of *Hevea* buds.** R. P. N. NAPPER (Rubber Res. Inst. Malaya, 1931, 2, 192—213).—Several well-known fungicides are examined as to their possible harmful effect on budwood of the *Hevea* rubber tree and the max. intensity of treatment without injury is indicated.

D. F. TWISS.

**Test spraying experiments on young rubber plants.** F. BEELEY (Rubber Res. Inst. Malaya, 1931, 2, 214—221).—A list of available spray liquids is given together with methods of prep. for such as are not proprietary. A single application of a spray mixture is sufficient to decide within 2 or 3 days whether or not the concentration is so high as to damage the leaves, but in order to ensure adequate control of leaf diseases it is advisable to give 2 or even 3 applications at intervals of 14 days.

D. F. TWISS.

**Fruit-tree oil emulsions.** S. JENČIČ [with B. BAJEC] (Kolloid-Z., 1931, 55, 212—228).—Analysis of the Jugoslavian fruit-tree products known as "arborin" and "karbokrimp" shows them to consist essentially of tar oils and similar products in resin soaps. The solubility, viscosity, sp. gr., stability, particle size, surface tension, and electrical conductivity of aq. emulsions of these products have been measured.

E. S. HEDGES.

**Factors modifying the toxicity of phenol.** W. S. EISENMENGER (Plant Physiol., 1931, 6, 325—332).—The toxicity of PhOH to freshly germinated soya-bean seedlings increases with the concentration of PhOH used. Addition of  $SiO_2$  gel to the PhOH solution reduced its toxicity, the effect decreasing proportionally as the concentration of PhOH increased. PhOH was partly adsorbed by the  $SiO_2$  gel. Mixed solutions of PhOH and  $Ca(NO_3)_2$  (0.006M total concentration) were more toxic than corresponding solutions of  $Ca(NO_3)_2$  alone. When the mol. proportion of  $Ca(NO_3)_2$  exceeded that of PhOH the toxic effect was less than that of the corresponding single solution PhOH.

A. G. POLLARD.

**Control of cereal rusts by Kolo [sulphur] dust.** W. C. BROADFOOT (Phytopath., 1931, 21, 347).—At least two dustings with colloidal S on wheat, one at the flowering period and one 5—9 days later, are necessary. Dusting of the flowers does not affect the setting of the seed. The protein in wheat from dusted plants was slightly lower than that of controls.

**Nutrients used for maintenance by growing dairy cattle.** T. W. GULLICKSON and C. H. ECKLES (J. Agric. Res., 1931, 42, 593—601).—Determinations of the maintenance requirement of immature dairy cattle by the live-wt. method indicate that when the revised net energy of lucerne hay (Eckles *et al.*, J. Dairy Sci., 1927, 10, 431) was used, values obtained were lower than the Armsby standard for weights below 350 lb., and higher for greater weights. Close agreement was obtained by the use of Armsby's earlier method of calculating net energy values. Expression of nutrient values in terms of total digestible nutrients led to less satisfactory agreement.

A. G. POLLARD.

**Nutrient requirements for normal growth of dairy cattle.** C. H. ECKLES and T. W. GULLICKSON (J. Agric. Res., 1931, 42, 603—616).—An experimental

comparison of the Wolff-Lehmann, Kellner, Armsby, and Morrison feeding standards for heifers is recorded. The Morrison is too low for normal growth up to 1 yr., and too high above that age. A. G. POLLARD.

**Animal and plant proteins in pig feeding.** PROBST (Deut. landw. Tierz., 1930, 34, 619—625; Bied. Zentr., 1931, 60A, 165).—In a criticism of published data concerning the protein requirement of fattening pigs, it is claimed that animal protein in pig rations may be replaced entirely by plant protein without affecting flesh production and with greater economy in feeding costs. A. G. POLLARD.

**Lime and nutrient material content of Hungarian soils.** G. DOBY, J. CSIKY, and F. SRASSEL (Mezőg. Kutat., 1930, 3, 497—504; Chem. Zentr., 1931, i, 1661).

**Mineral content of pastures. Feeding value of meadow hay etc.**—See XIX. **Organo-Hg compounds as bactericides.**—See XXIII.

#### PATENTS.

**Production of fertilisers.** A. M. CLARK, W. K. HALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,184, 8.2.30).—The caking of fertilisers containing  $(\text{NH}_4)_2\text{SO}_4$  or  $\text{NH}_4\text{NO}_3$  is prevented by adding 1—5% of a starchy material, *e.g.*, flour or rice meal, of such a fineness as to pass 100-mesh. W. J. WRIGHT.

**Production of fertilisers from phosphate rock and the like.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,224, 8.4.30).—Phosphate rock (etc.) is dissolved in  $\text{HNO}_3$ , the clear solution neutralised with aq.  $\text{NH}_3$ , and the  $\text{P}_2\text{O}_5$  precipitated as  $\text{FePO}_4$  by adding  $\text{Fe}(\text{NO}_3)_3$  and more aq.  $\text{NH}_3$  until  $p_{\text{H}}$  4.5 is reached. The ppt. is removed, the filtrate evaporated to recover  $\text{NH}_4\text{NO}_3$ , and the ppt. digested with aq.  $\text{NH}_3$  to obtain  $(\text{NH}_4)_2\text{HPO}_4$  and  $\text{Fe}(\text{OH})_3$ , which is reconverted into  $\text{Fe}(\text{NO}_3)_3$ . A. R. POWELL.

**Treating phosphates to obtain an artificial fertiliser.** J. KUUSK (B.P. 349,020, 13.2.30).—Phosphate rock is rendered sol. in citric acid by heating it with 18—40%  $\text{SiO}_2$  at 1300—1550° to produce the compounds  $6\text{Ca}_3\text{P}_2\text{O}_8 \cdot \text{Ca}_2\text{SiO}_4$  or  $3\text{Ca}_3\text{P}_2\text{O}_8 \cdot \text{CaSiO}_3$ . [Stat. ref.] A. R. POWELL.

**Manufacture of fertilisers [from distillery and beet-sugar waste liquors].** I. DUBINBAUM and L. R. CHRISTIE (B.P. 349,178, 11.3.30).—A mixture of the liquor with  $\text{H}_2\text{SO}_4$  is dried by atomisation, *e.g.*, by a revolving disc, in a current of hot air; the product, which contains N and K, is used direct or after admixture with  $\text{H}_3\text{PO}_4$  as a fertiliser. L. A. COLES.

**Production of artificial [mixed] fertilisers.** ARMOUR FERTILIZER WORKS (B.P. 349,066, 14.1.30. U.S., 1.2.29).—A mixture of superphosphate, mineral acid (*e.g.*,  $\text{H}_3\text{PO}_4$ ), and  $\text{H}_2\text{O}$ -sol. fertilisers (*e.g.*, "leunasalpetar" and KCl) is treated with sufficient liquid anhyd.  $\text{NH}_3$  to neutralise the free acid without causing loss of  $\text{H}_2\text{O}$ -solubility of the phosphates and to yield a "dry-to-the-touch" product on curing. L. A. COLES.

**Plant fertilisers.** G. WANSART and F. SILBERMANN (B.P. 348,056, 27.1.30).—Kieselguhr or other absorbent

material is mixed with granular superphosphate or granular mixtures of superphosphate,  $(\text{NH}_4)_2\text{SO}_4$ , and a K salt to increase their effectiveness. [Stat. ref.] W. J. WRIGHT.

**Granulation of fertilisers.** W. K. HALL, F. HEYWOOD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,007, 19.12.29).—A mixture of sol. fertiliser salts, with or without an inert insol. filler, is fed into a tube containing a rapidly rotating agitator and steam is passed into the mass until it contains 4—5%  $\text{H}_2\text{O}$ . *E.g.*, a mixed fertiliser containing 26% KCl, 25.7%  $\text{NH}_4\text{H}_2\text{PO}_4$ , and 48.3%  $(\text{NH}_4)_2\text{SO}_4$  is prepared by feeding the salts, ground to 100-mesh, into the tube with the agitator revolving at 120 r.p.m. and passing steam into the mass until the temp. reaches 60°. A. R. POWELL.

**Production of [non-caking] fertilisers.** A. M. CLARK, W. K. HALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 348,640, 8.2.30).—Up to 5% of  $\text{Na}_2\text{HPO}_4$  or  $\text{K}_2\text{HPO}_4$  is added to fertilisers containing  $\text{NH}_4\text{NO}_3$ . H. ROYAL-DAWSON.

**Manufacture of artificial turf.** H. KORTLANG, and TURFO CONSTRUCTION CO., LTD. (B.P. 348,814, 4.4.30).—Vegetable matter, *e.g.*, cotton-seed husks or hulls, is successively swelled by heat or saturation with  $\text{H}_2\text{O}$ , hardened by treatment with  $\text{Ca}(\text{OH})_2$ , drained, and compressed, *e.g.*, by rolling after laying. Dyes and fixing agents to prevent fading may be added. L. A. COLES.

**Non-caking salts.**—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Alteration of sweet dried [beet] slices during storage.** K. ŠANDERA and C. A. RUŽICKA (Z. Zuckerind. Czechoslov., 1931, 55, 299—303).—Samples of beet slices dried by the Oxford process at Eynsham factory were stored for 18 months under various indoor conditions. No deterioration occurred under normal conditions with temp. variations of 10—30°. Access of air tended to lower the invert sugar content. Very drastic deterioration occurred in the warm, moist air of a glasshouse. J. H. LANE.

**Acidity of cane juice during grinding.** F. S. GOMEZ (Philippine Agriculturalist, 1931, 19, 609—634).—Determinations made as the cane passed from the crusher through the different units of a 14-roller mill grinding Luzon White and Pampanga Red varieties showed the average  $p_{\text{H}}$  to increase from crusher to 4th mill, though the  $p_{\text{H}}$  of the mixed juice was little different from that of the crusher juice. But when all the mills were thoroughly washed down once in every shift, and when the juice troughs were kept free from bagasse accumulations, there was no increase in the  $p_{\text{H}}$  from crusher to mill juice. J. P. OGILVIE.

**Detecting inversion in and around the [sugar-cane] factory crushing plant.** M. BIRD (Internat. Sugar J., 1931, 33, 293).—If there is any serious inversion taking place at the milling plant, the glucose ratio in the mixed juice must be higher than that of the crusher or first mill. If this is found to be the case, an effective remedy is to allow a small stream of milk-of-lime to flow into the last mill juice, if this is being

returned, or into the maceration water, if this is being applied behind the first mill. J. P. OGILVIE.

**Use of lime and sulphurous acid in the defecation of cane juices.** N. LÉVY (Bull. Assoc. Chim. Sucr., 1930, 47, 500—503).—The amount of CaO required for defecation is best ascertained by a method previously described (B., 1910, 1401) and now republished. Sulphitation is better before than after liming.

J. H. LANE.

**Teatini's [sugar] juice purification process.** O. SPENGLER and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1931, 81, 233—245).—Laboratory experiments are described demonstrating (a) that the same results are obtained in respect of purity increase and colour decrease when in the Teatini process the addition of liquid SO<sub>2</sub> is omitted; (b) that when the same amount of CaO prescribed in the Teatini process is added in one instead of two fractions the results are inferior to those of that process, with or without the addition of SO<sub>2</sub>; and (c) that the use of the ordinary carbonatation method of clarification, using 1.8% of CaO (added at one time), gives distinctly superior results to the Teatini process as regards purity and colour. In a raw sugar factory the quality of the product in respect of affinity distinctly deteriorated when the Teatini process was put into operation, whilst in the Offstein factory an inferior white sugar resulted.

J. P. OGILVIE.

**Gums in raw sugar solutions.** H. F. BOMONTI (Proc. Hawaiian Sugar Planters' Assoc., 1930).—Laboratory experiments showed that 65% of gums (org. matter insol. in EtOH) is found in the filtrate obtained by passing solutions of raw sugars through kieselguhr; and further, that 45% of these "gums" actually pass through a semi-permeable membrane of collodion. Apparently the sp. gr. of the solution does not influence the physical state of the gums.

J. P. OGILVIE.

**Determination of sugar in carbonatation scums.** V. STANĚK and T. NEMES (Z. Zuckerind. Czechoslov., 1931, 55, 451—453).—In place of the Zn(NO<sub>3</sub>)<sub>2</sub> solution employed in Czechoslovakian beet-sugar factories for the decomposition of the Ca saccharate in the determination of sugar in carbonatation scums, a solution of ZnCl<sub>2</sub>, prepared in the following way, is recommended: 1 kg. of dil. (1:1) HCl is poured over 0.5 kg. of Zn scrap and after a few hr. the solution resulting (35—40% ZnCl<sub>2</sub>), after dilution with 7—8 pts. of H<sub>2</sub>O to d 1.032, is applied in the same way as the Zn(NO<sub>3</sub>)<sub>2</sub> solution officially prescribed; it gives closely corresponding results and costs less.

J. P. OGILVIE.

**Sweetening-off carbonatation scums.** V. STANĚK (Z. Zuckerind. Czechoslov., 1931, 55, 443—451).—The scum press is filled as rapidly as possible, working from the start with the valve full open. When the rate of filtration has slowed down moderately, the flow of juice is stopped, and the outlet valves of the corresponding plates are closed. Then through the washing-out valve an aq. suspension of scums is pumped in. In this way fissures and voids in the cakes are filled up and washing performed effectively with a min. of H<sub>2</sub>O.

J. P. OGILVIE.

**Methods of controlling the end-point of the first carbonatation.** O. SPENGLER, F. TÖDT, and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1931, 81, 246—259).—In  $\frac{1}{2}$ -hourly runs with four methods of alkalinity control of the first carbonatation it was possible to keep most closely to the prescribed alkalinity with the use of thymolphthalein paper, the usual titration method showing the greatest variations of all. Lindner's electrical conductivity apparatus and Bruckner's froth-pressure gauge gave about the same results, their place being intermediate between the first two mentioned.

J. P. OGILVIE.

**Over-carbonatation [of beet-factory juices].** R. KARGL (Z. Zuckerind. Czechoslov., 1931, 55, 479—493).—On over-carbonatation, the amount of MgO passing into solution will depend on the alkali content and on the conditions of the operation. Presence of NH<sub>3</sub> assists the dissolution, probably with formation of Mg(NH<sub>4</sub>)<sub>2</sub>(CO<sub>3</sub>)<sub>2</sub>, only about half of which is decomposed during the boiling of the carbonatated juice. Titration of the Ca salts by soap solution is unsuitable for determining the crit. alkalinity for over-saturation.

J. P. OGILVIE.

**Solubilities [in sucrose] of some inorganic substances [as related to carbonatation].** E. SAILLARD (Suppl. hebdom. No. 2198).—The solubility of MgO and of MgCO<sub>3</sub> at 20° in an alkaline 10—12% solution of sucrose increases as the alkalinity decreases. The solubility of CaSO<sub>4</sub> at 20° in a 12% solution of sucrose increases if the solution contains CO<sub>2</sub>, and remains higher after boiling. MgSO<sub>3</sub> is not more sol. at 20° in pure 10—40% sucrose solution than in distilled H<sub>2</sub>O. SiO<sub>2</sub> gel is only slightly sol. at 20° in distilled H<sub>2</sub>O and in a 13.5% solution of sucrose, rendered alkaline with 1 g. of CaO per litre, but is more sol. if the same alkalinity is reached with soda. It is more sol. at 40° than at 20°.

J. P. OGILVIE.

**Crystallisation of low-purity massecuites.** S. J. SAINT (Internat. Sugar J., 1931, 33, 288—292).—Mother-liquor of a cooled massecuite is considerably supersaturated; hence, as means of facilitating curing, its temp. might be raised, e.g., from 32° to 50°, without fear of dissolving grain—a more advantageous procedure than adding H<sub>2</sub>O to the crystalliser. Additional sugar is recoverable if while raising the temp. the supersaturation of the mother-liquor be kept constant by the concentration of the massecuite. Hence, instead of discharging the massecuite, boiling would be continued, and the vacuum gradually lowered as the concentration is increased, so that the supersaturation of the mother-liquor remained const. This operation would be controlled with the Zeiss industrial refractometer. A factor of importance which has never been considered in any quant. way in its bearing on supersaturation is the area of the crystal surface exposed to the mother-liquor.

J. P. OGILVIE.

**Unification of conductometric methods for determining salts in raw sugars.** I, II. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1931, 55, 199—204, 215—226).—I. The problem of unification of methods is discussed generally on the basis of replies to a questionnaire addressed to sugar factories in various countries.

II. The author's conductometer (B., 1927, 312, 920) indicates directly 100 times the ash content (sulphated minus  $\frac{1}{10}$ ) of raw beet sugars when a concentration of 26 g./100 c.c. is employed. A concentration of 5 g./100 c.c. may be used, with a conversion factor which is 0.0264 for Czechoslovakian raw sugars, but apparently somewhat higher for those of Germany and Sweden, viz., 0.028—0.0294. The instrument is applicable to Zerban's methods and formulae, the calculations for which are simplified by graphs. Sp. conductivities of Czechoslovakian raw sugars at various concentrations are tabulated; Lundén's characteristic consts. (B., 1925, 1003) calc. therefrom do not appear highly significant for these sugars. J. H. LANE.

**Applicability of conductometric methods to the official determination of ash in raw sugars.** J. PUCHERNA (Z. Zuckerind. Czechoslov., 1931, 55, 205—206).—The ash contents of over 600 samples of first-product raw beet sugar were determined by incineration in the producing factories and by Sándera's conductometer (B., 1927, 312) in the receiving refinery. Differences of more than 0.04% occurred in only 6% of the samples, and of more than 0.6% in only 1% of the samples. J. H. LANE.

**Factors influencing the exhaustability of [cane] molasses: value of the "ash per cent. non-sugar" ratio.** J. G. THIEME (Internat. Sugar J., 1931, 33, 244).—There is only a slight correlation between the glucose % non-sugar and the exhaustion; the glucose/ash quotient is the more valuable figure. A trustworthy figure is the ash % non-sugar, as is also the alkali % non-sugar, the ash being found from the electrical conductivity. Cultural conditions, soil, and climate can exert a greater influence on the exhaustion of a cane molasses than clarification, a soil high in sol. salts and a dry season resulting in factory molasses of high purity. Comparison of the value of the exhaustion of cane molasses on the basis of the usual purity value is impracticable, it being necessary at the same time to take its ash % non-sugar quotient into account.

J. P. OGILVIE.

**Contamination of pure sucrose solutions by containing vessels.** J. PELLER (Z. Zuckerind. Czechoslov., 1931, 55, 287—292).—Increases in conductivity of sucrose solutions in glass and other vessels, in 6 hr., indicated that the solubility of glass increases with the concentration of sugar solutions and is rather greater at 100° than at room temp. Under the best conditions about  $3\frac{1}{2}$  times as much glass is dissolved by 26% sucrose solutions as by H<sub>2</sub>O, and the greater part is dissolved within 1 hr.; a 200-c.c. flask would yield about 0.02 mg. to a 26% sucrose solution. Results with Cu- and Cr-plated vessels were irregular. J. H. LANE.

**$\beta$ -Lactose.** H. SANDQVIST and J. GORTON (Svensk farm. Tidskr., 1930, 34, 665—667; Chem. Zentr., 1931, i, 1597).—The preparation of  $\beta$ -lactose is described and its use as a substitute for sucrose discussed.

A. A. ELDRIDGE.

**Bagasse utilisation by dry distillation.** F. W. FREISE (Internat. Sugar J., 1931, 33, 225—226).—Bagasse on being dry-distilled in the laboratory gave: coke 34.5, condensable gases 8.5, non-condensable gases

31.6, and H<sub>2</sub>O 25.4%; the condensable fraction yielded AcOH and homologues 10.55, MeOH 0.62, oily substances 14.22, soft pitch 63.25, H<sub>2</sub>O 10.35, gases and losses 1.01%. Retort gases show the following composition: CO<sub>2</sub> 61, CO 29, CH<sub>4</sub> 2.75, H<sub>2</sub> 1.8%. Other data are: 1 ton of bagasse yields 2.3 tons of steam and the pulverised coke evaporates 7.0—7.2 tons of H<sub>2</sub>O from and at 100°. It contains C 85, H 3, O 11, N and ash 1%. One ton of bagasse yields 0.3 ton of this coke, which is very brittle and suitable only as powdered fuel in the boiler house. J. P. OGILVIE.

**Re-arrangement of Brix and gravity tables.** P. F. STOTON (Internat. Sugar J., 1931, 33, 285—287).—Tables are reproduced correlating for sugar solutions: the sp. gr. at 20°/20°, the Brix in 100 g. of solution, the Brix in 100 c.c. of solution (3.881—26.002), and the Brix solute in normal solution (22.42—100).

J. P. OGILVIE.

**Filtration experiments with gluten solutions.** T. RUEMELE (Z. ges. Getreidew., 1930, 17, 257—261; Chem. Zentr., 1931, i, 1534—1535).—A study of dispersivity in 0.05N-lactic acid solutions. A. A. ELDRIDGE.

**Active charcoals etc.**—See II. Corrosion of "Silumin."—See X.

#### PATENTS.

**Purification and decolorisation of [sugar] solutions containing colloidal impurities.** MAXIMINE (B.P. 348,738, 25.2.30. Belg., 25.2.29).—The solutions are treated with an active charcoal sol prepared by heating cellulosic material with ZnCl<sub>2</sub> and HCl in solution (or with a mixture of H<sub>2</sub>SO<sub>4</sub> and KHSO<sub>4</sub> of m.p. about 200°), first at 1 atm. and then in closed vessels at 3—5 atm. to remove H<sub>2</sub>O. The product is washed with H<sub>2</sub>O and used as a paste containing 80—90% H<sub>2</sub>O. L. A. COLES.

**Production of caramel.** F. ALBRECHT (B.P. 346,136, 19.3.30. Ger., 23.3.29).—Powdered sugar, e.g., dextrose, is liquefied by aq. (25%) NH<sub>3</sub>, by stirring and slightly warming if necessary, and then autoclaved at 105—120° for 1 hr. or longer, after which the pressure is released; when atm. pressure is restored, the vessel is again closed and heating is resumed at 105—120° until the desired degree of colour has been attained. Before the product is discharged, undesirable volatile matters may be removed by a current of steam. The process occupies much less time than others. J. H. LANE.

**Manufacture of starches swelling in cold water.** E. F. HOPPLER and J. W. HAAKE (B.P. 346,224, 13.6.30).—Raw or prepared starches or starchy materials, containing not more than 25% H<sub>2</sub>O, are subjected simultaneously to heat and pressure, with or without agents such as thiocyanates which facilitate gelatinisation; e.g., raw starch, in layers 10 mm. thick between surfaces heated to 140—160°, may be subjected for about 5 sec. to 2500 kg./sq. cm. Substances conferring special properties may be added before or after the treatment.

J. H. LANE.

**Manufacture of substances [from starch] suitable for use in size.** NAAML. VENN. W. A. SCHOLTEN'S CHEM. FABR. (B.P. 346,220, 10.6.30. Ger., 17.6.29).—Dry painters' size which does not deteriorate on storage



is made by incorporating with an alkaline cold-swelling starch, during or after its preparation, a sol. salt of naphthenic acid, e.g., 6--8% of the Na salt.

J. H. LANE.

**Manufacture of sugar.** DORR Co. (B.P. 349,632, 4.12.29. U.S., 4.12.28).—See U.S.P. 1,755,165; B., 1930, 1085.

**Treatment of cellulose materials. Saccharification of carbohydrates.**—See V. Fertilisers from waste liquors.—See XVI.

### XVIII.—FERMENTATION INDUSTRIES.

**Diastatic powers of barley malt and oat malt.** W. KILP (Z. Spiritusind., 1931, 54, 149—151).—The starch-saccharifying power of oat amylase, as measured by the Windisch-Kolbach method, is approx. 3% of that of barley amylase. With normal malting, the saccharifying power of barley reaches a max. in 15 days, whereas that of oats increases for at least 20 days, and, when the conditions of germination are the same, the saccharifying power of the green malt from oats is 10—15% of that of malted barley, the higher values being obtained with the longer germination periods. The relatively high starch-conversion power of the green malt from oats in spite of its low saccharifying power is explained by the starch-liquefying power of the malted oats being approx. twice that of the barley malt.

C. RANKEN.

**Importance of dehydrating agents in the azeotropic process for manufacture of absolute alcohol.** J. L. GENDRE (J. pr. Chem., 1931, [ii], 130, 23—34).—A critical review of the commercial methods of producing abs. EtOH. The use of  $C_6H_6$  alone as the dehydrating agent is not satisfactory.

R. CUTHILL.

**Water in power EtOH.**—See II. Honey diastase. **Spent-wash from potato-spirit distillery.**—See XIX.

### PATENTS.

**Applications of moulds [e.g., yeast and bacteria] and apparatus therefor.** IMPERIAL CHEM. INDUSTRIES, LTD., and C. H. LILLY (B.P. 349,654, 5.3.30).—Mould "felt" is developed on the outer surface of porous-walled tubes constructed of filter cloth, earthenware, etc., through which nutrient solution (e.g., carbohydrate) circulates at a regulated rate. The tubes are vertically suspended in a suitable tower supplied with sterilised air, if desired. Products such as citric acid, fumaric acid, mannitol, etc. may be obtained by means of the process.

C. RANKEN.

**Simultaneous production of butyl alcohol and acetone by fermentation.** H. WERTHEIM and W. POLLAK (B.P. 349,490—1, 17.2.30. Austr., 23.2.29).—(a) Strains of *B. amylobacter*, A.M. & Bredemann, are cultivated without neutralisation of the fermentation acids on nutrient media in a series of habituating fermentations between each of which is a sporulating fermentation in which the fermentation acids are neutralised. The initial acidity, the concentration of the carbohydrates, and the amount of buffer substances of the habituating fermentations are increased from one fermentation to another, and the cultures are heated for a short time to destroy vegetative growth

prior to being used to inoculate the next habituating fermentation. (b) The carbohydrate mashings of the main fermentation are acidified from the beginning by addition or fermentative generation of non-volatile org. acids, preferably of lactic acid.

C. RANKEN.

**Manufacture of organic acids [by fermentation].** F. J. CAHN (B.P. 348,214, 14.2.30. U.S., 16.2.29).—Acids (citric, malic, succinic, etc.) are manufactured by impregnating plant-tissue material with carbohydrates, and inoculating the mass with a selected strain of *Aspergillus*, *Mucor*, or other fungi. The tissue may be superficially powdered with  $CaCO_3$ ,  $ZnO$ , etc.

C. RANKEN.

**[Regeneration of sulphites in the] manufacture of glycerin by fermentation.** IMPERIAL CHEM. INDUSTRIES, LTD., and G. G. JONES (B.P. 348,787, 21.3.30).—The fermentation residues after removal of glycerin are calcined and the solution from the leached product is treated with  $SO_2$ ,  $H_2SO_3$ , or a bisulphite. Carbonaceous matter with or without chalk may be added before or during calcination.

C. RANKEN.

**Preventing frothing in vats.**—See I. **Treatment of cellulosic materials.**—See V. Fertilisers from waste liquors.—See XVI.

### XIX.—FOODS.

**Effect of bleaching on strength and colour of Saskatchewan hard red spring-wheat flour.** R. K. LARMOUR and F. D. MACHON (Canad. J. Res., 1931, 4, 399—420; cf. B., 1929, 794).—Apparatus for the rapid bleaching of 600-g. samples of flour with Cl is described. Novadel (benzoyl peroxide) bleaches flour, but has little maturing effect, whilst Cl performs both functions, the maturing being greater the higher is the protein content of the flour. Unbleached flour baked with addition of  $KBrO_3$  gives similar results to Cl-bleached flour. Cl-bleached flours are further improved by ageing, but not so much as in other cases. Blending qualities of flours are not improved by bleaching.

A. A. LEVI.

**Correlation between baking quality and crude protein content of New Zealand wheat.** H. E. WEST (New Zealand J. Sci. Tech., 1931, 12, 273—281).—Although there is a positive correlation between the protein content of flour and its baking quality as represented by loaf vol., the correlation coeffs. are in many cases too small to be of practical significance. A high positive correlation was obtained in New Zealand wheats when the baking quality was measured in terms of the internal and external loaf characteristics specified.

A. G. POLLARD.

**Variability in experimental baking. I. Influence of mechanical moulding.** W. F. GEDDES, C. H. GOULDEN, S. T. HADLEY, and H. N. BERGSTENSSON (Canad. J. Res., 1931, 4, 421—448).—A comparison of the effects of hand- and machine-moulding on variability of loaf vol. in baking tests, with a view of finding an improved standard procedure.

A. A. LEVI.

**Tables for the examination and detection of chemical products used in milling.** P. BRUÈRE and M. MOCOS (J. Pharm. Chim., 1931, [viii], 13, 574—577).—The composition and rapid methods for the identification

of commercial chemical products used in milling and baking are described. E. H. SHARPLES.

**Calculation of the egg content of pastry.** J. FIEBE (Z. Unters. Lebensm., 1931, 61, 428).—Juckenack's tables (1900), which are usually employed to calculate egg content from the % of lecithin phosphate, should be used with caution for amounts of an order corresponding with 70 eggs/100 kg., the lecithin phosphate content of dry egg-free wheat grits (mean val. 0.0228%) being allowed for in the computation. J. GRANT.

**Detection of Riegel's "new preservative for pumpnickel" [chloramine-T].** P. WEINSTEIN, J. MUESMANN, and W. BODSCHWINNA (Z. Unters. Lebensm., 1931, 61, 436—442).—"Antibacterin" was identified as chloramine-T, and is detected in 400 g. of crumbed bread by extraction with hot Et<sub>2</sub>O containing 0.4% HCl, evaporation, elimination of fat by extraction with hot 50% EtOH, and re-extraction of the residue after evaporation with Et<sub>2</sub>O. If the N from the Et<sub>2</sub>O extract is >10 mg./kg. and the BaSO<sub>4</sub> ppt. (after fusion with 2NaCO<sub>3</sub> + 1NaNO<sub>3</sub>, acidification, and addition of BaCl<sub>2</sub>) is >15 mg./kg., addition of antibacterin is indicated. "Mikrobin-P," a suggested alternative preservative, was shown to be BzOH with 10% of chlorobenzoic acid, and is detected in the Et<sub>2</sub>O extract in the usual way. J. GRANT.

**Rising of fat in milk.** G. JØRGENSEN (Analyst, 1931, 56, 380—382).—A criticism of the technique of Stock (cf. B., 1931, 133) and of his results. Jørgensen finds that, on settling, the greatest decrease in the fat content of the milk, at the bottom of the vessel, takes place during the first hr. T. McLACHLAN.

**Relation of certain plant processes to flavour development in market milk.** P. H. TRACY and H. A. RUEHE (J. Dairy Sci., 1931, 14, 250—267).—Detrimental effects on milk flavour are produced by use of plant made from Fe, Cu, Benedict Ni, and various plant sterilisers. A. G. POLLARD.

**Synthetic methods in the examination of milk. Milk as a colloid. Mechanism of action of rennin.** C. PORCHER (Lait, 1929, 9, 449—474, 572—612, 681—711, 793—816, 942—970, 1051—1076; 1930, 10, 47—68, 146—175, 291—323, 401—427, 667—683, 794—812, 900—918, 1011—1026, 1123—1137; 1931, 11, 1—11; Chem. Zentr., 1931, i, 1536).—A comprehensive investigation and discussion, for details of which the original must be consulted. A. A. ELDRIDGE.

**Detection of milk unstable to heat.** G. A. RAMSDALL, W. T. JOHNSON, JUN., and F. R. EVANS (J. Dairy Sci., 1931, 14, 93—106).—Whole-milk samples (2 c.c.) are heated with 0.2 c.c. of 0.5M-KH<sub>2</sub>PO<sub>4</sub> in boiling H<sub>2</sub>O for 5 min. The formation of curd indicates low heat stability. Composite samples which include low-stability milks are themselves of low stability. The minimum concentration of KH<sub>2</sub>PO<sub>4</sub> necessary to produce curdling ("phosphate number") is closely related to the heat stability as determined in a steriliser, but cannot be correlated with  $p_{\text{H}}$ . A. G. POLLARD.

**Effect of dilution on the titratable acidity of cows' milk.** H. H. SOMMER and J. MÉNOS (J. Dairy Sci., 1931, 14, 136—155).—Dilution of milk samples

with H<sub>2</sub>O lowers their titratable acidity. A precipitation of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> probably occurs during the titration of whole milk. Dilution decreases this precipitation and the titration is proportionally reduced. A similar effect with Ca caseinate is indicated. The lowered  $p_{\text{H}}$  of diluted milk at the phenolphthalein end-point of the titration is attributed to a "protein effect" and the fading of the end-point with whole milk to the precipitation of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. The higher titratable acidity of conc. milks, as compared with values calc. from those of the original samples, is similarly explained. Treatment of milk or cream with lime-water does not reduce its acidity to the calc. point, since the increased [Ca<sup>++</sup>] causes greater precipitation of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> in subsequent titration. A. G. POLLARD.

**Nitrate test for detection of added water in milk.** G. W. MONIER-WILLIAMS (Analyst, 1931, 56, 397—398).—Diphenylbenzidine is an intermediate product in the oxidation of diphenylamine to a quinone-immonium salt, which gives the blue colour. If an excess of diphenylamine is employed, all the nitrate is used in the first stage of the reaction and may not be detected. This may be avoided by using diphenylbenzidine as the reagent, and the test is then so sensitive that amounts of NO<sub>3</sub><sup>-</sup> ranging from 0.1—1.0 p.p.m. may be detected. T. McLACHLAN.

**Helmer test for formaldehyde [in milk].** C. C. FULFON (Ind. Eng. Chem. [Anal.], 1931, 3, 199—200).—The Helmer test can be improved by using Br for the oxidising agent and by diluting the H<sub>2</sub>SO<sub>4</sub> before adding the milk to be tested. The colour can be developed in a zone, or uniformly distributed throughout the solution. By the improved test 1 p.p.m. of CH<sub>2</sub>O in milk can be detected. E. S. HEDGES.

**Soluble soya-bean milk powder and its adaption to infant feeding.** K. CHANG and E. TSO (Chinese J. Physiol., 1931, 5, 199—203).—Milk reconstituted from soya-bean milk powder (prepared by a spray process) supplemented with cane sugar, Ca lactate, NaCl, cod-liver oil, and cabbage water has been satisfactorily employed in infant feeding. W. O. KERMACK.

**Dependence of the clotting of cream on the external experimental conditions and the specific properties of the milk.** A. SCHNECK (Milch. Zentr., 1931, 60, 151—156).—The degree of clotting is defined by the formula  $100 - (F/F_1)m\%$ , where  $F$  and  $F_1$  are the fat contents of the whole milk and whey, respectively, and  $m$  is vol.-% whey. At temp. below 37° the degree and the velocity of clotting decrease as the length of the storage increases. Compared with the values given by milk at cow-heat (37°), heating above 37° (up to 61°) causes an increase, and heat treatment below 37° a decrease, in the degree and velocity of clotting. The loss of cream-separating power by stale milk is largely restored by heat treatment. Mechanical stirring has no appreciable influence on the cream-rising process, and no definite relationships could be established between the sp. constants of the milk (viscosity, refraction, etc.) and the cream separation. H. J. DOWDEN.

**Dry skim milk in ice cream.** W. V. PRICE and R. WHITAKER (Cornell Univ. Agric. Exp. Sta. Bull., 1931, No. 516, 37 pp.).—Dry skim milk produced by

spray-drying methods is a desirable source of solids-not-fat for ice cream. The "atmospheric roll" process produces a dry milk of lower solubility and having a "cooked" flavour. A. G. POLLARD.

**Influence of sweetened frozen cream on the development of swell in ice cream.** W. V. PRICE (J. Dairy Sci., 1931, 14, 221—228).—The inclusion of sweetened frozen cream as the only source of milk fat in ice-cream mixtures produces better whipping properties than unsweetened cream. Invert syrup and sucrose in concentrations producing the same f.p. depression are equally effective for sweetening. A relation between lecithin content and swell development in mixtures is indicated. A. G. POLLARD.

**Standardisation of milk for the manufacture of American cheese.** W. V. PRICE and L. GERMAIN (Wisconsin Agric. Exp. Sta. Res. Bull., 1931, No. 108, 11 pp.).—Cheese made from milk the casein:fat ratio of which was adjusted to 0.7 showed a decreased yield, increased moisture and decreased fat contents, and was of slightly poorer quality than that made from normal milk. The total value of the products from unit quantity of milk was, however, increased. The effects of adjusting the composition of milk were greatest when the original fat of the milk was  $> 3.5\%$  and were not significant when the fat content was  $3.3\%$ . A. G. POLLARD.

**Caseinogen and lactic acid.** M. E. SCHULZ (Milch-wirt. Forsch., 1930, 11, 294—301; Chem. Zentr., 1931, i, 1690).—Caseinogen swells in lactic acid (up to  $2.25\%$ ), the increase being less in  $2.25$ — $5.63\%$  acid; it dissolves on warming and forms a jelly on cooling. Caseinogen is more viscous in acid than in alkaline solution; the viscosity depends on the concentration and quality, good lactic acid caseinogen with small mineral matter content having a lower viscosity than industrial caseinogen. A. A. ELDRIDGE.

**Examination of eggs suspected of being "preserved."** J. R. NICHOLLS (Analyst, 1931, 56, 383—390).—Unpreserved eggs are porous, whilst those preserved in oil, Na silicate, or lime water are non-porous. Eggs preserved in oil give marked surface effects, but absorb dyes normally; limed or silicated eggs show pronounced differences in reaction towards certain dyes, notably methylene-blue ( $0.1\%$ ) and Congo-red ( $0.1\%$ ). There is no difference in the  $[H^+]$  of fresh and preserved eggs. T. McLACHLAN.

**Differentiation of fresh, refrigerated, and preserved eggs.** J. J. J. DINGEMANS (Chem. Weekblad, 1931, 28, 350—351).—The egg is digested with  $H_2O$  in a metal vessel. If  $CaO$  or  $Ca$  silicate is present the  $H_2O$  becomes alkaline to phenolphthalein; differentiation is effected by evaporation to small vol. and testing for  $Ca$  and  $SiO_2$ . On viewing by the quartz Hg lamp refrigerated eggs may be differentiated from fresh eggs by the appearance of fluorescence. H. F. GILLBE.

**Evaluation of honey from the diastase content.** K. BRAUNSDORF (Z. Unters. Lebensm., 1931, 61, 411—420).—The Fiehe-Kordatzki (B., 1928, 385, 543) and phloroglucinol tests for diastase showed good general agreement for the 62 German honeys examined,

but the latter has a disadvantage in that it fails to distinguish between naturally low or non-diastatic honeys and honeys (particularly of this class) which have been heated so as partly to destroy the diastase and are therefore condemned by the German (1930) regulations. In the former test, however, the production of a pale yellow colour immediately on addition of the reagent is an indication of non-deleterious heating, whilst a golden-yellow colour results from strongly heated samples (e.g., after 1 hr. at  $60^\circ$  the colour from a fairly diastatic honey will match that of  $0.1N-K_2Cr_2O_7$ ). The diastatic values found were 62.5 (1 honey), 50—11 (36),  $<10$  (8); a first-class diastatic honey should therefore give  $>23.8$ , and unheated honeys  $>17.9$ , whilst 10 is the suggested limit for the German regulations (cf. Braunsdorf, B., 1931, 564). J. GRANT.

**Honey diastase.** J. FIEHE (Z. Unters. Lebensm., 1931, 61, 420—427).—The lower limit of 8.3 for the diastase value determined according to the Fiehe-Kordatzki method (B., 1928, 385, 543) is suggested for heated honeys assessed under the German (1930) regulations (cf. B., 1931, 564, and preceding abstract). The diastase value for honeys heated with stirring at  $60^\circ$  is seldom lowered to any great extent, final values of  $<8.3$  being obtained for only 2 out of 190 German and foreign samples. Californian "orange-blossom" and "belvedere" honeys are often naturally poor in diastase (diastase val. 2.5 and 1.0), but if such low values are not the result of heating, the samples cannot be said to contravene the regulations. The end-point of the Fiehe test may be sensitised by selective adsorption of the blue colour (as distinct from the sol. purple of the iododextrins) produced by addition of washed precipitated  $Al(OH)_3$ . J. GRANT.

**Trigonelline contents of coffees.** F. E. NOTTBOHM and F. MAYER (Z. Unters. Lebensm., 1931, 61, 429—435).—The "alkaloid value" of a coffee (caffeine plus trigonelline content; cf. B., 1931, 652) is 4.3—5.5 for all ordinary coffees, but higher (9.7—13.7) for wild African (especially Surinam-Liberian) coffees, which latter are exceptionally low in trigonelline ( $0.1$ — $0.2$  instead of  $0.23$ — $0.25\%$ ) and often high in caffeine ( $1.2$ — $2.2\%$ ). EtOH extracts of the latter type of coffee give pure yellow instead of orange-red lead ppts. (*loc. cit.*). Treated coffees or those roasted at  $250^\circ$  lose no trigonelline, but the latter give an oily ppt. with I in KI, indicating nitrogenous decomposition products. Aq. infusions of coffee remove about  $82\%$  of the caffeine and  $70\%$  of the trigonelline present. The orange-red salt  $C_7H_7NO_2 \cdot HCl \cdot PtCl_4$  has m.p.  $215^\circ$ . J. GRANT.

**Vitamins. III. Vitamin[-C] contents of fruits.** F. V. VON HAHN (Z. Unters. Lebensm., 1931, 61, 369—411; cf. B., 1930, 838).—On the basis of animal experiments 40 types of fruits are classified as: (a) very rich in vitamin-C (strawberry, orange, lemon); (b) rich (gooseberry, raspberry, dried or cooked hipberry); (c) containing vitamin-C (red currant, mandarin); (d) poor (freshly-picked apple, cherry, greengage, plum, peach, prune, banana, pineapple, cooked elderberry, heart cherry); (e) being practically free from vitamin-C

(raw or cooked bilberry, old apple, pear, raw or cooked quince, grape, dried fruits). J. GRANT.

**Citrus pectin in solid form.** U. WEGENER (Chem.-Ztg., 1931, 55, 473—474).—A method of preparation from citrus-fruit residues, which does not involve the use of EtOH, yields a stable, tasteless, odourless pectin in powder form, containing at least 45% of pectin. The powder is readily sol. in H<sub>2</sub>O. H. J. DOWDEN.

**Determination of soluble solids in jams etc.** T. MACARA (Analyst, 1931, 56, 391—396).—The % sugar in a jam may be read directly from a table using either the Abbé or butyro-refractometer. Other tables show corrections to be applied for invert sugar, glucose solids, and citric acid, when the solids have been determined either by the refractometer or from the sp. gr. Methods of calculating results are illustrated.

T. McLACHLAN.

**Mineral content of pastures.** B. C. ASTON, R. E. R. GRIMMETT, F. J. A. BROGAN, and P. H. SYKES (New Zealand J. Sci. Tech., 1931, 12, 304—320).—The phosphate content of the pastures examined reached a max. in spring, declining steadily to an autumn min. The Ca content was low in winter, rising through the season to a max. in autumn. The max. N content was attained in winter or spring and the min. in autumn. In wet seasons there was an increased proportion of N and P and a decrease in Ca. The P deficiency observed in these pastures is attributed to lack of summer and autumn rain rather than to soil defects. To remedy deficiency diseases in cattle, the immediate use of phosphatic fertilisers or the provision of salt licks containing Nauru phosphate or bone dust is recommended.

A. G. POLLARD.

**Determination of the feeding value of meadow hay and aftermath based on botanical and chemical analysis.** A. ZAITSCHEK and B. LÜSKE (Kisérlet. Közlemények, 1929, 32, 461—491; Bied. Zentr., 1931, 60A, 171—172).—Feeding values of a number of grasses for various animals are discussed. The digestibility of fibre-free org. matter of hay by horses is 5—13% lower than in the case of ruminants. The N-free extractives, crude fibre, and fat were less easily, and the protein more easily, utilised by horses than by ruminants. The amide content of hay is not characteristic of its quality, but depends on the period of cutting.

A. G. POLLARD.

**Quality of lucerne hay in relation to curing practice.** T. A. KIESSELBACH and A. ANDERSON (U.S. Dept. Agric. Tech. Bull., 1931, No. 235, 25 pp.).—To maintain a high protein content in the hay, all curing operations should aim at rapid drying with a min. of mechanical injury to leaves.

A. G. POLLARD.

**Replacement of oats in rations for working horses by fresh spent wash from potato-spirit distillery.** H. G. MÜLLER (Landw. Jahrb., 1931, 73, 169—217).—Warm, fresh spent wash may be substituted for oats at the rate of 8 litres of wash per 1 lb. of oats in rations for working horses without impoverishment of health conditions. The modified ration has a higher proportion of crude protein and a lower starch value.

A. G. POLLARD.

## PATENTS.

**Treatment of wheat and flour.** N. T. BRYAN (B.P. 348,594, 3.2.30).—Wheat containing buckwheat seeds is subjected to a series of impacts by passage through two drums containing rapidly rotating beaters. The impacts break the shells of the buckwheat without damaging the wheat berries, the shells are removed, and the remaining material containing buckwheat kernels is milled into flour. E. B. HUGHES.

**Production of wholemeal bread.** V. KLOFFER, and DR. V. KLOFFER'S NAHRUNGSMITTEL A.-G. (B.P. 348,029, 1.2.30).—The bran from rye flour is made into a dough with H<sub>2</sub>O mixed with grain extracts containing enzymes, lactic acid cultures, and innocuous metal salts or org. acids. The dough is heated and then dried *in vacuo* and the residue ground to fine powder and mixed with the rye flour. E. B. HUGHES.

**Prevention of stalk rot in fruit.** R. G. TOMKINS and R. M. WOODMAN (B.P. 348,755, 7.3.30).—The cut stalks of fruit and vegetables are rendered resistant or impermeable to micro-organisms by electrical cautery, which may be combined with a coating of animal, vegetable, or mineral oils, fats, waxes, gums, resins, cellulose derivatives, rubber mixtures, metallic soaps, etc., with or without the addition of thymol or other disinfectant.

E. B. HUGHES.

**Preservation by coating of fruit and like vegetable materials.** BROGDEN Co., Assees. of E. M. BROGDEN and M. L. TROWBRIDGE (B.P. 349,311, 29.5.30. U.S., 5.6.29).—A very thin, highly polished coating of waxy materials is applied. E. B. HUGHES.

**Preservation and storage of fruit and vegetables.** F. KIDD, R. G. TOMKINS, and S. A. TROUT (B.P. 349,561, 21.11.29).—Fungal rotting is inhibited or prevented on fruit and vegetables during storage by the presence of MeCHO and/or its derivatives, with or without other atm. control.

E. B. HUGHES.

**Extraction of pectin.** CALIFORNIA FRUIT GROWERS' EXCHANGE, Assees. of W. C. PLATT, G. M. COLE, and R. E. COX (B.P. 349,372, 25.7.30. U.S., 29.7.29).—A slow-setting pectin is extracted by means of acid from vegetables, fruits, etc., the *pH* being kept at 1.2—1.9, which results in the extract filtering easily. Jellies made with this pectin set in 5 min. as compared with 11—40 sec. taken by jellies made from pectins extracted at *pH* 3.3—3.5, and the product is better.

E. B. HUGHES.

**Manufacture of jellies.** J. E. POLLAK. From POMOSIN-WERKE GES.M.B.H. (B.P. 349,374, 28.7.30).—In the preparation in the cold of pectin jellies, a retarding agent such as glucose, starch paste, etc. is added to the pectin-sugar solution and the necessary acid introduced just before pouring out the mixture.

E. B. HUGHES.

**Making of jellies and the like.** A. LEO (B.P. 348,847, 22.4.30).—A dry mixture of sugar, pectin, and mucic acid is employed, the low solubility of the acid in cold H<sub>2</sub>O allowing all the pectin to dissolve, thus avoiding premature setting.

E. B. HUGHES.

**Chilling of beef.** W. W. TRIGGS. From SWIFT & Co. (B.P. 349,627, 31.12.29).

Drying or conditioning racks [for tea etc.]. J. A. MAIN (B.P. 349,639, 3.3.30).

Drying and cooling apparatus.—See I. Bleaching of flour.—See VI. Exterminating insect pests.—See XXIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Tinctures of cinchona. II. Compound tincture.** A. LICHTIN (Amer. J. Pharm., 1931, 103, 279—284; cf. B., 1931, 45).—The most stable compound tincture was prepared from COMe<sub>2</sub>-treated red cinchona bark (6% COMe<sub>2</sub> extract) by using a menstruum of 78% EtOH with the addition of glycerin (7.5%) before percolation (Lichtin process). HCl had no influence on the extraction of the alkaloids as above, but helped the extraction of the untreated bark (6.15% alkaloids), which, however, did not give as pure an extract. E. H. SHARPLES.

**Podophyllum rhizome—American and Indian. Crude fibre; tests for the resins; adulteration with guaiacum wood.** T. E. WALLIS and S. GOLDBERG (Quart. J. Pharm., 1931, 4, 28—32).—Determinations of crude fibre have been made by (a) the "Dutch" process, (b) the Ministry of Agriculture process, and (c) by process (a) after extraction with 90% EtOH. American podophyllum yields (a) 5.35%, (b) 7.5%, (c) 5.3% of crude fibre. Indian podophyllum yields (a) 7.2%, (b) 10.5%, (c) 6.7%. The addition of a 5% aq. solution of Cu(OAc)<sub>2</sub> to the alcoholic extract gives a bright green coloration with American and a brown ppt. with Indian podophyllum. A sample of American podophyllum was found to contain Indian podophyllum and guaiacum wood. R. K. CALLOW.

**Buffering subjects in ergot.** F. WOKES and G. K. ELPICK (Quart. J. Pharm., 1931, 4, 101).—A criticism (*ibid.*, 1930, 3, 599) of Thompson (B., 1930, 965) is withdrawn. R. K. CALLOW.

**Tobacco.**—See XVI. Organo-Hg compounds as bactericides.—See XXIII.

### PATENTS.

**Adhesive plasters.** JOHNSON & JOHNSON (GT. BRITAIN), LTD. From JOHNSON & JOHNSON (B.P. 348,694, 15.2.30).—The tendency of adhesive plaster to stick when rolled upon itself, and to become soiled when handled, is obviated by singeing the exposed fabric backing and applying thereto a coating of cellulose ester or viscose. D. J. NORMAN.

**Manufacture of perfumes.** I. G. FARBENIND. A.-G. (B.P. 347,052, 18.6.30. Ger., 18.6.29).— $\gamma$ -cyclo-Hexyl- $\alpha$ -alkylbutaldehydes, especially the 2:2:6-trimethyl homologues, have a bitter odour resembling nuts and leaves, and are valuable for blending in perfumes. C. HOLLINS.

**Manufacture of therapeutic agents.** SOC. CHEM. IND. IN BASLE (B.P. 346,180, 17.4.30. Switz., 17.4.29. Addn. to B.P. 270,339; B., 1928, 730).—A 2-halogenoquinolinecarboxylamide is condensed with an amino-alcohol (2- $\beta$ -diethylaminoethoxyquinoline-4-carboxyldiethylamide is excluded). The following are described: 2- $\beta$ -N-piperidinoethoxyquinoline-4-carboxylanilide, m.p.

172°; 2- $\beta$ -diethylaminoethoxyquinoline-4-carboxyl-ethylamide (oil), -methylanilide (m.p. 142°), -diisoamylamide (oil), -benzylanilide (oil), -benzylamide (m.p. 106°), -benzylethylamide (m.p. 110°), - $\beta$ -phenylethylamide (m.p. 87°), di-( $\beta$ -phenylethyl)amide (oil), -ac-tetrahydro- $\beta$ -naphthylamide (m.p. 113°); 2- $\beta$ -dimethylaminoethoxyquinoline-4-carboxylanilide (m.p. 147°); 2- $\beta$ -aminoethoxyquinoline-4-carboxylanilide (m.p. 215°); 2- $\beta$ -diethylaminoethoxyquinoline-3-carboxylanilide (m.p. 69°). The ethylanilide (m.p. 126°), methylanilide (m.p. 142°), diisoamylamide (b.p. 185°/0.015 mm.), benzylamide (m.p. 217°), benzylethylamide (m.p. 110°),  $\beta$ -phenylethylamide (m.p. 161°), di-( $\beta$ -phenylethyl)amide (m.p. 105°), and ac-tetrahydro- $\beta$ -naphthylamide (m.p. 182°) of 2-chloroquinoline-4-carboxylic acid, and the anilide (m.p. 184°) of the 3-carboxylic acid are mentioned. C. HOLLINS.

**Preparation of physiologically active quinazoline derivatives.** SOC. ANON. LEDOGA PRODOTTI CHIMICI E FARMACEUTICI (B.P. 346,118, 5.3.30. Ger., 30.5.29).—3:4-Dihydroquinazolines, carrying in position 6 either no substituent or an alkyl or alkoxy group, and in position 3 either phenyl or *p*-alkyl- or *p*-alkoxy-phenyl, yield quaternary bases valuable as local anaesthetics (alkoxy-compounds) or for lowering blood-pressure (alkyl and hydroxyalkyl compounds); if 6-substituent is absent the *N*-alkyl of the quaternary base should be at least C<sub>2</sub>. 6-Nitro-3-ethoxybenzaldehyde, m.p. 62°, is condensed with *p*-phenetidine to a Schiff's base, m.p. 92°, which is reduced to the amine, m.p. 156°, and further to 2-amino-5-ethoxybenzyl-*p*-phenetidine, m.p. 81°, from which by the action of formic acid there is obtained 6-ethoxy-3-*p*-phenetyl-3:4-dihydroquinazoline, m.p. 140°; the methiodide is converted by NaOH into the base, m.p. 114°. 3-*p*-Tolyl-6-methyl-3:4-dihydroquinazoline is treated with allyl bromide (base, m.p. 110°), or with ethylene chlorohydrin (hydrochloride, m.p. 214—215°). C. HOLLINS.

**Manufacture of aromatic arsinic acids.** I. G. FARBENIND. A.-G. (B.P. 347,083, 4.9.30. Ger., 4.9.29).—The halogen in halogenoacylaminoarsinic acids is exchanged for an acyloxy-group by interaction with an organic Na salt. The esters produced from chloroacetylarsanilic acid and AcONa or Na isovalerate or crotonate, and from 3-chloroacetamidotoluene-6-arsinic acid and Na isovalerate or crotonate have low toxicity and are good trypanocides. C. HOLLINS.

**Treatment of tobacco [with ultra-violet rays].** AMER. TOBACCO Co., Assess. of A. L. CHESLEY (B.P. 349,174, 10.3.30. U.S., 21.2.30).—Crude or aged tobacco (8—18% H<sub>2</sub>O) is subjected to the action of ultra-violet and infra-red rays at above 70° while being continuously agitated or tumbled during its passage through a specified apparatus. E. H. SHARPLES.

**Rendering non-poisonous the tobacco smoke during smoking.** BONICOT WARENVERTRIEBSGES. M.B.H. (B.P. 348,974, 18.8.30. Austr., 15.2.30).—The tobacco, in the form of cigarette, cigar, or in the pipe, is injected at specified points with a solution containing a Fe<sup>+++</sup> salt or a mixture of tartaric and citric acids, NaCl, Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>, coumarin, and, if desired, scents. E. H. SHARPLES.

**Manufacture of therapeutic substances.** SCHERING-KAHLBAUM A.-G. (B.P. 349,257, 25.4.30. Ger., 7.6.29).—Aq. or aq. salt-solution extracts of the central nervous organs of vertebrate animals are mixed with org. liquids to give a ppt. which has an exciting action on the central nerve system.  
E. H. SHARPLES.

**Manufacture of the hormone of the anterior lobe of the hypophysis.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,592, 31.1.30).—The hormone is obtained by salting out the urine of pregnant women or an aq. solution of the hormone prepared therefrom with any of the following salts either alone or in admixture:  $\text{NH}_4$ , Zn, and Al sulphates; Zn, Ba, and Ca chlorides;  $\text{K}_2\text{CO}_3$ ; Na and K acetates;  $\text{Ca}(\text{NO}_3)_2$ ; or a mixture of Na and Mg sulphates. The quantities used should be such that the concentration of anhyd. salt in the liquid is 20% or above. Fractional precipitation and/or ice-cooling may be employed.

E. H. SHARPLES.

**Odoriferous substances.**—See III. Al salts.—See VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Developers and development.** I. Quinol. V. O. DZISKO, M. V. BONDAREVA, and E. N. GAPON. II. Velocity of development and developing power in relation to temperature. III. Dependence of developing power on addition of sulphite. V. O. DZISKO and E. N. GAPON (Ukrain. Chem. J., 1930, 5, [Sci.], 295—307, 309—312, 313—315).—I. The velocity of development of AgBr by quinol has been determined at 25° and 35°. Development consists of a heterogeneous unimol. reaction between at. or mol. Ag and AgBr, catalysed by quinol, whereby crystals of Ag form and Br is liberated.

II. The developing effect of quinol and  $\text{Na}_2\text{SO}_3$  is a linear function of the temp. The velocity const. varies in accordance with the Arrhenius law over the interval 0—25°, whilst from 25° to 45° it is independent of temp. The percentage development ( $x$ ) is given by  $x = 41.9(1 + 0.0067T)(1 - e^{-0.212/T})$ , where  $t$  is time in min. and  $T$  is the temp. (25—45°).

III. The developing power of quinol is only slightly affected by variation in the concentration of the  $\text{Na}_2\text{SO}_3$  (0—1*M*), although it is considerably smaller in the absence of  $\text{Na}_2\text{SO}_3$ . The action of quinol does not depend exclusively on the formation of quinonesulphonic acid.  
R. TRUSZKOWSKI.

**Sensitising with desensitisers.** LÜPPO-CRAMER (Z. wiss. Phot., 1931, 30, 1—7; cf. Phot. Korr., 1931, 1).—Special emulsions (unripened), having large grains without sensitive nuclei, are sensitised by very dil. solutions of dyes which desensitise the ripened emulsion or normal plates. Plates of the ripened emulsion, treated with  $\text{CrO}_3$  to remove Ag nuclei, though not restored to as low sensitivity as the unripened emulsion, are similarly sensitisable. Hence desensitisation is dependent on the presence of Ag nuclei. The addition of the dyes at emulsification has no effect on these emulsions.  
J. LEWKOWITSCH.

**Solarisation phenomena in the negative blue-print process.** E. STENGER (Z. wiss. Phot., 1931, 30, 45—48).—The solarisation was quantitatively examined by bathing gelatin-coated plates in  $\text{Fe}^{+++}$  oxalate solution (A), exposing, and then developing with  $\text{K}_3\text{Fe}(\text{CN})_6$  solution (B). For a given exposure the degree of solarisation was directly proportional to the concentration of A, and inversely to that of B. The effect is removed by 1%  $\text{H}_2\text{SO}_4$ , and retarded by washing before the development. Solarisation is due to reduction of ferrous ferricyanide to ferrous ferrocyanide (a pale blue), which is oxidisable to Prussian blue. Prolonged exposure produces a second reversal due to a grey deposit in the film.  
J. LEWKOWITSCH.

**Lustre in fabrics.**—See VI.

## PATENTS.

**Manufacture of light-sensitive layers.** KALLE & Co. A.-G. (B.P. 347,430, 28.3.30. Ger., 30.3.29).—Diazotised arylamines having an acylamino- or thioacylamino-substituent are used as light-sensitive compounds. Examples are: diazotised 2-amino-5-benzamidoquinol diethyl ether with 8-hydroxy- $\beta$ -naphthyldiguanidine (blue), phloroglucinol (red-brown), 1:7-aminonaphthol (blue-grey), 2:7-dihydroxynaphthalene (red-violet), or phenyl-8-hydroxy- $\beta$ -naphthyldiguanidine (blue); tetrazotised diaminodiphenylthiocarbamide with phloroglucinol (brown-yellow); tetrazotised diaminotetramethoxydiphenylcarbamide with phloroglucinol (dark brown), 2:7-dihydroxynaphthalene (violet-blue), 1:7-aminonaphthol (green-blue), or 7-hydroxy- $\alpha$ -naphthyl-diguanidine (green-blue).  
C. HOLLINS.

**Production of films in colour.** A. F. DEGENHARDT (B.P. 348,507, 12.11.29).—In the two-colour process on double-coated film, the second bleaching and dyeing by the floating process is replaced by an immersion process;  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$  is added to the first ferricyanide bleacher (for the blue-green dyed side), which hardens that film and protects it from the action of the I bleacher, and orange-red dye used for the other side of the film.

J. LEWKOWITSCH.

## XXII.—EXPLOSIVES; MATCHES.

**Solvent recovery in the explosives industry, and the Bayer active-charcoal process.** E. REISEMANN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 145—149).—The characteristic feature of the Bayer process (G.P. 310,092; B., 1921, 204 A) is the use of highly adsorbent charcoal, so that even with very low concentrations of solvent vapour there is complete adsorption. The resultant possibility of drawing off large vols. of solvent vapour-air mixtures from the powder plant ensures the avoidance of pockets of solvent vapour or of concentrations between the explosive limits. During adsorption, the temp. of the charcoal does not rise more than 10°, and recovery of the vapours can be effected at not above 110°. The charcoal can be rapidly dried at a low temp. Loss of solvent from the vapour mixture delivered to the adsorbers does not exceed 2%. A brief description is given of the application of the process in the recovery of

Et<sub>2</sub>O and EtOH in a Chinese nitrocellulose powder factory. W. J. WRIGHT.

## PATENTS.

**Heating compositions for blasting cartridges.** D. HODGE and W. ESCHBACH (B.P. 348,657, 11.11.29. Cf. B.P. 345,284; B., 1931, 566).—In cartridges of the type containing solid or liquid CO<sub>2</sub>, the heating composition, *e.g.*, KClO<sub>4</sub> and Al, is rendered insensitive to shock or friction by adding about 5% of a phlegmatising agent, *e.g.*, paraffin wax. W. J. WRIGHT.

**Nitro-compounds of decreased inflammability.**—See III.

## XXIII.—SANITATION; WATER PURIFICATION.

**Properties of sewage colloids.** E. C. C. BALY (Trans. Faraday Soc., 1931, 27, 193–201).—The sign of the charge on sewage colloids has been determined by their behaviour when mixed with inorg. suspensions. Their isoelectric point, which ordinarily is at about *p*<sub>H</sub> 4.6, is displaced by 3% NaCl to *p*<sub>H</sub> 8.3. Gelatin behaves similarly. The amount of precipitable colloid decreases rapidly when fresh sewage is kept. The co-ppt. obtained by mixing with fine silt is bacterially active or inactive according to whether the silt or the sewage colloid is in excess. In presence of 3% NaCl under aerobic conditions spontaneous precipitation of bacteria-colloid complexes occurs after about 90 hr. F. L. USHER.

**Bacteriological evaluation of some new water-soluble organo-mercury compounds.** J. H. WALDO, H. A. SHONLE, and H. M. POWELL (J. Bact., 1931, 21, 323–329).—Among numerous organo-Hg compounds examined, ethyl mercurithiosalicylate possessed particularly valuable germicidal properties. A. G. POLLARD.

**Occurrence of hydrogen phosphide in spring waters.** O. LÜNING and K. BROHM (Z. Unters. Lebensm., 1931, 61, 443–446).—In Aug., 1928, a pronounced odour of PH<sub>3</sub> was observed from the waters of two neighbouring springs in the diluvial sands of Borstfeld (W. Brunswick), the AgNO<sub>3</sub> test giving a positive reaction. One water was slightly alkaline and hazy, with a flocculent deposit, and contained 6.9 mg. P<sub>2</sub>O<sub>5</sub> and 20 mg. Fe per litre; it had the high KMnO<sub>4</sub> absorption of 41–45 mg. per litre and a carbonate hardness of 28.3–42.6°. The water was undrinkable, and its condition may be due to reduction of P compounds by org. material or Fe<sup>2+</sup> during stagnation. J. GRANT.

**Water purification by precipitation and coagulation.** H. A. J. PIETERS and W. J. DE KOK (Chem. Weekblad, 1931, 28, 365–368).—The relative influence of alum, Ca(OH)<sub>2</sub>, BaCl<sub>2</sub>, and a number of other salts on the rate of clearing of H<sub>2</sub>O-loam suspensions has been determined, and is in the order stated. The rate of clearing is not influenced by the quantity of precipitant added above a definite limit. Similar results are obtained with clay suspensions and with the effluents from coal-washing plants; if Ca(OH)<sub>2</sub> is used as the precipitant, addition of starch greatly accelerates the settling. By filtration through weathered spent oxide, effluents may readily be clarified, as a result of the action of electrolytes produced during the weathering. H. F. GILLIE.

**Preventing boiler-scale formation.**—See I. Germicidal soaps.—See XII. Sanitation.—See XV.

## PATENTS.

**Testing air for impurities such as carbon monoxide.** W. H. PRITCHARD (B.P. 349,514, 26.2.30).—The presence of certain impurities can be shown by means of the effect they produce on the character of the light transmitted or reflected by substances or solutions with which they react, *e.g.*, CO on NH<sub>3</sub>-Ag<sub>2</sub>O solution (transmitted) or Cl on a mixture of I<sub>2</sub>O<sub>5</sub> and starch (reflected light). The change produced is indicated by a light-sensitive cell, and by means of suitable electrical devices a warning mechanism may be operated. C. JEPSON.

**Disinfectant compositions.** A. SCIVER and E. L. JACKSON (B.P. 349,479, 20.12.29).—A disinfectant to be used also as a sealing liquid for a chemical closet consists of an emulsion of fuel oil, neutral tar oils, cresols, or similar tar acids insol. in H<sub>2</sub>O, and an aq. dispersion of a protein, *e.g.*, glue. W. G. CAREY.

**Combating or exterminating animal or insect pests.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,685, 7.11.29).—Grain weevils are destroyed by vapour of alkyl sulphites at a dilution of 2–10 mg./litre; sulphites of ethyl (b.p. 158°), propyl (b.p. 194°), isopropyl (b.p. 70°/15 mm.), isobutyl (b.p. 96°/15 mm.), β-chloroethyl (b.p. 137°/15 mm.), and glycol (b.p. 66°/16 mm.) are mentioned. C. HOLLINS.

**Dechlorination of chlorinated water.** J. MUCHKA (B.P. 349,781, 30.5.30. Austr., 14.8.29).—C used for dechlorination is circulated in the apparatus by a nozzle action caused by the entry of the chlorinated H<sub>2</sub>O and is sterilised by this H<sub>2</sub>O, an excess of Cl being used for sterilisation, and an excess of C for dechlorination. W. G. CAREY.

**Treatment of water.** F. D. C. ALLEN (B.P. 348,654, 15.2. and 10.11.30).—Water which has been chlorinated is mixed with an indicator and flows continuously through a glass-walled tank through which light passes before falling on to a photoelectric cell; the change in current thereby produced actuates a dosing valve and controls the rate at which the reagent is supplied to the bulk of the water. The current through the cell is amplified by a thermionic valve, and the dosing valve is provided with devices retarding its opening and closing. W. G. CAREY.

**Plant for treatment of sewage.** T. B. SIMS (B.P. 349,605, 27.2.30).—See U.S.P. 1,757,262; B., 1930, 1094.

**[Apparatus for] controlling atmospheric conditions in rooms etc.** HALL & KAY, LTD., P. KAY, and S. HALL (B.P. 349,429, 23.11.29).

**Respiration apparatus with automatic nutrient gas supply controlled by the lungs.** O. H. DRÄGER, Assee. of E. DRÄGER (B.P. 349,752, 6.5.30. Ger., 25.5.29).

**Liquid heating and storage. Filtration of liquids. Purification of feed water. Air filters and conditioning.**—See I.



# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

AUG. 14 and 21, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Industrial furnaces.** J. FALLON (*Fuel Econ. Rev.*, 1931, 10, 40—47).—Various types of metallurgical annealing furnaces are described and illustrated.

D. K. MOORE.

**The Rimmey fusion-test furnace.** G. B. REMMEY (*J. Amer. Ceram. Soc.*, 1931, 14, 358—364).—A portable  $O_2$ - $C_2H_2$ -fired furnace is described which can reach temp. up to cone 37 in an oxidising atm. Synchronised gas valves enable results to be reproduced within  $\frac{1}{4}$  of a cone error.

J. A. SUGDEN.

**Application of coke-oven surplus gas for boiler heating.** H. GUTSMANN (*Gas- u. Wasserfach*, 1931, 74, 561—562).—Two water-tube boilers (transporter grates) were modified to burn surplus gas by enlarging the combustion chambers and fitting four "torsion" gas burners in the lower front part of the chamber. Elimination of fuel and ash transport costs, decreased attack of the chamber walls, and increased working control led to greater efficiency. Secondary gas burners were required for superheating. Two similar boilers were fitted with four gas burners above the grate and behind the coal feed. Gas burned in amounts up to 10% of the total fuel consumption allowed the use of second-rate and waste fuel and gave increased outputs from the boiler of up to 20%. Heating with coke-oven gas costs little more than with coal, but it is considered warrantable only when the interests of producer and user of the gas are combined.

H. E. BLAYDEN.

**Prevention of boiler scale by trisodium phosphate.** P. KOEPEL (*Oesterr. Chem.-Ztg.*, 1931, 34, 97).—The use of  $Na_3PO_4$  for softening boiler feed-water effectively reduces the hardness to  $0^\circ$  by precipitating quantitatively the Ca, Mg, and Fe. The flocculent ppt. coagulates floating particles of dust and adsorbs any oil in the  $H_2O$ , thus preventing foaming. A slight excess of  $Na_3PO_4$  is advantageous in reducing corrosion of the boiler parts.

A. R. POWELL.

**Dangers of an imperfect water purification in steam plant.** V. D. I. BELANI (*Petroleum*, 1931, 27, 469—474).—Experiences of the excessive scaling of a boiler are given and the causes of scaling and its elimination are discussed. The Steinmüller water-purifying plant is described in detail. H. E. BLAYDEN.

**Measurement of smoke under industrial conditions.** W. A. WORDLEY (*Fuel Econ. Rev.*, 1931, 10, 89—96).—The Ringelmann chart is not satisfactory for measuring the colour of smoke. Holes, diametrically opposite, are cut in the chimney and rays of light from a lamp pass through the smoke on to a Radiovisor bridge. The varying resistance of the bridge, due to

the varying intensity of the light, is used to operate a warning bell when a predetermined limit is exceeded, and also to give a chart which may be graduated in terms of smoke opacity.

D. K. MOORE.

**Rapid method for the determination of the dew point of gases.** A. N. GABE (*Gas World*, 1931, 94, Coking Sect., 66—67).—A mirror is attached to the bulb of a thermometer which is supported in a stopper inside a glass tube open at the bottom and provided with a side tube and tap at its upper end. The tube is lagged with asbestos in which two windows are cut opposite the mirror (about midway between the ends of the tube) at  $90^\circ$  to each other. The gas to be tested is made to flow slowly from the tap through the tube and the light from one window is viewed through the other window by reflection from the mirror. The temp. of formation of mist on the mirror is observed. A semi-circular wire covered with chamois leather serves as an internal wiper for the windows. The dew point of clean blast-furnace gas can be determined in 15—20 min. The results are generally higher than those of gravimetric methods, but a correction factor may be used. A filter may be required in some cases.

H. E. BLAYDEN.

**Spectral colorimeter.** O. SPENGLER and E. LANDT (*Z. Ver. deut. Zucker-Ind.*, 1931, 81, 13—24).—Schmidt and Haensch have elaborated from the Stammer or Duboseq type of colorimeter an instrument by which comparison of one liquid with another or with a Stammer colour-glass can be carried out with monochromatic light of three different wave-lengths, viz., 610, 560, and 480 m $\mu$ . The authors recommend the instrument for technical sugar work.

J. H. LANE.

**Laboratory furnace for coal distillation.**—See II. **Determining  $O_2$  in gases.**—See VII. **Conservation of heat in the annealing of glass.**—See VIII. **Seitz deposited-fibre filter.**—See XVII.

See also A., July, 793, **Solubilities of gases in liquids.** 795, **Size of pores of ultra-filters.** 799,  $Na_2S_2O_3 \cdot 5H_2O$  as standard in thermometry.

### PATENTS.

**Reduction of dust losses from shaft furnaces.** G. EICHENBERG and N. WARK (B.P. 349,092 and Addn. B.P. 349,093, [A] 13.2.30, [B] 14.2.30. Ger., [A] 14.9.29, [B] 4.10.29).—(A) A fluid under pressure is caused to act in a direction countercurrent to that of the furnace gases at the point, or just before, where they leave the furnace, so as to produce changes in speed and direction of the gases. Atomised  $H_2O$  and/or gases may be used as the accelerating fluid. Swivelling double nozzles are described for simultaneous use of air and  $H_2O$ . (B) The

\* The remainder of this set of Abstracts will appear in next week's issue.

forced currents are intermittent or pulsating. A form of nozzle for  $H_2O$  alone is described. B. M. VENABLES.

**Heat accumulators.** RUTHSACCUMULATOR AKTIEBOLAG (B.P. 349,350, 1.7.30. Ger., 6.7.29. Addn. to B.P. 298,601; B., 1929, 625).—The method of agitating the contents of a tall steam accumulator, as described in the prior patent, is modified by the addition of a mixing injector by which a small supply of high-pressure steam draws a larger quantity of vapour from the upper part of the accumulator and injects the mixture into the  $H_2O$ . The quantity of this high-pressure steam is so small that an electric boiler may be used to produce it, and the quantity injected is so large that it may be admitted through the main charging pipe. The high-pressure steam valve and the non-return valve in the main charging pipe are interconnected mechanically or electrically, so that when one is open the other is closed.

B. M. VENABLES.

**Heat-interchanging apparatus.** HOLDEN & BROOKE, LTD., and R. W. BROOKE (B.P. 349,234, 11.4.30).—A form of exchanger having spiral flow of the outer fluid and longitudinal flow of the inner, adapted to be made of cast metal, is described. The inner passage may be contracted longitudinally by tapered radial fins.

B. M. VENABLES.

**Heat-exchanger intended particularly for heating water.** P. H. MELLOR (B.P. 348,075, 6.2.30).—The exchanger comprises a flat casing having sinuous passages formed between ribs on the inside of the casing and spikes on the outside in continuation of the ribs.

B. M. VENABLES.

**Tubulous heat exchangers particularly applicable to desuperheating steam.** SUPERHEATER CO., LTD. From SUPERHEATER Co. (B.P. 349,327, 6.6.30).—The apparatus comprises a number of U-tubes of which the lower parts are submerged in  $H_2O$ , the vapour from which is allowed to mix with the steam partly as it enters and partly as it leaves the tubes.

B. M. VENABLES.

**Separator.** E. S. ROYER and R. J. O'DONNELL (B.P. 348,836, 14.4.30).—An apparatus for such purposes as reclaiming foundry sand is constructed with a sloping bed formed of intercalating discs rotating in the same direction. The shafts carrying the discs are capable of being moved nearer or further apart to vary the openings, and fingers between the discs are provided on the underside.

B. M. VENABLES.

**Strainers and filters for liquids and gases.** T. W. ELEY (B.P. 349,145, 1.3.30).—The filter comprises a cylindrical coil of wire supported so that the filtrate may pass inwards between the turns. The outside halves of the wires are machined off so that flat surfaces and sharp corners are presented to the prefit.

B. M. VENABLES.

**Continuous filter drum.** I. G. FARBERIND. A.-G. (B.P. 349,296, 21.5.30. Ger., 21.5.29).—The apparatus is of the type in which cake is removed from a filter drum by a number of belts running over it and another drum which is provided with scrapers, forks, or other means of detaching the cakes. In this invention the belts are combined in one continuous narrow length, the axes of the drums are not quite parallel, and a pair

of idle pulleys are provided to bring the belt from one end of a drum to the other and to tension it.

B. M. VENABLES.

**Apparatus for (A) controlling the flow of fluids, (B) indicating the rate of flow of fluids.** PATERSON ENG. CO., LTD., and W. SMALLEY (B.P. 349,569 and 349,631, [A] 19.2.30, [B] 3.12.29).—(A) The control desired, e.g., the addition of another fluid in strictly proportional quantity, is effected by a lever the other arm of which sustains a weight which is either slidable under the action of floats, of which the level is varied by connexion to a Venturi tube, a cam being introduced to secure movement of the weight proportional to the flow of the main liquid, or, if a V-notch is used to produce a variable head of the main fluid, the weight may take the form of a vessel connected to the main flow above the weir and shaped so that its content of liquid is proportional to the flow. (B) The bulk of the fluid is passed through a Venturi tube and a minor flow is permitted through a by-pass connected to the Venturi, and is measured by a known form of indicator comprising a plunger in a tapered or slotted tube. B. M. VENABLES.

**Production of aqueous dispersions.** A. L. CLAPP, ASSR. to BENNETT INC. (U.S.P. 1,787,338, 30.12.30. Appl., 8.9.25).—Regulated streams of molten thermoplastic material, e.g., bitumen, waxes, or resins, and a hot aq. dispersing liquor, e.g., alkali silicate, are mixed in an elongated mixing chamber and chilled to the congealing point before leaving the chamber.

C. A. KING.

**Furnace walls.** F. B. BIGELOW (B.P. 351,349, 26.9.30. U.S., 7.10.29).

**Insulating coverings for steam and similar heated pipes.** CONCENTRIC AIR CELLS AUTOMATIC CO. (B.P. 350,956, 14.1.30. U.S., 30.1.29).

**Storage and transport vessels for liquefied gas.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 351,158, 29.4.30. Fr., 30.4.29).

**Boiler tubes.**—See X. Gas purifier.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Chemical and physical survey of coal seams.** J. E. CHRISTOPHER, D. R. WATTLEWORTH, and G. V. CARTER (Fuel, 1931, 10, 257—267).—Some Cumberland seams have been examined with special reference to their use in by-product coke ovens. The distribution of the banded constituents is shown graphically, and analyses of certain of the seams are tabulated. The seams high in durain gave strong and dense cokes. Float-and-sink and Henry tube tests have been carried out on the coals, and integrated ash-, S-, and P-content curves have been constructed, as well as charts showing the distribution of true coal, middlings, and dirt in the different sized fractions of the mine sample. From the data obtained it was possible to select seams or districts suitable for the specific requirements of the blast furnace.

A. B. MANNING.

**Sampling of small coal.** E. S. GRUMELL and A. C. DUNNINGHAM (Fuel Econ. Rev., 1931, 10, 4—8).—The number of wagons to be sampled to control deliveries and for the determination of the average calorific val.,

in the case of large and small plants, is given. This number is related to the average characteristic error of each coal. Each coal has a nearly const. calorific val., calc. on the dry ash-free basis, and this obviates frequent calorific determinations. D. K. MOORE.

**Significance of solvent analysis as applied to coal.** E. B. KESTER (Fuel, 1931, 10, 277—285; U.S. Bur. Mines Inform. Circ. No. 6486).—The investigations carried out up to the present on the solvent analysis of coal are critically surveyed. The desirability of attempting to correlate the results obtained by different systems of solvent analysis is emphasised. Such an investigation is being carried out on American coals at the Pittsburgh Experiment Station, U.S. Bureau of Mines. A. B. MANNING.

**Importance of the degree of fineness of admixtures to coking coal.** H. HOCK (Glückauf, 1931, 636; Fuel, 1931, 10, 254—256).—The effect of mixing a material, *e.g.*, dull coal, fusain, etc., with a coking coal for the purpose of lowering its swelling capacity and expansive pressure is considerably greater the finer the material is ground. A tentative explanation of this phenomenon is suggested, based on the consequent increased permeability of the mass to the escaping volatile products. Uniform mixing of the materials is essential in order to attain the max. effect. A. B. MANNING.

**Utilisation of the sensible heat of the coke and the distillation gases in coke-oven operation.** P. STOLLER (Brennstoff-Chem., 1931, 12, 212—213, 232—234).—The "Collin" system is described. The coke is discharged into cooling chambers through which flue gases are circulated. The gases leaving the chambers are burned with the requisite amount of air, passed to the waste-heat boiler, and, after passing through a cooling conduit, are returned to the cooling chambers. The coke is cooled to 250° in 6—7 hr., and the steam production amounts to 390 kg. per ton of coke. The coke is of better quality than when quenched wet. The ascension pipes are provided with boiler-like jackets, whereby the sensible heat of the gases is utilised for the production of steam, and the quality of the tar is improved. A. B. MANNING.

**Determination of the expansion pressure exerted in laboratory and large-scale coking tests.** H. KOPPERS and A. JENKNER (Glückauf, 1931, 67, 353; Fuel, 1931, 10, 232—239, 273—277. Cf. B., 1931, 373).—The laboratory determinations were carried out in an apparatus, developed from that used by Korten and Damm, in which the vol. of coal is maintained const. and the variation in pressure recorded by a hydraulic arrangement attached to the same lever as the piston in the coal crucible. A specially constructed coke-oven wall, 4.5 m. high and 2 m. long, was damaged when subjected to lateral pressures as low as 0.1 kg. per sq. cm. A coke-oven chamber with movable heating walls was constructed, one wall being provided with a hydraulic measuring apparatus for determining the pressure, or the expansion under const. pressure, produced during carbonisation. Tests carried out with this apparatus show that the expansive pressures determined in the laboratory are far higher than those

which actually occur in the oven. This is accounted for by the difference in the conditions of carbonisation, and especially by the difference in the relative widths of the plastic layer and its adjacent layers of coal and coke. Some coals which in the laboratory show a temporary expansion followed by a shrinkage have shown no expansive pressure in large-scale tests even with charges of higher bulk density. Except for such coals the large-scale and laboratory tests gave roughly parallel results. In particular, the large-scale results confirm the laboratory results: that the higher the bulk density the greater is the expansive pressure. The time of carbonisation has little influence on the expansive pressure, and the difficulties in oven operation observed in some plants with shorter carbonising times are due to reduced shrinkage of the coke following the expansion. The expansion pressure exerted by a coal can be reduced by suitable blending, especially with a dull coal. A. B. MANNING.

**Regeneration of bone charcoal.** N. S. VOLKOV (Nauk. Zapiski Tzuk. Prom., 1930, 10, 294—307).—A discussion of the washing process.

#### CHEMICAL ABSTRACTS.

**Rate of evolution of gas from coke.** G. E. FOXWELL (Fuel, 1931, 10, 285—287).—A sample of coal (20 g.) was heated to a definite coking temp. (700°, 800°, or 900°) and the subsequent rate of gas evolution from the coke at const. temp. was determined. After about the first hr. the rate of gas evolution can be represented by the equation for a unimol. reaction. It is concluded that the rate of evolution is dependent on the surface area of the coke, and that such measurements may be utilised to distinguish between different types of coke structure. A. B. MANNING.

**Rapid generation of water-gas.** L. V. POLONSKI (J. Chem. Ind. Russ., 1931, 8, 485—490).—The addition of 5% of  $\text{Fe}_2\text{O}_3$  or  $\text{Na}_2\text{CO}_3$  increases the velocity of generation of water-gas from coke tenfold, and of 5% of CaO about fivefold; the quality of the product is unaffected. R. TRUSZKOWSKI.

**Influence of various forms of carbon on water-gas formation.** B. NEUMANN, C. KRÖGER, and E. FINGAS (Gas- u. Wasserfach, 1931, 74, 565—572).—The action of steam, alone or diluted with  $\text{N}_2$ , on wood charcoal, graphite, graphite + 8% of  $\text{Fe}_2\text{O}_3$ , C black (from  $\text{C}_2\text{H}_2$ ), and activated C, at temp. up to 1000° has been studied quantitatively. In the experiments with  $\text{N}_2$  as a diluent a const. stream of  $\text{N}_2$  (1.3 litres/hr.) was saturated with  $\text{H}_2\text{O}$  vapour at a definite temp. and passed through a superheater to the reaction tube (porcelain tube, 60 cm. long and 16 mm. in diam., heated electrically). The C was packed in the central 20 cm. of the tube. In each experiment the gases were passed for 1 hr., the excess steam and the  $\text{CO}_2$  formed were absorbed and weighed, and the vol. and composition of the residual gas were determined. The initial temp. of reaction, *i.e.*, the temp. at which formation of  $\text{CO}_2$  was first observed, was: wood charcoal 500°, C black 450°, graphite 730°, graphite +  $\text{Fe}_2\text{O}_3$  660°. At temp. in the neighbourhood of those at which the reaction began, small quantities of  $\text{O}_2$  (and with wood charcoal some  $\text{C}_2\text{H}_4$ ) appeared in the products. At

higher temp. appreciable quantities of  $\text{CH}_4$  were formed. With graphite the % of steam decomposed at  $810^\circ$  and  $987^\circ$  were 7.1 and 95, respectively. With graphite +  $\text{Fe}_2\text{O}_3$  12.0% of steam was decomposed at  $810^\circ$ . The following methods of obtaining a const. current of steam were tried: (a) combustion of a const. current of  $\text{H}_2$  over  $\text{CuO}$  or  $\text{PbO}_2$ , (b) combination of a  $\text{H}_2$ - $\text{O}_2$  mixture ("Knallgas") over suitable contact substances, and (c) evaporation of  $\text{H}_2\text{O}$  in an "adiabatic flask," i.e., a flask with a capillary tube outlet, heated externally by the vapour of boiling  $\text{PhMe}$ . The first method was unsatisfactory, but either of the others yielded a sufficiently const. slow current of steam (about 1 g./hr.). The initial temp. of reaction of graphite in steam alone was  $643^\circ$ ; at  $973^\circ$  95.9% of the steam was decomposed, producing a gas consisting of  $\text{CO}_2$  2.44,  $\text{CO}$  46.7,  $\text{H}_2$  47.5,  $\text{CH}_4$  3.36%,  $\text{O}_2$  nil. A graphical method of representing the results is described.

A. B. MANNING.

**Conversion of semi-coking gas.** N. A. KLUKVIN and S. S. KLUKVINA (J. Chem. Ind. Russ., 1931, 8, 337—342).—The gas obtained from the semi-coking of Moscow lignite has 5.2—8.6%  $\text{H}_2\text{S}$ , 13—19%  $\text{CO}_2$ , 3—8%  $\text{C}_n\text{H}_{2n}$ , 0.8—3%  $\text{O}_2$ , 5—10%  $\text{CO}$ , 5.5—12%  $\text{H}_2$ , 27—31%  $\text{CH}_4$ , and 21.8—23.8%  $\text{N}_2$ . This gas is readily converted, by passing over coke with  $\text{H}_2\text{O}$  vapour at  $900$ — $950^\circ$ , into a gas containing 7.8%  $\text{CO}_2$ , 16.8%  $\text{CO}$ , 67.6%  $\text{H}_2$ , and 7.6%  $\text{N}_2$ , but no  $\text{H}_2\text{S}$ ,  $\text{C}_n\text{H}_{2n}$ ,  $\text{CH}_4$ , or  $\text{O}_2$ .

R. TRUSZKOWSKI.

**Gasification of water hyacinth (*Eichornia crassipes*).** H. K. SEN and H. N. CHATTERJEE (J. Indian Chem. Soc., 1931, 8, 1—7).—Water hyacinth containing 16%  $\text{H}_2\text{O}$  decomposes on heating to  $700$ — $980^\circ$  in a current of moist air giving a gas of calorific val. 142 B.Th.U. in yield of 40,000 cu. ft./ton, the residue containing about 2 cwt.  $\text{KCl}$  (cf. A., 1929, 1348). Bacterial fermentation at  $32^\circ$  forms a gas of calorific value 602 B.Th.U., containing  $\text{CH}_4$ ,  $\text{H}_2$ , and  $\text{CO}_2$  in 50—70, 3—25, and 22—25% proportions, respectively, according to the period of fermentation. Pure cellulose yields a similar gas containing more  $\text{CH}_4$  on fermentation under similar conditions.

G. DISCOMBE.

**Use of non-corroding steel tubes in Jäger's method of determining nitrogen [in coal gas etc.].** W. ZWIEG (Gas- u. Wasserfach, 1931, 74, 576).—Owing to the rapid deterioration of the  $\text{Si}$  tubes which contain the  $\text{CuO}$ , it is recommended that they be replaced by tubes of Krupp's  $\text{NCT}_3$  steel, which show no appreciable deterioration after 1 year's use.

A. B. MANNING.

**Continuous-action rotary laboratory furnace for low-temperature dry distillation of coal.** W. SWIENTOSLAWSKI and H. NARKIEWICZ (Przemysl Chem., 1931, 15, 217—223).—A furnace adapted for temp. up to  $700^\circ$  is described.

R. TRUSZKOWSKI.

**Benzol refining.** M. KREMER (Petroleum, 1931, 27, 459—464).—Methods applied for the purification of crude benzol are discussed. Refluxing gas-works' benzol with 1—3% of anhyd.  $\text{AlCl}_3$  and washing, drying, and distilling removes most of the harmful unsaturated hydrocarbons and gives a product suitable for motor spirit (as indicated by the Br val. and  $\text{H}_2\text{SO}_4$  test). Refluxing with larger amounts (up to 10%) of  $\text{AlCl}_3$

gives benzol practically free from undesirable unsaturated compounds. The various fractions obtained after treatment with  $\text{AlCl}_3$  and distilling are more uniformly suitable for motor spirit than before treatment. It is suggested that  $\text{AlCl}_3$  reduces refining losses by inducing polymerisation of diolefines etc. to benzene hydrocarbons. Comparison of the action of  $\text{AlCl}_3$ ,  $\text{FeCl}_3$ , and  $\text{ZnCl}_2$  on the benzol showed  $\text{AlCl}_3$  to be the most active and  $\text{ZnCl}_2$  the least. The action of  $\text{AlCl}_3$  on brown-coal tar benzol was investigated.

H. E. BLAYDEN.

**Elimination of hydrogen sulphide from [gas-works] ammoniacal liquor by copper sulphate, and regeneration of the latter.** M. A. MINIOVITSCH and J. F. DISHEVSKI (Ukrain. Chem. J., 1930, 5, [Tech.], 193—203).—Addition of  $\text{CuSO}_4$  to the liquor gives the following ppts.:  $\text{CuS}$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{Cu}(\text{OH})_2$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{CuS}$ ,  $\text{Cu}_2\text{S}$ ,  $\text{Cu}_2(\text{OH})_2\text{CO}_3$ ,  $\text{CuCO}_3$ , and other complex salts. The ppt. on boiling with  $\text{H}_2\text{O}$  yields a mixture of sulphides, carbonate, and hydroxide of  $\text{Cu}$ ,  $(\text{NH}_4)_2\text{SO}_4$  passing into solution. The residue on roasting in air is converted into oxide, the  $\text{SO}_2$  evolved being absorbed by  $\text{NH}_3$ .  $\text{CuO}$  is converted into  $\text{CuSO}_4$  by dissolution in  $\text{H}_2\text{SO}_4$ .

R. TRUSZKOWSKI.

**Absorption and retention of hydrocarbons by solid fuels. II.** B. MOORE (Fuel, 1931, 10, 244—253. Cf. B., 1927, 593).—The rates of absorption of  $\text{C}_n\text{H}_{2n+2}$  (where  $n = 5$ —10), from air charged with the vapour at the ordinary temp., by a bituminous coal, an anthracite, and a coke, respectively, and the rates of loss of the hydrocarbon on subsequent exposure of the saturated material to the air, have been determined. In general, the rate of absorption increased with decrease in mol. wt. of the hydrocarbon. Most of the absorbed hydrocarbon was easily removed, but a measurable amount was retained by the fuel even after prolonged exposure to the air. The hydrocarbon so retained slightly raised the spontaneous ignition temp. of the fuel, but did not affect its capacity for absorbing  $\text{H}_2\text{O}$  vapour.

A. B. MANNING.

**Determination of benzene and *n*-hexane contents in a mid-continent petroleum.** J. H. BRUN and M. M. HICKS-BRUN (Bur. Stand. J. Res., 1931, 6, 869—879).—A crude Oklahoma petroleum contained 0.08% of  $\text{C}_6\text{H}_6$  and about 0.3% of *n*-hexane.

R. CUTHILL.

**Distillation of petroleum.** R. FUSSTEIG (Petroleum, 1931, 27, 520—522).—The "Weymann" method, which permits continuous distillation and simultaneous cracking of hydrocarbons, is described. The advantages of the process are that no crude oil or cooled residuum from the distillation can enter the cracking process, considerably higher yields are obtained, and carbonisation of the tubes etc. is diminished.

E. DOCTOR.

**Suitability of nickel catalysts for the synthesis of benzene.** F. FISCHER and K. MEYER (Brennstoff-Chem., 1931, 12, 225—232).—An efficient  $\text{Ni}$  catalyst has been prepared using  $\text{ThO}_2$  as a promoter. The mixed  $\text{Ni}$  and  $\text{Th}$  nitrates were dissolved in  $\text{H}_2\text{O}$ , kieselguhr was added, and the metals were precipitated as carbonates; the product was then washed, dried, and reduced with  $\text{H}_2$  at  $450^\circ$ . The optimum results were obtained with 18% of  $\text{ThO}_2$ , calc. on the wt. of  $\text{Ni}$ , and

a quantity of kieselguhr equal to the wt. of Ni.  $K_2CO_3$  was the best precipitating agent, and the catalyst was more effective if it was not washed completely free from K salts. The highest yield obtained with one passage of the gas (26%  $CO$ , 56%  $H_2$ ) over the catalyst (at  $178^\circ$ ) was 120 c.c. of liquid hydrocarbons per cu. m. Of the total  $CO$  consumed, 65% was converted into liquid and 35% into gaseous hydrocarbons. The catalyst was very stable; after 5 weeks' uninterrupted use the yield fell by only 17%. A very efficient catalyst was produced also by replacing the Th by Mn, and the Ni-Mn catalyst was further improved by a slight addition of Al. A. B. MANNING.

**Importance of ageing for transformer and switch oils.** F. EVERS (*Z. angew. Chem.*, 1931, **44**, 323—326).—A good mineral oil for electrical insulation should age at such a rate that its original saponif. val. of 2.0 increases by 0.1 or less per annum. By using the apparatus described previously (B., 1930, 801), with 60 g. of a catalyst comprising  $SiO_2$  gel impregnated with 1 mol.-%  $Fe_2O_3$  and 15.5 g. of oil, and maintaining the manometer at a const. value by introducing electrolytic  $O_2$  from a cell working at a const. rate for 100 min., the rate of ageing of the oil can be determined; under these conditions the  $O_2$  absorption in the test is approx. equal to that absorbed by the oil in a year under normal working conditions, and hence is a measure of the ageing rate of the oil. A. R. POWELL.

**Core oils [for foundries].** E. BRÜHL (*Chem.-Ztg.*, 1931, **55**, 442—443).—The various properties of an oil which render it of value for binding sand cores in the foundry are discussed. A. R. POWELL.

**Water content of power-generating substances and its determination.** M. KREMER (*Petroleum*, 1931, **27**, 443—445).—To determine  $H_2O$  from the clarification point is not possible in all cases. Henley's method (B., 1920, 501 A) is satisfactory for determining up to 0.05%  $H_2O$  in EtOH. In any mixture of petrol or benzol with EtOH, 0.2—0.6%  $H_2O$  can easily be ascertained. The Adickes method (A., 1931, 61) serves the same purpose, but as yet has not been applied to EtOH-hydrocarbon mixtures. E. DOCTOR.

**Boiler heating. Dew point of gases.**—See I.  $(NH_4)_2SO_4$ .  $C_2H_2$  in  $CaC_2$ . Determining  $O_2$  in gases. —See VII. Porcelain firing. —See VIII. Examinations with ultra-violet light. —See XI.

See also A., July, 793, Solubilities of gases in liquids. 796, Colloidal C. 804, Decomp. of  $H_2O$  vapour on C. 812, Volumetric determination of  $CO$ . 856, Identifying a mixture of hydrocarbons.

#### PATENTS.

**Manufacture of mixed oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 349,376, 30.7.30. U.S., 3.9.29).—A carburetted water-gas generator is operated in the following steps: oil is vaporised and lightly cracked in the carburettor and superheater, and the resulting gases and vapours are passed through the ignited fuel bed, wherein they are cracked with the deposition of C; the fuel bed is then air-blasted and steamed for the production of blue

water-gas, part of which is burned, together with the blow gases, in the carburettor and superheater. In this manner the desired balance of temp. conditions is maintained and the excess C deposited in the fuel bed is progressively consumed. A. B. MANNING.

**Distillation of tar and like hydrocarbons.** BARRETT Co., Assees. of S. P. MILLER (B.P. 349,088, 18.12.29. U.S., 18.12.29).—The hot gases from a number of individual coke ovens are mixed in a heat-insulated header or collecting pipe, and the mixed gases are passed into a still into which tar is sprayed or atomised at such a rate that a rapid heat exchange takes place between the gases and tar. The pitch residue is withdrawn continuously from the still while the gases and vapours are passed to a condenser. A. B. MANNING.

**Apparatus for distillation of hydrocarbon oils.** C. C. MILLER, Assr. to STANDARD OIL Co. (U.S.P. 1,786,357, 23.12.30. Appl., 15.6.27).—Cracked oil leaving a pipe still is fed to a large chamber, in which the heavy portions collect, at the base of a fractionating column. Operation is controlled by a valve, of special construction, between the outlet of the still and the condensing system, which regulates not only the flow of oil, but also the pressures at different points. R. H. GRIFFITH.

**Distillation of hydrocarbon oils.** G. W. WATTS, P. L. KRAUEL, and T. E. STOCKDALE, Assrs. to STANDARD OIL Co. (U.S.P. 1,784,561, 9.12.30. Appl., 3.4.25).—Heavy oil is mixed with superheated steam at about  $450$ — $500^\circ$  and injected into a chamber so that it becomes atomised. The system is operated under reduced pressure and is attached to condensers in which oil fractions and  $H_2O$  are separated. R. H. GRIFFITH.

**Vacuum distillation of hydrocarbon oils.** H. C. WIESS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,786,639, 30.12.30. Appl., 25.9.25).—High-boiling oils are heated in a still, at a pressure below 50 mm., while the vapour of a naphtha fraction, boiling above  $100^\circ$ , is injected into the still. Lubricants and other valuable products are thus recovered at a reasonable rate without overheating of the material. R. H. GRIFFITH.

**Fractional extraction of petroleum hydrocarbons with alcohol.** T. A. WERKENTHIN, Assr. to SOLAR REFINING Co. (U.S.P. 1,783,203, 2.12.30. Appl., 30.12.27).—High-boiling hydrocarbons, especially distillation residues, are extracted with EtOH for the recovery of lubricants or waxes. The temp. of operation varies from  $40^\circ$  to  $78^\circ$ , but the solvent may be introduced as vapour. On cooling, the mixture separates into two layers and the alcoholic portion is worked up by further cooling. No distillation of the EtOH is necessary before it is used again. Traces of solvent left in the oil or wax have no deleterious effects. R. H. GRIFFITH.

**Decolorisation and stabilisation of distillate petroleum products with respect to colour and odour.** STANDARD OIL Co. OF NEW YORK (B.P. 348,011, 29.10.29. U.S., 29.10.28).—Petroleum oils discoloured by internal pigmentation are improved and stabilised by the addition of reducing agents such as hydroxybenzenes (e.g., quinol, orcinol, pyrogallol), hydroxylated

$C_{10}H_8$  (e.g.,  $\alpha$ - or  $\beta$ -naphthol), phenylhydrazine,  $Sn^{++}$  oleate,  $SnCl_2$ ,  $MeONa$ , etc. If the reducing agent is solid it is dissolved in an aliphatic alcohol ( $EtOH$  or  $BuOH$ ). One pt. by wt. of reagent is used to treat 50–100,000 pts. of oil. [Stat. ref.] T. A. SMITH.

**Purification of hydrocarbon oils.** E. B. HUNN, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,786,246, 23.12.30. Appl., 1.7.27).—Hydrocarbon oils are "sweetened" by agitation with a solution of  $PbO$  in  $NaOH$ , separating the oil, agitating with  $Me$ ,  $Et$ , or  $Pr$  disulphide or a mixture of these, and filtering. When oils of high S content are to be treated a quantity of S insufficient to ppt. the contaminating  $Pb$  is added before treatment with alkyl disulphide.

H. E. BLAYDEN.

**Treating hydrocarbon oil.** J. M. GOODWIN, ASSR. to TEXAS CO. (U.S.P. 1,786,650, 30.12.30. Appl., 16.3.28).—Petroleum naphtha (from which undesirable fractions of low b.p. may be removed by rectifying) is stabilised and purified by dissolving in it small amounts of  $H_2S$ , treating with cone.  $H_2SO_4$ , and washing.

H. E. BLAYDEN.

**Cracking of oils, tars, and the like.** S. SEELIG (B.P. 348,242, 20.2.30).—Hot oil (etc.) after passing under pressure through a cracking tube is sprayed under released pressure on to a hot surface in a vaporiser. The hot surface is treated either by hot flue gases or by hot oil from the cracking plant before the oil spray enters the vaporiser.

T. A. SMITH.

**[Petroleum] oil cracking.** J. H. WELCH (U.S.P. 1,784,562, 9.12.30. Appl., 6.7.27).—Vapours obtained by heating hydrocarbons in a cracking apparatus are bubbled through a further quantity of heated oil. The products are separated centrifugally from entrained liquid before fractional condensation. The primary oil-heating tubes and the vertical reaction vessel are assembled so that they can all be heated from a single furnace.

R. H. GRIFFITH.

**Cracking of petroleum oil.** R. T. POLLOCK, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,783,259, 2.12.30. Appl., 14.11.29).—Oil is cracked under pressure at a raised temp. and the high-boiling residue is passed on, after removal of light products, to a second cracking stage at a higher temp. and pressure.

R. H. GRIFFITH.

**Converting [cracking of] hydrocarbons.** J. C. MORRELL and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,783,257, 2.12.30. Appl., 17.7.24).—Deposition of solid on the walls of the cracking chamber is prevented by forcing a stream of compressed light oil on to the surface of the residual oil so that the whole vessel is swept by a stream of liquid.

R. H. GRIFFITH.

**Cracking of [hydrocarbon] oils.** R. C. HOLMES and F. J. MANLEY, ASSRS. to TEXAS CO. (U.S.P. 1,786,947, 30.12.30. Appl., 9.4.26).—Oil under treatment passes in series through a number of vertical converters, which are heated in their central portions and of which the first is at the highest working temp. The level of oil is maintained above the top of the heated zone, and heavy residue is withdrawn from the last vessel to be returned for further cracking.

R. H. GRIFFITH.

**Cracking of hydrocarbons.** E. W. BEARDSLEY and M. W. COLONY, ASSRS. to PETROLEUM CONVERSION CORP. (U.S.P. 1,784,126, 9.12.30. Appl., 28.6.27. Cf. U.S.P. 1,715,239; B., 1929, 744).—Oils are cracked in the vapour phase by contact with preheated hydrocarbon gases; means are provided for thorough heat exchange between raw material, circulating gas, and reaction products.

R. H. GRIFFITH.

**Apparatus for cracking hydrocarbons.** W. MILLER, ASSR. to CONTINENTAL OIL CO. (U.S.P. 1,785,042, 16.12.30. Appl., 8.6.27).—A fraction of intermediate b.p., obtained from a cracking plant, is separated in a dephlegmator and is not returned to the cracking zone, but is employed as fuel oil. As this fraction is the most difficult to crack, the procedure increases throughput and prolongs the life of the plant.

R. H. GRIFFITH.

**Cracking of hydrocarbons.** SINCLAIR REFINING CO., ASSEES. OF H. L. PELZER (B.P. 348,764, 11.3.30. U.S., 14.5.29).—A stream of high-boiling oil is heated to cracking temp. under atm. pressure and the products are passed into a coking vessel. A stream of still higher-boiling oil is heated to a lower cracking temp. under pressure and then passed into the same coking vessel with reduction in pressure. Vapours from the coking vessel are then passed into a supply of oil in a scrubbing vessel, the unvaporised oil being returned to the coking vessel and reduced to coke. By further fractionation of the vapours discharged, gasoline may be obtained.

T. A. SMITH.

**Apparatus for producing lower-b.p. hydrocarbons.** C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,783,185, 2.12.30. Appl., 4.11.18. Renewed 9.6.26).—Oil to be cracked is circulated rapidly, under pressure, through a heating coil and passes thence to an expansion chamber. In this its velocity is greatly reduced and low-boiling products are withdrawn; heavy residues are returned to the furnace and carbonaceous deposits are drained off as a sludge.

R. H. GRIFFITH.

**Mineral oil composition.** C. F. KAEGBEHN, ASSR. to R. T. VANDERBILT CO., INC. (U.S.P. 1,784,359, 9.12.30. Appl., 31.1.29).—The decomp. of mineral oils due to air etc. is inhibited by addition of 0.2–0.5% of 2- or 4-hydroxydiphenyl.

H. E. BLAYDEN.

**Motor fuel.** F. J. KOBLITZ (B.P. 347,132, 22.1.30).—About 1–5% of a dope consisting of a mixture of  $NH_2Ph$ ,  $C_{14}H_{10}$ , camphor, castor oil,  $C_{10}H_8$  and its tetra-, hexa-, and deca-hydro- and nitro-derivatives, picric acid,  $Et_2SO_4$ , and cresol is added to the fuel. The dope may be improved by the addition of such substances as  $COMe_2$ , quinol, acetaldehyde, etc.

T. A. SMITH.

**Production of non-knocking motor spirit.** D. A. HOWES, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,380, 28.2.30).—The waste gases (preferably desulphurised) produced in the destructive hydrogenation of carbonaceous material are pyrolysed to produce aromatic substances. The products of pyrolysis of the gas are added to the oil obtained by hydrogenation, and may also be utilised as a source of  $H_2$  for the hydrogenation process.

T. A. SMITH.

**Production of lubricant.** N. C. BEIM (U.S.P. 1,784,809, 16.12.30. Appl., 19.3.28).—Lubricating oil

for internal-combustion engines is prepared by inter-mixing two commercial grades of lubricating oils by injecting them under pressure into a closed, steam-heated chamber provided with baffles, heating the mixture under reduced pressure to about 218° to remove volatile fractions, mixing with approx. 0.3% of Mg stearate, and filtering the product. H. E. BLAYDEN.

**Obtaining [oil-soluble] mineral oil sulphonates.** W. T. REDDISH and L. D. MYERS (B.P. 347,164, 18.10.29).—Lubricating oil stocks are treated with 5–10 wt.-% of oleum in the preparation of technical or medicinal white oils. After treatment the oils are extracted with 70% EtOH. The alcoholic extract, which contains all the sulphonic acids, is exactly neutralised and evaporated, pure sulphonates being obtained. The oil is free from sulphonic acids, EtOH, and H<sub>2</sub>O, and has good emulsifying and fat-splitting properties. T. A. SMITH.

**Manufacture of sulphonated conversion products of higher paraffin hydrocarbons.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,829, 11.9.29. Cf. B.P. 343,948; B., 1931, 578).—Paraffin hydrocarbons (above C<sub>8</sub>) are halogenated to introduce more than two halogen atoms per mol. and the product is treated with aqueous or alcoholic solutions or suspensions of alkali, alkali carbonates, etc., and finally with a sulphonating agent in the presence or absence of a diluent (*e.g.*, CCl<sub>4</sub>) or compounds which assist the reactions (*e.g.*, fatty acids or their anhydrides or chlorides). H. E. BLAYDEN.

**Production of [aqueous] bitumen emulsions.** T. NESS, LTD., W. A. WALMSLEY, and R. H. THOMPSON (B.P. 350,744, 24.6.30).—Alkali salts of di- or tri-hydric phenols (*e.g.*, resorcinol or pyrogallol), of  $\alpha$ - or  $\beta$ -naphthol, or of carbazole, with the addition of soap etc., if desired, are used as emulsifying agents. L. A. COLES.

**Manufacture of [bitumen] dispersions.** R. W. LEWIS (U.S.P. 1,783,365—6 and 1,787,418, [A, B] 2.12.30, [C] 30.12.30. Appl., [A] 28.2.27, [B] 12.4.28, [C] 27.5.25).—(A) Portland cement with or without colloidal Al(OH)<sub>3</sub> forms the dispersion agent for bitumen and H<sub>2</sub>O. (B) The dispersion consists of bitumen 9 pts., Al(OH)<sub>3</sub> 1–3 pts., and clay 1 pt., in H<sub>2</sub>O. (C) A dispersion agent is used which may be formed in or wetted by H<sub>2</sub>O, but which after drying will adhere to the dispersed bitumen particles, *e.g.*, gelatinous metallic oxides. Such a dispersion in H<sub>2</sub>O may contain bitumen 80%, Al(OH)<sub>3</sub> paste 80%, asbestos 10%. C. A. KING.

**Checker blocks or the like fillers for regenerative coke ovens.** GAS CHAMBERS & COKE OVENS, LTD. From COLLIN & Co. (B.P. 351,365, 17.10.30).

**Apparatus for charging carbonising chambers.** N.V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & COMP. G.M.B.H. (B.P. 350,781, 18.7.30. Ger., 20.7.29).

**Fuel burners.** J. F. HURLEY (B.P. 350,581, 20.8.30).

**Burners for firing hot-blast stoves, furnaces, and the like with gaseous fuel.** J. E. WEYMAN (B.P. 351,180, 10.5.30).

**Aq. dispersions.**—See I. Gas mixtures containing H<sub>2</sub>.—See VII. Recovery of Mo.—See X. Mn rosinate.—See XIII. Insecticides.—See XVI.

### III.—ORGANIC INTERMEDIATES.

**Compound catalysts for synthesis of methyl alcohol.** V. A. PLOTNIKOV, K. N. IVANOV, and D. A. POSPECHOV (J. Chem. Ind. Russ., 1931, 8, 472–478).—Cu has a feeble catalytic action in the prep. of MeOH from CO and H<sub>2</sub> under pressure; addition of ZnO slightly activates Cu. Cr<sub>2</sub>O<sub>3</sub> has a more powerful action at 265°, chiefly catalysing the reaction  $2\text{CO} + 2\text{H}_2 \rightarrow \text{CH}_4 + \text{CO}_2$ . Mixtures of Cu and Cr<sub>2</sub>O<sub>3</sub> are slightly more active than is Cu alone; the reaction proceeds at 265–285°, and the products contain up to 65% of MeOH. The activity of a catalyst containing Cu, ZnO, and Cr<sub>2</sub>O<sub>3</sub> is twice as great as that of one containing Cu and either ZnO or Cr<sub>2</sub>O<sub>3</sub>. The further addition of Ag, Na<sub>2</sub>CO<sub>3</sub>, or MgO to the catalyst depresses its activity. The activity of the mixtures CuO, MgO, Cr<sub>2</sub>O<sub>3</sub> and Cd, ZnO, Cr<sub>2</sub>O<sub>3</sub> is of the same order as that of Cu, ZnO, Cr<sub>2</sub>O<sub>3</sub>, whilst that of Ag, ZnO, Cr<sub>2</sub>O<sub>3</sub> is inferior. R. TRUSZKOWSKI.

**Determination of pentaerythritol.** M. J. KRAFT (J. Chem. Ind. Russ., 1931, 8, 507).—The solution containing pentaerythritol is strongly acidified with HCl, an equal vol. of PhCHO in EtOH is added, and the mixture is left overnight. The cryst. deposit of dibenzylidene-pentaerythritol is washed and dried to const. wt. R. TRUSZKOWSKI.

**Anthraquinonesulphonic acids.** I. Separation, identification, and quantitative determination. II. Fission of sulpho-groups from anthraquinone- $\alpha$ -sulphonic acids. III. Anthraquinone- $\beta$ -sulphonic acids. IV. Anthraquinone- $\alpha$ -sulphonic acids. V. Use of mercury in sulphonation of anthraquinone. VI. Influence of impurities on sulphonation of anthraquinone. VII. Influence of inorganic sulphates on sulphonation of anthraquinone. K. LAUER (J. pr. Chem., 1931, [ii], 130, 185–194, 194–197, 198–213, 214–225, 225–239, 239–248, 248–254).—Anthraquinone-2-sulphonic acid is best separated either from neutral or acid solution as its Na salt, which is washed free from acid with “2 : 1” (*i.e.*, two-thirds saturated) aq. NaCl, dried at 180°, and the NaCl determined by titration with AgNO<sub>3</sub>. The 1-sulphonic acid is separated and determined as K salt by a similar method. The purity of these salts is checked by conversion into the corresponding chloroanthraquinones. The arylamine method (A., 1923, i, 234) is not applicable to technical mixtures, but is of use in removing the bulk of sulphonic acids from sulphonation liquors before determination of free H<sub>2</sub>SO<sub>4</sub> as BaSO<sub>4</sub>. A technical  $\alpha$ -sulphonation mixture is best separated by conversion into K salts, and filtering at 60°, which gives pure 1-sulphonate; the filtrate is cooled, filtered, and the residue washed with 2 : 1 KCl, which removes disulphonic acids and leaves a residue of 1- and 2-sulphonates, which is converted into the chloroanthraquinones and analysed by a mixed-m.p. determination of the resulting mixture. The filtrate from the prep. of sodium anthraquinone-2-sulphonate contains all the 1- and some 2-sulphonate; these are isolated by Dünschmann's method (A., 1904, i, 326), and the mixed Na salts heated with 85% H<sub>2</sub>SO<sub>4</sub> and Hg at 180°, whereby anthraquinone is regenerated from its 1-sulphonic acid without the 2-sulphonic acid being affected. The main products of disulphonation,



the 2:6- and 2:7-disulphonic acids, are separated almost completely from by-products on salting out, and on washing the Na salts obtained with saturated aq. NaCl only a little of the latter is dissolved. They may readily be separated from each other by crystallisation from  $H_2O$ , but are best determined by conversion into the corresponding dichloroanthraquinones by Ullmann's method. Small amounts of the 1:6- and 1:7-disulphonic acids occur in all the above cases and are completely extracted, together with the small amounts of hydroxy-acids also formed, by 2:1 NaCl. By desulphonation with aq.  $H_2SO_4$  and Hg the 1:6- and 1:7-disulphonic acids are converted into the 2-sulphonic acid; a separate portion of the extract on chlorination gives a mixture of dichloro- and chlorohydroxy-anthraquinones from which the latter can be separated by aq. NaOH; by fractional crystallisation of the extracted materials the 1:4-derivative was isolated. Anthraquinone-1:5- and -1:8-disulphonic acids are by-products of  $\alpha$ -monosulphonation, and the principal products of  $\alpha$ -disulphonation. In the former case they are separated as already described, and are determined in part by conversion into dichloroanthraquinones, and in part by desulphonation. The disulphonation mixture gives the majority of the 1:5-disulphonic acid in a state of purity on salting out. The conversion of anthraquinonesulphonic acids into the corresponding chloroanthraquinones by Ullmann's method is reliable as an analytical method, and gives yields of 95–98% of the theoretical, but the presence of the salts of heavy metals must be avoided (cf. A., 1927, 463). New data are given for the solubility of Na or K salts of the above sulphonic acids in  $H_2O$  and various saline solutions, and also m.p.-composition curves for 1- and 2-chloroanthraquinones and 1:5- and 1:8-dichloroanthraquinones.

II. Treatment of anthraquinone-1-sulphonic acid with aq.  $H_2SO_4$  and Hg at 170–200° displaces the sulpho-group with regeneration of anthraquinone. There is no tendency to rearrangement to the 2-sulphonic acid, but at 210° the anthraquinone produced is converted into the 2-sulphonic acid.  $\alpha$ -Substitution of anthraquinone occurs normally, although to a limited extent as a result of the high temp., with oleum in presence of Hg, the normal yield of 2-sulphonic acid being also obtained; use of aq.  $H_2SO_4$  and Hg, or dilution of the oleum sulphonation at a sufficiently high temp., however, gives 2-sulphonic acid exclusively. Similar treatment of  $\alpha\beta$ -disulphonic acids removes the  $\alpha$ -groups alone, and the non-occurrence of  $\beta$ -sulphonation below 200° makes the method valuable for the determination of  $\alpha$ -sulpho-groups.

III (cf. A., 1927, 463).  $\beta$ -Monosulphonation is best effected with 20% oleum at 140° for 4 hr. Use of insufficient oleum for disulphonation leads always to a mixture of unchanged anthraquinone, mono- and di-sulphonic acids, sulphonation ending at a  $SO_3$  concentration of 2–3% at 140°. A high conversion into monosulphonic acid is therefore accompanied by a relatively low degree of total sulphonation. Disulphonation is best effected with a 10% excess of 40% oleum at 190°, a yield of 85% of the theoretical of 2:6- and 2:7-disulphonic acids in the ratio 1:1.15, accompanied by about 10% of 1:6- and 1:7-disulphonic and hydroxysulphonic acids, being

obtained. Lower temps. favour slightly the formation of the 2:7-disulphonic acid, but no conversion of 2:6- into 2:7-acid was observed. Increase of the reaction time or of the  $SO_3$  concentration produces oxidation at the expense of the disulphonic acids.

IV. Sulphonation in the  $\alpha$ -positions is carried out in presence of Hg, although it occurs to the extent of 2–3% in its complete absence. The best yields of monosulphonic acid (53% overall, or 75% on anthraquinone actually used) are obtained with a 5% excess of 20–25% oleum at 140° for 2 hr. More  $SO_3$  or a higher temp. causes disulphonation, but prolongation of the reaction time is without effect. As by-products are formed 2–3% of 2-mono- and probably also 1:6- and 1:7-di-sulphonic acids. Disulphonation is best carried out with 110–115% of the theoretical quantity of 40% oleum at 130° for 4 hr. The process of heating-up should be gradual. It yields approx. 47% of 1:5-, 27% of 1:8-, 11% of 1:7-, 5% of 1:6-disulphonic acids, and 2% of hydroxysulphonic acids. Formation of  $\alpha\beta$ -disulphonic acids appears to occur rapidly in the initial stages, but soon ceases; its extent is not reduced by use of more Hg, the best results being given by 1% of Hg or an equiv. amount of a salt.

V. Under otherwise equal conditions the  $\alpha$ -sulphonation of anthraquinone in presence of Hg is more rapid than  $\beta$ -sulphonation in its absence; the velocity increases with the concentration of Hg up to 1%, when  $HgSO_4$  begins to separate. The Hg used is combined almost exclusively with the unchanged anthraquinone and the sulphonic acids. At a sufficiently high temp. the  $\alpha$ -sulpho-group is split off, and  $\beta$ -sulphonation then occurs. In the sulphonation of recovered anthraquinone (with addition of Hg) an abnormally high proportion of disulphonic acid is formed; the same result is obtained if fresh anthraquinone is heated with Hg and  $H_2SO_4$  at 100° before sulphonation. On the other hand the 1-sulphonic acid, if carefully freed from Hg, gives on further sulphonation the 1:6- and 1:7-disulphonic acids. The existence of an  $\alpha$ -sulpho-group does not, therefore, render the other  $\alpha$ -positions liable to substitution, and the phenomenon of  $\alpha$ -substitution can be due only to the preliminary formation of  $\alpha$ -mercurianthraquinone-derivatives, and direct replacement of the metallic group by the entering sulpho-group.

VI. The presence of 1% or more of 2-methylantraquinone in anthraquinone renders the isolation of the 2-sulphonic acid difficult, and considerably reduces the yield. The product is difficult to filter on account of the presence of methylantraquinonesulphonic acids, and much of the desired substance remains in solution. The effect of paraffins, anthracene, and sucrose is to reduce the amount of free  $SO_3$  available for sulphonation, and so reduce the yield under normal working conditions, but no loss of anthraquinone occurs. Carbazole and dianthrone are without noticeable effect. Sulphonation of a technical anthraquinone of 96% purity gave a poor conversion and a low yield of isolated sulphonic acid, but again an abnormal amount of 2-sulphonic acid remained in solution. In this case also slightly better results were obtained with a greater excess of  $SO_3$ .

VII. Addition of inorg. sulphates in mol. quantities during the  $\beta$ -sulphonation of anthraquinone favours formation of the monosulphonic acid, but the tendency

to sulphonation and the degree of sulphonation are considerably less, and a higher temp., but less time, is needed. The degree of sulphonation decreases in the order  $\text{Na} > \text{K} > \text{Mg} > \text{Ca}$ , and the yield of monosulphonic acid increases, but very slightly, in the same order. The best yield of 2-sulphonic acid is obtained with a 40% excess of oleum; beyond this point disulphonation is considerable. Under suitable conditions complete disulphonation can be effected, and appears to be unaccompanied by the formation of  $\alpha\beta$ -disulphonic and hydroxy-sulphonic acids. Nevertheless, on account of the low degree of sulphonation, and the difficulties caused by the presence of inorg. salts during working up, it is considered that the process is without technical advantage.

H. A. PIGGOTT.

**Beyer's formula for the titrimetric determination of saccharin.** O. BEYER (Chem.-Ztg., 1931, 55, 509—510).—Saccharin and parasaccharin may be determined in admixture (*i.e.*, in commercial saccharin) by titration. Saccharin (%) =  $(2.01256 C - 100)/0.09868$ , where  $C$  = c.c. of 0.1N-KOH per g. of substance. Parasaccharin =  $100 - \% \text{ saccharin}$ . E. H. SHARPLES.

**Abs. EtOH.**—See XVIII.

See also A., July, 793, **System  $n$ -BuOH-MeOH- $\text{H}_2\text{O}$ .** 794, **Adsorption of  $\text{H}_2\text{O}$  from aq. EtOH.** 803, **Prep. of catalysts for hydrogenation.** 805, **Liquid partial oxidation.** 820, **Prep. of isoamyl ether.** 831, **Prep. of aliphatic amides.** 842, **Prep. of phenylglyoxal.** 844, **Derivatives of benzanthrone.** 852, **Triazole compounds.** 856, **Colour test for  $o$ -dihydroxyphenols.** Detection of  $\text{BzOH}$ . 877,  **$\text{COMe}_2$ -BuOH by fermentation.**

## PATENTS.

**Production of acetone from acetylene.** HOLZVERKOHLUNGS-IND. A.-G. (B.P. 347,695, 22.1.30. Ger., 24.1.29).— $\text{C}_2\text{H}_2$  and steam are passed through an Fe oxide catalyst layer about 10 cm. thick; *e.g.*, a series of Fe-gauze sheets superficially oxidised and impregnated with additional catalysts or promoters may be piled horizontally or rolled into a cylinder. C. HOLLINS.

**Production of ketones [from alcohols etc.].** HOLZVERKOHLUNGS-IND. A.-G. (B.P. 347,593, 22.1.30. Ger., 29.1.29).—Alcohols, aldehydes, esters, acids, acetals, and aldols above  $\text{C}_1$  are led with steam at  $500^\circ$  over a catalyst comprising oxides or carbonates of Ni and/or Co, preferably with Fe oxide, with or without oxides of Ca, Sr, Ba, or other light metals. EtOH with  $\text{H}_2\text{O}$  over rusted spongy Fe impregnated with NiO gives 88% yield of  $\text{COMe}_2$ . C. HOLLINS.

**Production of benzoylbenzoic acid derivatives and anthraquinone derivatives therefrom.** R. J. LOVELUCK, J. THOMAS, E. G. BECKETT, and SCOTTISH DYES, LTD. (B.P. 347,613, 17.10.29).—A dichlorobenzoylbenzoic acid, obtained from a dichlorophthalic anhydride and  $\text{C}_6\text{H}_6$ , is heated with  $\text{NH}_3$  or  $\text{NH}_2\text{Me}$  at  $120$ — $140^\circ$ , and the product is cyclised to give a chloro-aminoanthraquinone. 2-Chloro-1-aminoanthraquinone is prepared from 3:4-dichlorophthalic anhydride, the 2:3-isomeride and 2-chloro-3-methylaminoanthraquinone from the 4:5-dichloro-anhydride. C. HOLLINS.

**Production of halogenophthalic anhydrides.** P. F. BANGHAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 347,666, 24.12.29).—A salt, especially an acid salt, of 4-chloro(or other halogeno)-phthalic acid is heated with an equiv. of  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ ,  $\text{H}_2\text{O}$  is distilled off, and the anhydride is extracted, if desired, by solvents. 3- and 4-Chloro- and 3:4- and 3:6-dichloro-anhydrides are so prepared. C. HOLLINS.

**Mineral oil etc. sulphonates.**—See II.

## IV.—DYESTUFFS.

**Spectrophotometric measurements in the dye-stuffs industry.** C. Z. DRAVES (J. Opt. Soc. Amer., 1931, 21, 336—346).—The applications of the spectrophotometer, and its limitations, are discussed.

C. W. GIBBY.

See also A., July, 804, **Adsorption of org. dyes by  $\text{ZnO}$ .** 806, **Fastness to light of azo dyes.** 835, **Colour and constitution.** New mordant azo dye. 840, **Tetraphenylmethane derivatives.** 844, **Derivatives of benzanthrone.** 853, **Bilirubinoid dyes.**

## PATENTS.

**Manufacture of a vat dye [of the dibenzanthrone series].** SOC. CHEM. IND. IN BASLE (B.P. 347,681, 27.1.30. Switz., 25.1.29).—Aminodibenzanthrone is condensed with tetrachloro- or 2:5-dichloro-*p*-benzoquinone in  $\text{PhNO}_2$  at  $190$ — $200^\circ$  to give a blue-green vat dye fast to  $\text{Cl}_2$ .

C. HOLLINS.

**Manufacture of disazo dyes [for cotton and viscose silk].** SOC. CHEM. IND. IN BASLE (B.P. 347,742, 30.1.30. Switz., 2.2.29).—A diazo compound having no *o*- or *p*-OH group is coupled with a 1:2-aminonaphthol ether or its sulphonic acid, and the product is diazotised and coupled with J-acid or an aryl-J-acid, for blue cotton and viscose dyes. Examples are: aniline-*o*- or -*m*-sulphonic acid  $\rightarrow$  2-methoxy-Cleve acid  $\rightarrow$  phenyl-J-acid;  $\beta$ -naphthylamine-4:8-disulphonic acid  $\rightarrow$  2-ethoxy-Cleve acid  $\rightarrow$  J-acid. The dyes may be pre coppered etc.

C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Production of cellulose by means of chlorine.** IV. **Chlorolignin.** J. KAWAMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 173—174 B; cf. B., 1931, 581).—Increasing the concentration of  $\text{Cl}_2$  or repetition of chlorination has no great influence on the  $\text{Cl}_2$  content of chlorolignin or on its OMe content. Chlorination of the lignin hydrochloride obtained by the Kalb-Lieser method causes no removal of OMe groups. E. H. SHARPLES.

**Sulphite-cellulose from pinewood.** II. O. RUTALA and J. SEVÓN (Cellulosechem., 1931, 12, 131—137; cf. B., 1929, 592).—The addition of salts of weak acids to sulphite liquors enables woods high in resin content to be pulped satisfactorily. The liquors may even become alkaline during the digestion. Sulphite waste liquors are used as a cheap source of weak acids, Ca being precipitated by means of  $\text{Na}_2\text{SO}_4$ , NaOAc being the principal buffer salt formed. Formation of  $\text{H}_2\text{SO}_4$  during cooking is also minimised by the addition of

buffers. Experimental digestions of pinewood, employing this method, have produced pulps of good quality.

T. T. PORTS.

**Colour specification in the pulp and paper industry.** W. B. VAN ARSDEL (J. Opt. Soc. Amer., 1931, 21, 347—357).—A discussion of the conditions which must be satisfied by any method of colorimetry suggested for use in the pulp and paper industry.

C. W. GIBBY.

**Cellulose bleaching. Hydrolysis number and alkali consumption of cellulose.**—See VI. **Roasting of pyrites.**—See X. **Examinations with ultra-violet light.**—See XI. **Paper-sizing materials.**—See XIII. **Photochemistry on paper.**—See XXI.

See also A., July, 793, **Solvent power of alcohols for cellulose nitrate.** 828, **Viscosity of cellulose acetate solutions.** 849, **Cellulose furoate.** 886, **Cellulose films of fossil plants.**

#### PATENTS.

**Preparation of artificial silk filaments.** LUSTRAFIL, LTD., and S. W. BARKER (B.P. 349,658, 6.3. and 31.5.30).—Viscose silk of subdued lustre is obtained by adding to the viscose solution prior to spinning a pre-formed aq. emulsion of a chlorinated aliphatic hydrocarbon. *E.g.*, 3 lb. of  $\text{CCl}_4$  emulsified in 2 gals. of  $\text{H}_2\text{O}$  with the aid of gum arabic or starch is a suitable quantity per 100 gals. of viscose solution.

D. J. NORMAN.

**Production of artificial filaments.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 349,999, 26.2.30).—Continuously with their production, filaments of cellulose acetate while in a sticky condition, brought about by incomplete evaporation of solvent, by treatment with a softener, or by heat, are stretched and caused to merge together into a single filament, which is afterwards treated with a lubricant to remove stickiness; other threads such as wool, metal, etc. may also be incorporated in the composite thread.

F. R. ENNOS.

**Dry-spinning of artificial fibres.** I. G. FARBENIND. A.-G. (B.P. 349,793, 12.6.30. Ger., 13.6.29).—The filaments are extruded in the form of a ring and the evaporative medium is introduced or withdrawn at the centre of the ring close to the spinning nozzle, so that a current of evaporative medium flows radially across the filaments. Suitable devices are described.

D. J. NORMAN.

**Washing and after-treating [centrifugally produced] artificial silk spinning cakes.** I. G. FARBENIND. A.-G. (B.P. 349,681, 14.3.30. Ger., 16.3.29).—A number of spinning cakes are mounted on a long horizontal perforated tube (optionally rotatable) and the washing or treating liquor is passed through the cake from the inside. In this way entanglement of the fibres during washing is avoided.

D. J. NORMAN.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 350,137, 14.4.30. Switz., 13.4.29).—Cotton is immunised to direct dyes with retention of its structure by treatment first with a conc. solution of a salt having a swelling action ( $\text{KCNS}$ ,  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ) and then, after drying if desired, with hot  $\text{Ac}_2\text{O}$  in absence of a diluent.

F. R. ENNOS.

**Manufacture of cellulose acetate.** KODAK, LTD., Assecs. of C. J. MALM (B.P. 349,959 and 350,310, [A] 1.2.30, [B] 25.7.30. U.S., [A] 1.2.29, [B] 10.8.29).—(A) After pretreatment of cellulosic material with  $\text{AcOH}$ , the mixture is distilled with a liquid ( $\text{C}_2\text{H}_4\text{Cl}_2$ ) forming an azeotropic mixture with  $\text{H}_2\text{O}$ , and esterification is completed by addition of  $\text{Ac}_2\text{O}$  and a catalyst ( $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$ ). (B) After hydrolysis of the ester to the desired extent, a solution of  $\text{HCl}$  in  $\text{AcOH}$  is added to the hydrolysing bath, whereby a cellulose acetate free from haze is obtained.

F. R. ENNOS.

**Impregnated and/or smoothed articles and mixtures for their production.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,638, 3.3.30).—Wood, leather, paper, textiles, etc. are impregnated with solutions or emulsions of the viscous or solid polymerisation products of alkylene oxides (cf. B.P. 346,550; B., 1931, 666) either alone or in admixture with waxes, natural or artificial resins, cellulose derivatives, colouring agents, or the like.

D. J. NORMAN.

**Utilisation of celluloid waste.** W. SATOR (B.P. 350,084, 21.3.40. Ger., 2.4.29).— $\text{C}_2\text{HCl}_3$  is added to a filtered solution of the waste in  $\text{COMe}_2$ , to which the requisite amount of  $\text{HCl}$  has been added to dissolve the  $\text{ZnO}$  (in the case of white pigmented waste). The  $\text{COMe}_2$  is distilled off and the nitrocellulose ppt. is separated, washed with  $\text{C}_2\text{HCl}_3$ , and carefully dried. From the  $\text{C}_2\text{HCl}_3$ -camphor solution, the camphor is recovered on distillation in steam.

H. ROYAL-DAWSON.

**Insulating material.**—See XI. **Rubber substitute.**—See XIV. [Wrappers for] eggs.—See XIX.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Viscosity control in cellulose bleaching.** H. OKADA and E. HAYAKAWA (Cellulose chem., 1931, 12, 153—162).—The cuprammonium method of determining the viscosity of a cellulose can be replaced by measuring the viscosity in a 1%  $\text{COMe}_2$  solution of its nitrate prepared by nitration below  $0^\circ$  of cellulose (1 pt.) with mixed acid (75 pts.) containing 63% of  $\text{H}_2\text{SO}_4$ , 27.5% of  $\text{HNO}_3$ , and 9.5% of  $\text{H}_2\text{O}$ , washing first with ice water and then with luke-warm water, and drying over 40—50%  $\text{H}_2\text{SO}_4$ . The Cu numbers,  $\alpha$ -cellulose contents, and viscosities are given of celluloses unbleached and bleached with  $\text{Cl}_2$  under various conditions.

A. RENFREW.

**Hydrolysis number [of cellulose].** O. FAUST (Cellulosechem., 1931, 12, 125—126).—The effect of mercerisation on the Schwalbe "hydrolysis number" of cotton linters is discussed. The starting material has  $\alpha$ -cellulose 99.72—99.86%, Cu number 0.51, hydrolysis number 3.39. Treatment with mercerising solutions and "ripening" for varying periods shows that the hydrolysis number rises to 7.43 (max.) after 8 days' ripening, falling to 6.88 after 17 days, the Cu number falling to 0.09, due to dissolution of degradation products.

T. T. PORTS.

**Determination of alkali consumption of cellulose.** W. SCHRAMEK, C. SCHUBERT, and H. VELTEN (Cellulosechem., 1931, 12, 126—131).—An analysis of the methods employed for determining the alkali adsorbed

from mercerising solutions by cellulose, with particular reference to the work of Schwarzkopf (B., 1931, 437).

T. T. POTTS.

**Deterioration of fabrics exposed on a roof after treatment with fishing-net preservatives.** W. R. G. ATKINS (J. Marine Biol. Assoc., 1931, 17, 473—477).—Cotton and linen fabrics exposed to the air at Plymouth lose  $\frac{1}{3}$ — $\frac{2}{3}$  of their strength in two years. Cu oleate, mixed Cu soaps, and mixtures of the latter with tar or resin have very little effect in preventing deterioration.

C. W. GIBBY.

**Spectrophotometric measurements in the dye-stuffs industry.**—See IV.

See also A., July, 798, **Effect of gelatin and salts on Congo-red.** 802, **Kinetics of chlorine bleaching.**

#### PATENTS.

**Degumming of silk [before dyeing]. Treatment of rayon.** L. MELLERSH-JACKSON. From TWITCHELL PROCESS Co. (B.P. 349,961—2, 3.2.30).—(A) Sericin is rendered sol. with a 0.01—0.03% NaOH solution and emulsified by addition of mineral oil (mahogany) sulphonates. (B) Prior to weaving and knitting, rayon fibres are given a protective coating of mineral oil and true mahogany sulphonates, which is removed by immersion in  $H_2O$  before dyeing and finishing.

F. R. ENNOS.

**Dyeing [of knitted tubular fabrics].** J. H. HEAP (B.P. 347,235, 26.10.29 and 22.3.30).—Fabric is passed through a dye liquor while being simultaneously exposed to submerged jets of the liquor. Tubular fabric is dyed while being drawn along a submerged perforated pipe through which dye liquor is forced. A. J. HALL.

**Decorative treatment of [cellulose acetate silk] fabrics.** CALICO PRINTERS' ASSOC., LTD., and J. R. WHINFIELD (B.P. 348,715, 21.2.30).—White resist or discharge effects are obtained by overall-printing cellulose acetate materials, before or after the printing with  $Zn(OAc)_2$ ,  $ZnO$ , or other metal compound, with dyes which have an affinity for cellulose acetate and are capable of forming insol. complexes with such metal compounds, then steaming and heating with a dil. acid to remove the insol. complex. Coloured effects are obtained by adding to the  $Zn(OAc)_2$  printing paste dyes which have an affinity for cellulose acetate but form no insol. complex with metal compounds.

A. J. HALL.

**Impermeable cloth.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 350,358, 3.10.30. U.S., 3.10.29).—The cloth, suitable as a substitute for 'goldbeaters' skin etc., is prepared by coating a fabric with a mixture of viscose, rubber latex, and glycerin, drying the coating, and regenerating the cellulose from the viscose by exposure to an acid gas (e.g.,  $SO_2$ ) at room temp. It is advantageous to coat the fabric with a rubber composition, and then to size it with an adhesive such as gelatin before applying the viscose-latex mixture.

D. F. TWISS.

**Treatment [sizing and finishing] of textile materials and the like.** H. BOLLMANN and B. REWALD (B.P. 348,783, 20.3.30. Ger., 20.4.29).—Yarns and fabrics are impregnated with fatty acids and oils

which have been emulsified with vegetable phosphatides extracted from soya beans

A. J. HALL.

**Treatment of wool [to give non-shrink finish].** L. B. SMITH and C. E. RUBY (U.S.P. 1,781,415, 11.11.30. Appl., 3.6.24).—More accurate control of the chlorination non-shrink finish for wool materials is obtained by using solutions of NaOCl in which the concentration of OH ion is  $10^{-3}$ — $10^{-5}$  molal equiv. per litre. A satisfactory solution in which the  $[OH^-]$  is  $10^{-3}$  is prepared by adding 0.2 g. of NaOH to 1 litre of a 0.0185% solution of NaOCl.

A. J. HALL.

**Apparatus for wet treatment of textiles.** F. H. ROGERS. From SMITH, DRUM & Co. (B.P. 350,556, 14.3.30).

**Drying and carbonising machines [for textiles].** H. KRANTZ (B.P. 351, 212, 2.6.30).

**Rubberised fibres. Rubberised sheet.**—See XIV.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**New contact [sulphuric] acid plant.** D. S. DINSMOOR (Chem. Met. Eng., 1931, 38, 330—333).—A plant recently erected for use with a V catalyst occupies a floor space of 112 sq. ft./ton of S/day as against 1000 sq. ft. for an older contact plant. S is melted by steam in brick-lined pits and pumped by plunger pumps, the barrels of which are immersed in the liquid. A stationary S burner is used consisting of a vertical steel cylinder lined with fire-brick packed with chequerwork and having primary and secondary air inlets. The gases are air-cooled in a cast Fe shell surrounded by a steel shell and after dust filtration pass to two cast Fe converters in series. The first effects 80% conversion and the second a further 16.5—17.0%. The granular catalyst mass is packed on Fe screen shelves having baffles between them. Absorption takes place in a steel tower lined with acid bricks and packed with rings. The total back-pressure is 3 in. Hg, and one operator can handle the plant.

C. IRWIN.

**Preparation of potassium carbonate by the magnesia process.** G. I. TSCHUFAROV and V. S. KNUTAREV (J. Chem. Ind. Russ., 1931, 8, 232—238).—Engel's process, depending on the conversion of  $MgCO_3 \cdot 3H_2O$  and KCl into  $MgCO_3 \cdot KHCO_3 \cdot 4H_2O$ , is studied. The optimal conditions for prep. of  $MgCO_3 \cdot 3H_2O$  from MgO and  $CO_2$  are: 1000 c.c.  $H_2O$  per 40 g. MgO, the presence of 10% KCl in the reaction mixture, effective stirring, and a partial pressure of 0.5—1 atm.  $CO_2$ . Optimal yields of double salt are obtained under the following conditions: 17—24°, const. stirring, and use of 22.5 g. of KCl per 18.36 g. of MgO. A max. of 62% of the KCl present is converted into double salt, keeping the concentration of MgO const. and varying that of KCl (optimal 14%), whilst a max. of 75% of the Mg present is combined using 33% KCl and 12% MgO. The double salt dissolves to the extent of 1 g. per 100 c.c. of wash-water. Washing removes chlorides but not Mg from the double salt.  $K_2CO_3$  may be prepared from the double salt by heating in an autoclave at 140°, or in open vessels at 150—180°, or by the action of  $Mg(OH)_2$ . The first two methods yield conc. solutions (20%), but

the residue of inactive  $\text{MgCO}_3$  has to be calcined for the regeneration of  $\text{MgO}$ , whilst using the third method  $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$  remains, but the concentration of  $\text{K}_2\text{CO}_3$  obtained does not exceed 7%. R. TRUSZKOWSKI.

**Preparation of potassium nitrate by action of nitric acid or oxides of nitrogen on chloride.** I. A. MIRKIN (J. Chem. Ind. Russ., 1931, 8, 351—359).—The purity of the product obtained by the action of  $\text{HNO}_3$  on  $\text{KCl}$  increases with the relative concentration of  $\text{HNO}_3$ . The higher the temp. and the more dil. the acid used, the smaller is the ratio  $\text{HNO}_3 : \text{KCl}$  necessary to obtain pure  $\text{KNO}_3$ . The quantity and composition of the gases evolved during the reaction depend on the reaction temp. and the concentration of acid taken; using 65% acid at  $60^\circ$ , about 10% of the N added is lost as  $\text{NOCl}$ , whilst using 30% acid at  $60^\circ$  very little gas at all is evolved. The yield of  $\text{KNO}_3$  amounts to 45% on the acid used, and to 60—75% on the  $\text{KCl}$  taken, according to the concentration of  $\text{HNO}_3$ . If oxides of N are passed through  $\text{KCl}$ , the yield of nitrate increases with the moisture content of the  $\text{KCl}$ ; where this is small,  $\text{NOCl}$  and  $\text{Cl}_2$  are evolved, whilst with excess  $\text{H}_2\text{O}$  neither  $\text{Cl}_2$  nor  $\text{NOCl}$  is evolved. The max. yields by this method are 60%. R. TRUSZKOWSKI.

**Neutralisation of [gasworks'] ammonia sulphate by gaseous ammonia.** W. E. BUCKLEY (Gas World, 1931, 95, Coking Sect., 83—85).—The  $(\text{NH}_4)_2\text{SO}_4$  is fed into a neutraliser supplied with  $\text{NH}_3$  gas, and dried by passing into a Phillipson machine fitted with an Archimedean screw and a steam-jacket. The crystals are more uniform in composition and structure than those ordinarily obtained, and if steel is used min. corrosion of machinery takes place.

A. H. EDWARDS.

**Bromometric determination of nitrogen in ammonium salts.** B. I. LEVI (J. Chem. Ind. Russ., 1931, 8, 393—396).— $\text{NH}_3$  is oxidised by  $\text{Br}$  in the presence of  $N\text{-NaHCO}_3$ , and excess  $\text{Br}$  is determined iodometrically by addition of  $\text{KI}$ . Where  $\text{NaOH}$  is used in place of  $\text{NaHCO}_3$  a definite end-point is not obtained, owing to formation of  $\text{NaNO}_2$ . The  $\text{pH}$  of the solution should not exceed 8. R. TRUSZKOWSKI.

**Bromometric determination of nitrogen in ammonium salts.** M. L. TSCHPELEVETSKI, S. I. POZDNIakov, and R. D. FAİN (J. Chem. Ind. Russ., 1931, 8, 396—401).—At below  $\text{pH}$  7 the oxidation of  $\text{NH}_4$  salts by  $\text{Br}$  is incomplete, between  $\text{pH}$  7.5 and 9.5 it proceeds quantitatively, between  $\text{pH}$  10 and 12 nitrite is formed, whilst at over  $\text{pH}$  12 both  $\text{NH}_3$  and nitrite are found in the reaction mixture. Levi's method, using phosphate buffer at  $\text{pH}$  8.5 in place of  $\text{NaHCO}_3$ , gives accurate results in the analysis of  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{HCO}_3$ , and of Kjeldahl- $\text{NH}_3$  originating from the combustion of foodstuffs.

R. TRUSZKOWSKI.

**Gravimetric determination of acetylene in calcium carbide.** A. A. VASSILIEV (Z. anal. Chem., 1931, 84, 217—220).—An improved form of Bamberger's method (B., 1898, 382) is described. R. CUTBILL.

**Stability of magnesium peroxide.** A. WOHLK (Dansk Tidsskr. Farm., 1931, 5, 106—108).— $\text{MgO}_2$  slowly loses  $\text{O}_2$  on preservation, a number of samples

showing a loss of from 3—4%  $\text{MgO}_2$  in 6 months. The decomp. is increased by exposure to air.

H. F. HARWOOD.

**Use of hot flue gases in manufacture of copper sulphate.** V. ZEMLANITZIN and P. DOBROVOLSKI (J. Chem. Ind. Russ., 1931, 8, 277—279).—Flue gases from Humboldt furnaces may be substituted for steam for passing through vats in which  $\text{Cu}$  is undergoing dissolution in  $\text{H}_2\text{SO}_4$ .

R. TRUSZKOWSKI.

**Manufacture of basic lead carbonate.** J. F. SACHER (Z. angew. Chem., 1931, 44, 549—550).— $\text{PbO}$  is ground finely with  $\text{H}_2\text{O}$  in a pressure vessel and a mixture of  $\text{NH}_3$  and  $\text{CO}_2$  is passed into the pulp. The  $\text{PbO}$  is thus quantitatively converted into  $2\text{PbCO}_3 \cdot \text{Pb(OH)}_2$ . The excess  $\text{NH}_3$  and  $\text{CO}_2$  are recovered by heating the white-lead paste above  $60^\circ$  and are used again in the process.

A. R. POWELL.

**Utilisation of the gases obtained in the process of volatilising phosphorus and the production of soluble phosphates.** A. P. DUNAIEV (Udobr. Urozhai, 1930, 2, 397—409).—The oxidation of  $\text{P}$  with  $\text{H}_2\text{O}$  over  $\text{CaO}$  at  $550\text{--}950^\circ$  is completed to  $\text{H}_3\text{PO}_4$  at a fairly high gas velocity; very little  $\text{PH}_3$  is present, and the sum  $\text{CO} + \text{H}_2$  is not decreased. In the oxidation of the gases no air in excess of that needed for oxidation of  $\text{P}$  was necessary. When  $\text{NaCl}$  was used, the temp. had to be kept at  $400\text{--}500^\circ$ ; oxidation of  $\text{P}$  took place with  $\text{O}_2$ , but not with  $\text{H}_2\text{O}$ .

CHEMICAL ABSTRACTS.

**Superphosphates enriched with ammonia.** S. I. VOLFKOVICH, L. E. BERLIN, I. L. HOFMAN, and A. A. IONAS (Udobr. Urozhai, 1930, 2, 495—504).—The  $\text{H}_2\text{O}$ -sol. and citrate-sol.  $\text{P}_2\text{O}_5$  in the products obtained by mixing  $\text{H}_2\text{SO}_4$  with  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $(\text{NH}_4)_2\text{HPO}_4$ , and  $(\text{NH}_4)_3\text{PO}_4$  under various conditions were determined.

CHEMICAL ABSTRACTS.

**Constitution and citrate-solubility of tricalcium phosphate and phosphate rock.** K. D. JACOB (Phosphorus Digest, 1931, Apr., 7—9).—When 0.5-g. samples are used, 65—75% of the total  $\text{P}_2\text{O}_5$  in precipitated  $\text{Ca}_3(\text{PO}_4)_2$  and steamed bone meal is sol. in neutral  $\text{NH}_4$  citrate solution; for 2.0 g., 25—30% is sol. The citrate-solubility of ground phosphate rock is only about 12% of that of  $\text{Ca}_3(\text{PO}_4)_2$  or bone, principally owing to the presence of  $\text{F}$  as  $\text{Ca fluophosphate}$ .

CHEMICAL ABSTRACTS.

**Determination of small quantities of oxygen in gases.** J. WIERCIŃSKI (Przemysł Chem., 1931, 15, 188—191).— $\text{O}_2$  is absorbed by  $\text{Fe(OH)}_2$  in aq.  $\text{NH}_3$ , the mixed hydroxides are dissolved in conc.  $\text{HCl}$ , and residual  $\text{Fe}^{++}$  is determined by potentiometric titration with  $\text{KMnO}_4$ . 0.0002% of  $\text{O}_2$  in a gaseous mixture can thus be determined with an accuracy of 0.001%.

R. TRUSZKOWSKI.

**Ammoniacal liquor.**—See II. Catalysts for  $\text{MeOH}$  synthesis.—See III. Titanium-white.—See XIII. Salt for curing hides etc.—See XV. Fungicide.—See XVI. Solubility of  $\text{CaO}$  in sucrose solutions.—See XVII.

See also A., July, 793, Solubilities of gases in liquids. 796, Screening smokes. 797, Sols and gels of  $\text{Th(OH)}_4$  and  $\text{Fe(OH)}_3$ . Colloidal  $\text{Fe(OH)}_3$ .

803, Absorption of  $O_2$  in conc. system  $HNO_3$ - $NO_2$ - $H_2O$ . Kinetics of decomp. of  $NH_3$  on Cu. Prep. of Cu-Cr $_2O_3$  catalysts for hydrogenation. 805, (CN) $_2$  synthesis. 806,  $Na_2O$ . 807, Prep. of pure Ca(Mg)CN $_2$ . 809, Removal of traces of  $O_2$  from  $N_2$ . Formation of N fluorides. 810, Prep. of HI. 812, Electrometric titration of  $H_3PO_4$ . Determination of  $H_3BO_3$ . 813, Determination of  $Hg_2Cl_2$ . Prep. of Er oxide. 855, Trichlorosilicane and SiCl $_2$ .

# PATENTS.

**Elimination of arsenic from acids, particularly sulphuric acid.** METALLGES. A.-G. (B.P. 349,472, 26.2.30. Ger., 13.11.29).—Precipitation of the As as sulphide by means of an oxyacid of S or a salt thereof, e.g.,  $Na_2S_2O_3$ , is effective even with acids of  $d$  1.526 or over, if the temp. is kept below 60–70°.

W. J. WRIGHT.

**Separation of arsenic sulphide precipitates from acids.** A. L. MOND. From METALLGES. A.-G. (B.P. 349,715, 10.4.30).—Separation is facilitated by stirring with a finely-divided substance, such as sand, or an org. liquid, e.g., benzol, in conjunction with a frothing agent, e.g., turpentine oil, or by introducing  $N_2$ ,  $H_2$ ,  $O_2$ , or air in a dispersed form.

W. J. WRIGHT.

**Purification of phosphoric acid and the like.** A. H. FISKE, Assr. to RUMFORD CHEM. WORKS (U.S.P. 1,787,192, 30.12.30. Appl., 11.8.28).— $H_2S$  or a sol. sulphide is added to dil.  $H_3PO_4$  and, after removal of the ppt., more  $H_2S$  etc. is added prior to and during concentration of the acid in apparatus with a corrodible lining, means being provided for sedimentation.

L. A. COLES.

**Apparatus for the manufacture of ammonia.** SOC. ANON. INDUSTRIA AMMONIACA, and F. LOPEZ (B.P. 350,643, 9.4.30).—The upper and lower cylinders containing the reaction chamber and the heat exchanger are separated by a perforated disc which serves to support these operative parts and protect them from deformation. The chamber containing the catalyst is provided with a flange, resting in a seat on the surrounding tubular chamber, and an airtight joint, so that it can be readily lifted and replaced.

W. J. WRIGHT.

**Purification of gases for synthesis of ammonia.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, Assees. of SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 350,120, 4.4.30. Fr., 9.7.29).—Sufficient  $H_2O$  vapour is admitted with the fresh gas to dissolve on condensation the (NH $_4$ ) $_2CO_3$  formed by combination of the traces of  $CO_2$  in the fresh gas with the  $NH_3$  in the returned gas, and the solution is withdrawn from the apparatus.

L. A. COLES.

**Working up of sylvinitic crude salts to a mixture of potassium and sodium nitrates or this mixture and soda.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 349,823, 11.7.30. Ger., 23.8.29).—KCl is mixed with the sylvinite to make the K : Na ratio equal to that in glaserite, and the mixture is stirred with  $NH_4Cl$  solution containing (NH $_4$ ) $_2SO_4$ .  $NH_3$  being passed in. The glaserite ppt. is treated with  $CaCO_3$  and  $HNO_3$ , the

gypsum separated, and the solution evaporated to dryness. The mother-liquor from the glaserite precipitation is treated with  $CO_2$  and the gypsum added, the (NH $_4$ ) $_2SO_4$  produced being added to the original  $NH_4Cl$  solution.

W. J. WRIGHT.

**Production of sodium nitrate and ammonium chloride.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 350,343, 12.9.30. Ger., 3.10.29).—The mother-liquor from the process, which contains (NH $_4$ ) $_2SO_4$ ,  $NH_4Cl$ , and NaCl in solution together with  $CaCO_3$  in suspension, is treated with a wt. of NaCl equiv. to the (NH $_4$ ) $_2SO_4$  and with  $NH_3$ ; precipitated  $Na_2SO_4$  and the  $CaCO_3$  are removed together and treated with  $HNO_3$ ; precipitated  $CaSO_4$  is removed, and the solution is evaporated or cooled to recover  $NaNO_3$ . The  $Na_2SO_4$  mother-liquor is cooled, preferably after addition of NaCl, to crystallise and remove  $NH_4Cl$ , and the residual liquor is saturated with  $CO_2$  and then stirred with  $CaSO_4$  to yield a liquor for re-use in the process.

L. A. COLES.

**Manufacture of easily-soluble stable hypochlorites.** P. R. HERSHMAN, Assr. to C. O. and C. H. SETHNESS and P. RUDNICK (U.S.P. 1,784,286, 9.12.30. Appl., 15.4.27).—Ca(OCl) $_2$  or Mg(OCl) $_2$  is mixed with 1 equiv. of NaOAc in the presence of  $H_2O$ , preferably with the addition of a solid acid or, e.g., 15% of sol. starch, and the product is dried.

L. A. COLES.

**Production of sulphates.** METALLGES. A.-G., C. (BARON) VON GIRSEWALD, and E. STAHL (B.P. 350,050, 13.3.30).— $SO_2$  and an excess of air or  $O_2$  are introduced as extremely fine bubbles, e.g., by passage through porous material or by comminution by rapidly rotating vanes, into  $H_2O$  to which  $NH_3$ , CuO, ZnO, etc. is added simultaneously at a rate such as to maintain  $p_H$  4–5; the process may be effected at room or at a raised temp., e.g., 80°, and catalysts (CuSO $_4$ , FeSO $_4$ , Cu $_2Cl_2$ ) may be present. Alternatively, air alone is introduced as described into sulphite solutions.

L. A. COLES.

**Treatment [purification] of barytes.** C. P. DE LORE and B. B. McHAN (U.S.P. 1,783,778, 2.12.30. Appl., 7.3.27).—Finely-divided barytes, after treatment with  $H_2SO_4$ , is treated with  $SO_2$  solution to reduce Fe $^{+++}$  to Fe $^{++}$ , and is then washed and dried.

L. A. COLES.

**Production [precipitation] of sodium stannate.** W. T. LITTLE (U.S.P. 1,787,078, 30.12.30. Appl., 10.9.29).— $Na_2SnO_3$  is salted out of solution by the addition of conc. NaOH solution (>50 g./100 c.c.) and the mother-liquor is conc. for re-use.

L. A. COLES.

**Decomposition of zirconium ores.** DEUTS. GAS-GLÜHLICHT-AUER-GES.M.B.H. (B.P. 350,728, 18.6.30. Ger., 29.6.29).—Ores containing ZrO $_2$  and SiO $_2$  are treated with alkali oxide (or carbonate) in the proportion of 1 mol. of the latter per mol. of ZrO $_2$ .

W. J. WRIGHT.

**[Production of] granular [mono]calcium phosphate.** H. ADLER and G. A. McDONALD, Assrs. to VICTOR CHEM. WORKS (U.S.P. 1,785,473, 16.12.30. Appl., 7.1.29).—Granular CaH $_4$ (PO $_4$ ) $_2$  containing <0.5% FePO $_4$ , suitable for use in baking powders, is obtained by treating  $H_3PO_4$  prepared by volatilisation processes with CaO,  $CaCO_3$ , etc. containing sufficient Mg compounds to yield

a product containing 0.6—2% (preferably 1—1.5%) Mg compounds, calc. as MgO. L. A. COLES.

**Production of dicalcium phosphate from sulphurous solutions.** E. THILO, Assr. to A. HEICKE (U.S.P. 1,786,097, 23.12.30. Appl., 24.9.29. Ger., 11.2.28).—An aq. suspension of crude  $\text{Ca}_3(\text{PO}_4)_2$  is saturated with  $\text{SO}_2$ , filtered after about 24 hr., treated with HCl, and blown with air at room temp. to expel the  $\text{SO}_2$ ; the ppt. of  $\text{CaHPO}_4$  is removed. L. A. COLES.

**Manufacture of solid carbon dioxide products.** DRY-ICE CORP. OF AMERICA, Assees. of C. L. JONES and J. D. SMALL (B.P. 350,532, 11.3.30. U.S., 28.3.29).—Liquid  $\text{CO}_2$  is evaporated at or near the triple point and cryst. under such conditions as to give crystals not exceeding 0.01 in. in length, these being compressed at a temp. and pressure to cause partial recrystallisation. The resultant blocks are of uniform density and do not crumble. W. J. WRIGHT.

**Treatment of recovered sulphur. Removal of arsenic and chlorine from sulphur.** S. I. LEVY (B.P. 350,573—4, 15.3.30).—(A) Purification from As is effected by treating the molten S with an alkali compound, such as  $\text{Na}_2\text{S}$ , either in the solid state or as an aq. solution, the S being separated and the As compound removed from the residue by treatment with  $\text{H}_2\text{O}$ . (B)  $\text{Cl}_2$  or  $\text{S}_2\text{Cl}_2$  is added to the vaporised or molten S, the product being fractionated to separate the greater part of the volatile chlorides and then subjected to reduced pressure or passed down a scrubbing tower in countercurrent to air or an inert gas, the exit gases being decomposed by scrubbing with  $\text{H}_2\text{O}$ . Any remaining traces of impurities are removed by steam treatment. W. J. WRIGHT.

**Production of sulphur [from sulphur dioxide].** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 350,124, 5.4.30).—Gases containing, e.g., 5—15%  $\text{SO}_2$  are passed into the lower end of a shaft furnace containing coke at 1000—1300°, and the escaping gases, before treatment for recovery of S, pass around the furnace through annular flues supplied with secondary air containing insufficient  $\text{O}_2$  to combine with all the CO. L. A. COLES.

**Preparation of sulphur dioxide.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. W. NORDLANDER (B.P. 350,739, 23.6.30. U.S., 22.6.29).— $\text{O}_2$ , preferably preheated, is conducted in the form of fine bubbles into a S burner containing liquid S, supplied from a jacketed container. A vessel, filled with pebbles, above the burner serves to condense S carried up with the  $\text{SO}_2$ . The burner is provided with a lateral device for determining the level of the liquid S. W. J. WRIGHT.

**Production of hydrogen or gas mixtures containing the same.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 349,060, 18.11.29).—Hydrocarbons, e.g., gases containing  $\text{CH}_4$ , are burned with sufficient  $\text{O}_2$  or air to oxidise them completely, and the hot gases formed, to which a further quantity of steam may be added if desired, are brought into contact with coke at a temp. at which the steam is converted completely into  $\text{H}_2$  and CO. The gases may then be passed together with  $\text{H}_2\text{O}$  vapour over a catalyst, in known manner, to remove the CO. A. B. MANNING.

**Complete separation of liquid air into oxygen and nitrogen.** A. MESSER (U.S.P. 1,785,491, 16.12.30. Appl., 18.2.27. Ger., 18.2.26).—In a two-column apparatus in which the upper column is operated at a lower pressure than the lower, the liquid rich in  $\text{O}_2$  collecting in the lower column enters the upper at an intermediate point through a reduction valve, the liquid  $\text{O}_2$  collecting in the upper column passes through and evaporates in a coil in the top section of the lower column, and liquid  $\text{N}_2$  collecting in the upper column is conveyed to a reduction valve at the top of the column. L. A. COLES.

**Purification of hydrogen peroxide solutions.** KALI-CHEMIE A.-G. (B.P. 349,771, 26.5.30. Ger., 15.6.29).—The solutions are purified by electro-osmosis and then conc. by evaporation or distillation. W. J. WRIGHT.

**Apparatus for the production of ozone.** V. GAMBAROTTA (B.P. 350,774, 16.7.30. Ger., 18.7.29).—The electrodes and the dielectric between them are mounted on an insulating base, which fits into a socket, contacts being provided on the latter and the outer surface of the base. W. J. WRIGHT.

**Electrolytic manufacture of chlorine.** F. S. LOW (B.P. 350,479, 7.3.30).—See U.S.P. 1,746,542; B., 1930, 1065.

**Cellulose derivatives.**—See V. Fertilisers.—See XVI.

## VIII.—GLASS; CERAMICS.

**Conservation of heat in the annealing of glass.** T. C. MOORSHEAD and E. A. COAD-PRYOR (Fuel Econ. Rev., 1931, 10, 35—38).—The improvement in annealing furnaces has been from updraught kilns using 90—100 therms per ton of glass to tunnel furnaces with endless belts of both the open and muffle type using 20 therms per ton; to efficiently insulated tunnel furnaces consuming 5.5 therms per ton, rendered possible by the speeding up of the process of glass manufacture; and to furnaces requiring no auxiliary heating. The last-named are tunnel furnaces heavily insulated at the inlet end and fitted with a conveyor of endless wire mattress. The return part of the conveyor is preheated by the cooling glassware. When operating with light loads heat is supplied by electric heaters. D. K. MOORE.

**Direct determination of soda in soda-lime glasses by precipitation as uranyl zinc sodium acetate.** F. W. GLAZE (J. Amer. Ceram. Soc., 1931, 14, 450—453).—Full details are given of the precipitation of  $\text{Na}_2\text{O}$  in the presence of other basic oxides, as  $(\text{UO}_2)_3\text{ZnNa}(\text{OAc})_9 \cdot 6\text{H}_2\text{O}$ , according to Barber and Kolthoff's method (A., 1928, 859). J. A. SUGDEN.

**Selenium dioxide as a constituent of glasses.** L. NAVIAS and J. GALLUP (J. Amer. Ceram. Soc., 1931, 14, 441—449).—Melts with  $\text{SeO}_2$ ,  $\text{H}_2\text{SeO}_3$ , or  $\text{Na}_2\text{SeO}_3$  as a major constituent of the batch lose a large proportion of the  $\text{SeO}_2$  by volatilisation. Approx. 25%  $\text{SeO}_2$  can be retained in low-melting  $\text{Na}_2\text{O}$ — $\text{CaO}$ — $\text{B}_2\text{O}_3$  and  $\text{Na}_2\text{O}$ — $\text{Pb}_2\text{O}_3$ — $\text{B}_2\text{O}_3$  glasses, but these glasses are readily affected by  $\text{H}_2\text{O}$ . Addition of  $\text{SiO}_2$  produces a stable glass, but causes a lowering of the  $\text{SeO}_2$  content to approx. 10%, due to the higher melting temp. required. J. A. SUGDEN.



**Granular and ground felspar with a uniformly low iron content.** J. H. WEIS (J. Amer. Ceram. Soc., 1931, 14, 413—418).—The crushed mineral is passed through a Rowand-Wetherill magnetic separator. The output of this machine is low because the Fe impurities must be picked up against gravity. In an improved induction type of machine more efficient action of the magnetic flux is attained by passing the material over the actual surface of a series of magnetic rotors, the polarity of which is automatically changed during rotation, thus facilitating the release of the separated magnetic particles. The plant delivers a material extremely low in Fe and equal or superior to the hand-picked product. J. A. SUGDEN.

**Method for eliminating water-marks from table-ware.** G. A. BOLE (J. Amer. Ceram. Soc., 1931, 14, 454—456).—Drops of  $H_2O$  which collect on the glazed surface after decoration leave a residue which causes a dull spot on the surface after firing. More efficient draining of the ware is obtained if it is dipped in a saponin solution (1 pt. in 4000) after the decorating process. The tendency of the saponin solution to foam is overcome by the addition of small amounts of a solution (1 pt. in 3000) of Turkey-red oil.

J. A. SUGDEN.

**Development of porcelain firing in the tunnel oven.** W. HENTSCHEL (Gas- u. Wasserfach, 1931, 74, 549—553).—The evolution of the tunnel oven, the substitution of gaseous for solid fuels in the firing process, and the comparative costs of the process with tunnel and chamber ovens are discussed. The use of gaseous fuel is more economical and gives more uniform temperatures. Utilisation of producer gas in a tunnel oven necessitated frequent cleaning of the gas inlets from dust and tar; this disadvantage was overcome and greater control of the oven condition was obtained by substitution of the producer gas by gas from the mains (grid system).

H. E. BLAYDEN.

**Effect of flint on the modulus of elasticity of a soft-fired material.** C. G. HARMAN (J. Amer. Ceram. Soc., 1931, 14, 356—357).—The well-known break in the elasticity-temp. curve of quartz at  $573^\circ$  is not exhibited by a soft-fired clay-quartz body. The elasticity is dependent on the size and quantity and not on the kind of grog. J. A. SUGDEN.

**Effect of furnace atmospheres on the quality of certain types of glazes.** A. P. WATTS (J. Amer. Ceram. Soc., 1931, 14, 460—468).—Laboratory tests on Pb and leadless glazes show that  $H_2O$  vapour,  $SO_2$ , CO, and unburnt hydrocarbons produce discoloured and sometimes scummed and dull glazes and that an oxidising atm. is essential for good results. J. A. SUGDEN.

**Opacity of enamels in terms of colours.** R. E. STARK (J. Amer. Ceram. Soc., 1931, 14, 457—459).—The opacities of both white and coloured enamels have been measured with a Pfund colorimeter.

J. A. SUGDEN.

**Service spalling test for refractories.** S. M. PHELPS, S. M. SWAIN, and R. F. FERGUSON (J. Amer. Ceram. Soc., 1931, 14, 389—402).—The test panel comprising 10 bricks (stretchers) held under a pressure of 10 lb./sq. in. in a vertical metal frame is first pre-

heated to  $1600^\circ$  for 24 hr. While in the preheating furnace the panel is backed by 8 in. of diatomite. After cooling and photographing, the panel is treated in the spalling furnace (gas-fired), where it is alternately heated to  $1000^\circ$  and rapidly cooled by exposure to a blast of air carrying a spray of  $H_2O$ , each period lasting 10 min. After 12 cycles the panel is cooled, dismantled, and the spalling expressed as % loss in wt. The test differentiates between structural and thermal spalling. The data obtained emphasise the influence of insulation in increasing spalling, owing to the greater temp. gradients. J. A. SUGDEN.

**Fusion-test furnace.**—See I. Photochemistry on glass.—See XXI.

See also A., July, 800, Reaction between  $Na_2CO_3$  and  $SiO_2$ . 802, Velocity of hydrolysis of Na silicates.

## PATENTS.

**Conduits for use in the production of glass.** PILKINGTON BROS., LTD., and E. B. LEMARE (B.P. 350,480, 11.3.30).—The conduits are constructed of semi-fused, dense refractory material of the porcelain type. L. A. COLES.

**Prevention of steaming and the formation of raindrops and the like on glass surfaces etc.** COMP. INTERNAT. DES INDUSTRIES CHIM. "INTERCHIMIC," SOC. ANON. (B.P. 350,847, 18.9.30. Belg., 28.9.29).—A coating of cellulose or a derivative, glycerin and/or solution of nicotine in EtOH, and diluents is applied. L. A. COLES.

**Spall-burning kiln.** M. E. SQUIRE (U.S.P. 1,783,632, 2.12.30. Appl., 25.11.27).—The upper and lower halves of a vertical kiln are connected by a passageway inclined at an angle of  $60^\circ$ , below the lower end of which are situated the gas ports and combustion chambers. While traversing the slanting passageway the descending charge of limestone rolls over and becomes thoroughly mixed and the ascending flames also cross over and give uniform distribution of the heat. Poke and observation holes are provided in case of sticking of the charge, and the rate of discharge of CaO is regulated by a discharge door. J. A. SUGDEN.

**Drying and burning of moulded ware.** J. M. PILLATZKE (U.S.P. 1,786,364, 23.12.30. Appl., 18.3.29).—A chamberless continuous kiln is provided with a series of outlets in the roof, which are connected by means of removable pipes to the main waste-gas flue running down the length of the kiln. The ware is set so that longitudinal fire spaces are provided, along which the fires creep. The removable connecting flues are moved forward when the gases entering the first set of flues reach approx.  $100^\circ$ . As each fresh section of "green" ware is set, the forward end is sealed with paper, which is later burned off by the fires. The cooling ware preheats the air to the fires.

J. A. SUGDEN.

**Sintering of clay.** J. E. GREENAWALT (U.S.P. 1,786,714, 30.12.30. Appl., 16.2.29).—The argillaceous mixture is granulated (with or without passing through the intermediate stage of slurry), covered with pulverised (or mixed with crushed) fuel, and spread as a

porous bed upon a porous hearth. The fuel is ignited and combustion propagated throughout the mass by air drawn downward through the hearth. A material suitable for use as a light, porous aggregate for concrete is rapidly and economically produced. J. A. SUGDEN.

**Device for testing [the bonding strength of] clays and the like.** J. F. MACKIN (U.S.P. 1,785,521, 16.12.30. Appl., 15.11.28).—A beam pivoted about its mid point carries on each arm a vertical pivoted thrust member terminating in a thrust head lying a short distance above a companion head on the base plate. A wt. is provided to slide across the graduated beam, so that pressure may be applied to the test material placed on the pressure heads on the base plate. Either single or comparative tests may be carried out.

J. A. SUGDEN.

**Prevention of efflorescence in ceramics.** A. HOUGH and J. G. HARRISON (U.S.P. 1,785,309, 16.12.30. Appl., 15.6.27).—BaSiO<sub>3</sub> (or other silicate of Ba) is substituted for the usual and more expensive BaCO<sub>3</sub>, BaCl<sub>2</sub>, etc.

J. A. SUGDEN.

**Recrystallised refractory composition.** T. S. CURTIS, Assr. to VITREFRAX CORP. (U.S.P. 1,786,482, 30.12.30. Appl., 26.6.28).—A cryst. mullite aggregate is bonded with amorphous mullite and fired to cone 17.

J. A. SUGDEN.

**Aluminous abrasive.** NORTON Co. (B.P. 350,561, 14.3.30. U.S., 8.4.29).—Crude Al<sub>2</sub>O<sub>3</sub> mixed with about 1% of FeS is fused with sufficient C to reduce extraneous oxides, to convert the FeS into Al<sub>2</sub>S<sub>3</sub> and Fe, and to reduce about 2–4% of the Al<sub>2</sub>O<sub>3</sub> to Al<sub>4</sub>C<sub>3</sub>, Al, etc.; the product, after it has been disintegrated by wetting and exposure to the air, is washed, screened, dried, passed through a magnetic separator, calcined, and then fused under superheating conditions and cooled slowly to yield large crystals of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, *d* 3.97.

L. A. COLES.

**Reheating and cooling glass in sheets or slabs.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY ET CIREY (B.P. 350,422, 9.2.30. Fr. 7.2.29).

**Electric furnaces. Crucibles.**—See XI.

## IX.—BUILDING MATERIALS.

**Composition of Portland cement.** A. C. DAVIS (Cement, 1931, 4, 637–641).—A digest of Brit. Standard and other specifications with relation to the SiO<sub>2</sub> and hydraulic moduli, together with analyses of representative cements, is given.

C. A. KING.

**Resistance of concrete to chemical attack.** K. MANDE (I Comm. New Int. Assoc. Testing Mat., 1930, B, 149–156).—A discussion of deterioration by H<sub>2</sub>O containing sulphate.

CHEMICAL ABSTRACTS.

**Compactness of concrete and its resistance to chemical action.** A. POULSEN (I Comm. New Int. Assoc. Testing Mat., 1930, B, 157–161).—A discussion, relating particularly to addition of SiO<sub>2</sub> sol. in alkalis.

CHEMICAL ABSTRACTS.

**Strengths of brick and other structural silicates.** J. H. GRIFFITH (J. Amer. Ceram. Soc., 1931, 14, 325–355).—The stress-strain relationship of the average

group of the system CaO–SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> (+ Mg, Fe, Na, etc.) is treated as being nothing more than a pressure-vol. relationship as defined by the Boyle–Mariotte law. If the strength at the stress limit is expressed in terms of density, porosity, or adsorption (these being mutually convertible), the data on the strengths of structural silicates collected during the last 50 years can be correlated by the formula  $p = cS(A + A_0)^{-1}(1 \pm e)$ , where  $p$  is the stress at rupture,  $c$  a const.,  $S$  dimension stress,  $A$  porosity (H<sub>2</sub>O adsorption),  $A_0$  insensible or colloidal pore space, and  $e$  the probable error for a single datum. The strength of a structural silicate is therefore a function of the density and will be equal to that of (say) granite if it has the adsorption or porosity of granite or equal to that of limestone if it has the porosity of limestone etc.

J. A. SUGDEN.

**Thermal conductivity coefficient of "Eternit."** G. BOZZA and I. SECCHI (Giorn. Chim. Ind. Appl., 1931, 13, 212–215).—The thermal conductivity coeff. of "Eternit" (highly compressed mixture of cement and asbestos) between room temp. and 40° is 0.45 kg.-cal. per m./hr./° C.

O. J. WALKER.

**Healthy and frozen wood.** K. BRASS and W. EHM (Cellulosechem., 1931, 12, 173–178).—Analysis of frozen and unfrozen wood of *Gleditschia monosperma* are given. The effect of freezing is similar to that of certain fungi and bacteria. From the COMe<sub>2</sub>-sol. fractions of frozen wood and unfrozen wood compounds, m.p. 203–204° and about 185°, and 280–282°, respectively, can be isolated.

A. RENFREW.

See also A., July, 802, **Velocity of hydrolysis of Na silicates.** 807, **Ca sulphate hydrate.** 886, **Dry rot in wood.**

## PATENTS.

**Manufacture of Portland cement.** G. S. LA FORGE (U.S.P. 1,784,840, 16.12.30. Appl., 23.7.29).—The raw components of cement are mixed with 10–50% of Portland cement clinker, and the mixture is pulverised prior to calcination.

C. A. KING.

**Production of Portland cement.** E. R. WILNER, Assr. to EDDYSTONE CEMENT CORP. (U.S.P. 1,785,508, 16.12.30. Appl., 28.7.28).—CaO and clay or other silicious earths are heated separately to a much lower temp. than is necessary in the production of Portland cement, and are then mixed and ground together. A jet of H<sub>2</sub>O or steam is then introduced into the mixture under agitation to produce active SiO<sub>2</sub> which will combine with free CaO in the mixture.

C. A. KING.

**Production of ground raw material for Portland cement manufacture.** J. A. MILLER and E. P. NEWHARD (U.S.P. 1,784,462, 9.12.30. Appl., 10.5.27).—The raw batch is ground and graded, e.g., by air separation, the coarser portion being reground and again separated. After a final fine grinding of the residue the various portions are mixed together.

C. A. KING.

**Manufacture of cementitious material.** H. BERRY (B.P. 350,129, 10.4.30).—Natural CaCO<sub>3</sub> or MgCO<sub>3</sub> is calcined in the presence of HCl admitted either directly into the kiln or in the form of chlorides, e.g., AlCl<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, Al salts, or sol. SiO<sub>2</sub> may also be introduced.

C. A. KING.

[Light] mortar, concrete, etc. F. E. MARECHAL (B.P.349,957, 1.1.30. Belg., 2.1.29).—Insol. material, e.g., sand, is pulverised until colloidal and mixed with a binder, e.g., CaO, cement, plaster, or MgO, so as to entrap air bubbles. Chemicals to produce additional gas in the mixture may be added. C. A. KING.

Treatment of brick, concrete, tile, or the like surfaces. H. E. DOUGHTY (B.P. 350,445, 8. and 28.3.30).—Dry cement powder and colouring matter are sprinkled or blown by compressed air on to the wetted surfaces. L. A. COLES.

Forming greyish-black slate granules. H. C. FISHER, Assr. to PHILIP CAREY MANUFG. CO. (U.S.P. 1,782,649, 25.11.30. Appl., 20.6.25).—Greenish slate granules are wetted with a solution of alkali dichromate, metallic sulphates, and other pigments which on heating to incipient fusion impregnate the slate with the oxides of the reacting substances. Fe and Cu are suitable metals. C. A. KING.

Treatment of wood. A. P. ALLEN (U.S.P. 1,785,571, 16.12.30. Appl., 23.5.25).—Wood is immersed in a 20% solution of mono-, di-, or poly-saccharide at 90–150° under pressure. Protecting oils or chemicals and latex may be added to the solution. C. A. KING.

Aq. dispersions.—See I. Bitumen dispersions.—See II. Impregnated articles.—See V. Sintering of clay.—See VIII. Nitrocellulose products.—See XIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Quality and fuel-consumption factors in smelting and remelting processes for production of wrought iron and iron castings. J. E. FLETCHER (Fuel Econ. Rev., 1931, 10, 51–57).—The remelting of scrap Fe increases the S content by 0.03–0.07% and its use, in large proportion, in the production of castings is to be deprecated. To obtain pig Fe of uniform composition, homogeneous structure, and low in total C, necessary for the production of high-class cast and wrought Fe, lean ores containing sufficient Si, Mn, and P, and regular charging rates into slowly-driven small furnaces with cool blast at a pressure barely sufficient to generate CO in the hearth, are required and the furnaces should be tapped about once every 12 hr. Good-quality cast Fe is being produced from blast-furnace flue dust and fine ores. Open-hearth furnaces of the rotating or oscillating type, with oil, gas, or powdered-coal firing, are in use for remelting cast Fe and producing wrought Fe. An improved cupola has several rows of tuyères connected to the wind belt, the bottom row being fitted with valves. The reduction of the air supply at the bottom tuyères increases the CO content of the gases in the lower portion of the coke bed and reduces metal oxidation and erosion of the furnace lining. The air introduced through the higher tuyères raises the temp. as the top of the coke bed is approached.

D. K. MOORE.

Influence of the cooling velocity and melting temperature on the graphitisation of cast iron. H. TANIMURA (Mem. Coll. Eng. Kyushu, 1931, 6, 115–188).—Measurements of the influence of the rate of

cooling on the graphitisation of cast Fe containing various quantities of C and Si show that for each series of const. C content there is a limiting Si content above which graphitisation takes place readily at any rate of cooling. At a lower Si content slower cooling is necessary, and at the lowest Si contents separation of graphite takes place only with difficulty. The cooling rate necessary to produce white cast Fe diminishes with increase of the melting temp., whilst with slow cooling the influence of melting temp. on the degree of graphitisation varies considerably with the composition of the Fe. Melting at high temp. produces frequently a heterogeneous structure in the cast Fe, and if the cooling is rapid or the C content low the graphite separates in a fine granular form; it is probable that indirect graphitisation takes place, whereas the separation of graphite is direct when the Fe is melted at a low temp. The influence of the melting temp. on the stability of the cementite of a white cast Fe has been studied.

H. F. GILLBE.

Steel production in large coreless induction furnaces. F. PÖLZGUTER (Stahl u. Eisen, 1931, 51, 513–520).—A medium-frequency induction furnace for melting 1 ton of steel is described with reference to numerous diagrams and its efficiency is compared with that of a 7-ton arc furnace. The relative energy efficiencies are 63 and 66%, respectively, but 66% of the total energy loss in the first case is accounted for by transforming losses. On the other hand, oxidation and casting losses are only 1–2% in the induction furnace as compared with 2.5–4.5% in the arc furnace, and the former permits a more rapid and efficient removal of S and P and a greater and more ready control of the composition of the finished steel. A. R. POWELL.

Effect of degree of reduction by rolling, final rolling temperature, and heat treatment on the mechanical properties, sensitivity to ageing, and structure of thick [steel] sheets. F. KÖRBER and K. WALLMANN (Stahl u. Eisen, 1931, 51, 491–497).—The elastic limit and yield point of mild Si steel are raised without affecting the ultimate strength by severe hot-rolling, finishing at a low temp. (e.g., 700°), and the resulting sheet has a close fine-grained structure. A low finishing temp. for rolling Si steel with a low C content (0.09%) results in low values being obtained in the notched-bar impact test, but for steel containing 0.2–0.4% C this treatment has little effect on the impact strength. Annealing just below the A3 point produces a fine-grained structure without greatly improving the elongation and reduction in area values obtained in the tensile test, but retarded cooling produces a coarser grain structure and poor mechanical properties. The sensitivity to ageing decreases with severity of rolling, i.e., thin sheets are less prone to suffer deterioration from ageing phenomena than are thick sheets. A. R. POWELL.

Crystallographical investigation of some mechanical properties of metals. V. Fatigue of metals under alternating torsion. Y. KIDANI (J. Fac. Eng., Tokyo, 1931, 19, 177–190).—The behaviour of annealed steel with 0.1, 0.3, and 0.6% C and of Cu under a const. alternating torque up to the plastic range has been

investigated. The repetition curve shows that the transition point between the hardening and fatigue effects coincides with the beginning of fatigue in the individual grains and indicates that there is an energy relation between the slip strain corresponding to the applied torque and the number of repetitions required to cause fracture. The fatigue of Cu is chiefly due to the growth of twinning slip bands which develop in the axial or peripheral direction of the grains according to their orientation. The rate of damping of the vibration of the specimens is a measure of the degree of fatigue of the metal.

A. R. POWELL.

**Determination of sulphur [in iron and steel] by the evolution method; influence of volatile phosphorus [compounds].** F. JUNGBLUT (Ann. Chim. analyt., 1931, [ii], 13, 161—170).—The chief cause of error in the determination of S in ferrous alloys by the evolution method is the absorption of  $\text{PH}_3$  by the  $\text{Cd}(\text{OAc})_2$  solution. This may be avoided by passing a rapid current of  $\text{CO}_2$  through the absorption vessel before the addition of the I solution. The amount of P evolved from mild steels is 60—70% of the total present, whereas <20% of the total P is evolved on dissolving cast Fe in HCl.

A. R. POWELL.

**Detection of traces of nickel and cobalt in steel without destruction of the sample.** R. JIRKOVSKY (Chem. Listy, 1931, 25, 254—256).—A current is passed from an anode made of the steel under examination, through a filter paper soaked in ammoniacal dimethylglyoxime solution, to a metal anode. The paper is then soaked in a solution containing  $\text{K}_2\text{C}_2\text{O}_4$  and  $\text{K}_4\text{Fe}(\text{CN})_6$ , washed, and the blue coloration due to Fe is removed by 10% KOH; a red coloration indicates the presence of Ni. Co is detected similarly by using paper soaked in a solution containing  $\text{NH}_4\text{CNS}$ ,  $\text{NH}_4\text{OAc}$ , and tartaric acid. An orange stain, due to Fe, appears; this changes to blue on extraction with  $\text{Et}_2\text{O}$  (in presence of Co), or green (Ni), or blue-green (Co + Ni).

R. TRUSZKOWSKI.

**Determination of molybdenum in steel.** H. C. WEIRICK and C. H. MCCOLLAM (Heat Treat. Forg., 1930, 16, 1145—1146, 1155).—If Al or V is present the sulphide precipitation or Maag and McCollam's colorimetric method should be used. Precautions are indicated.

CHEMICAL ABSTRACTS.

**Relation of the moisture in rust to the critical corrosion humidity.** W. S. PATTERSON and L. HEBBS (Trans. Faraday Soc., 1931, 27, 277—283).—Samples of natural rust from various sources have been dried and then exposed to atm. of gradually increasing and then gradually decreasing humidity, the change in wt. under these conditions being observed. The marked hysteresis which is observed in the values obtained in the two cases are interpreted as indicating that Fe rust has a gel structure, the results being almost identical with those obtained with precipitated  $\text{Fe}(\text{OH})_3$ . The  $\text{H}_2\text{O}$  first absorbed by the gel must be held very tenaciously, and not be available for promoting the corrosion. At above the crit. humidity it is supposed that the capillaries in the gel commence to fill with  $\text{H}_2\text{O}$ , which furthers the corrosion process.

J. W. SMITH.

### Reaction for the detection of corrosion of underground water-pipes and cables by stray currents.

MEDINGER (Z. angew. Chem., 1931, 44, 550—551).—Several cases of corrosion of underground Fe water-pipes were found to be due to migration of Cl ions towards the Fe pipe under the action of stray d.c.; in one case, although the surrounding earth contained only traces of chlorides, the corrosion product on the Fe contained 23.5% Cl. It is therefore concluded that the accelerating action of stray currents on corrosion is due entirely to anodic migration of Cl ions, and, conversely, the fact that a corrosion product contains a high % of Cl is regarded as sufficient indication of the presence of stray currents.

A. R. POWELL.

**Evans pile and protection [of iron] by buffer action.** E. HERZOG and G. CHAUDRON (Bull. Soc. chim., 1931, [iv], 49, 702—703).—The Evans pile (cf. A., 1930, 1126) has been constructed with 0.5N-NaCl as the electrolyte, a horizontal cathode of which the upper surface functions as the oxygenated electrode and the lower is insulated by a film of varnish, a vertical sol. electrode, and a diaphragm. The e.m.f. increases at first to a const. value of 0.4—0.43 volt, whereas the current increases with rise of the  $\text{O}_2$  pressure. The effect is due to the formation of alkaline and acid films at the cathode and anode, respectively, and, unlike the Fe-C pile, the pile is almost completely polarised by buffer agents.

H. F. GILLBE.

**The Parker and Bonderite processes [for protecting iron], and their bearing in paint technology.** F. KOLKE (Farben-Ztg., 1931, 36, 1687—1690, 1727—1728).—These proprietary processes, depositing films of Fe Mn phosphates and Cu phosphates, respectively, on Fe, are described in detail. "Parkerising" is a rustproofing treatment, which, however, is usually "fixed" by application of suitable oils etc., whilst "Bonderising" provides a chemical undercoat for subsequent painting etc. The results of comparative tests of lacquers, varnishes, and japans over parkerised and unparkerised plates are tabulated: they show that in some cases parkerising delays the breakdown of the system. Bonderising of metal is shown to improve the adhesion of paint films thereto.

S. S. WOOLF.

**Flash-combustion roasting of pyrites.** M. FREEMAN (Chem. Met. Eng., 1931, 38, 334—336).—Pyrites ground to 200-mesh, as produced by flotation, is difficult to roast in ordinary mechanical furnaces on account of the very rapid combustion. In the flash-combustion process the ore is passed through a ball mill and then blown with warm air into a firebrick combustion chamber, where it meets a further upward current of air. The gases, after passing through a dust chamber and a fire-tube boiler, are rapidly cooled from 1100° to 400°. The rapid cooling minimises  $\text{SO}_3$  formation. No trouble with slag occurs in the combustion chamber if sufficient space is given. The yield of S as  $\text{SO}_2$  exceeds 98%, and 1 lb. of steam is produced per lb. of pyrites burned. The burnt residue contains 0.2% S. The process is intended for use in paper mills.

C. IRWIN.

**Combustion of pyrites in a Herreshoff furnace.** F. W. ADAMS (Chem. Met. Eng., 1931, 38, 337—338).—

Analyses of pyrites fines passing through a 12-shelf air-cooled Herreshoff furnace were made for total sulphide- and sulphate-S on each shelf, the temp. being recorded. At no point was more than 3% of the S present in the form of sulphide (i.e., FeS [?]). Sulphate formation does not occur above 600°. In the furnace studied it was found desirable to fit radiation coolers to the top six shelves to distribute the heat more evenly through the furnace. C. IRWIN.

**Laboratory study of the atmospheric corrosion of metals. I. Corrosion of copper in certain synthetic atmospheres, with particular reference to the influence of sulphur dioxide in air of various relative humidities.** W. H. J. VERNON (Trans. Faraday Soc., 1931, 27, 255—277).—In dry air, concentrations of SO<sub>2</sub> up to 10% produce no visible corrosion of Cu. In the presence of H<sub>2</sub>O vapour corrosion occurs at all concentrations. The process is controlled by the catalytic oxidation of SO<sub>2</sub> at the metal surface, the rate of attack falling off as the active centres are used up. Between 63 and 75% R.H. there is generally a great increase in the rate of attack, indicating the existence of a crit. humidity lying between these values. The corrosion is minimal at about 1% SO<sub>2</sub>, at which concentration the product is CuSO<sub>4</sub>. At lower concentrations it is accompanied by excess of base, and at higher by excess of H<sub>2</sub>SO<sub>4</sub>. CO<sub>2</sub> present in large excess over the SO<sub>2</sub> has no influence, but HCl pursues an independent attack on the metal. High-conductivity Cu is more easily attacked by SO<sub>2</sub> than is arsenical Cu. The hygroscopicity of the dried corrosion product shows a marked increase between 50 and 75% R.H., corresponding with the crit. humidity. The influence of the latter is less pronounced with arsenical Cu than with high-conductivity Cu, but the products are less hygroscopic, providing an additional explanation for the greater corrosion-resistance of arsenical Cu. J. W. SMITH.

**Aluminium light alloy "Chlumin."** I. JITAKA (Proc. Imp. Akad. Tokyo, 1931, 7, 161—164).—An Al alloy containing Cr, Mg, and Fe has been made which is extremely resistant to corrosion by sea-water. The composition is not given. W. E. DOWNEY.

**Changes in the dimensions of metallic wires produced by torsion. II. Silver, gold, aluminium, and nickel. III. Lead.** T. LONSDALE (Phil. Mag., 1931, [vii], 11, 1169—1187, 1187—1196; cf. A., 1930, 23).—Full data are tabulated, and an expression is found relating the elongation, twist, initial tension, and diam. for wires of the 4 metals of the face-centred cubic class twisted under a small const. load.

III. Corresponding investigations were made for Pb. The elongations are considerably greater, and depend on the rate of twisting. N. M. BUGH.

**Determination of the precious metals in Transvaal platinum ores and concentrates.** H. R. ADAM and R. J. WESTWOOD (J. Chem. Met. Soc. S. Afr., 1931, 270—273; cf. B., 1929, 213).—The precious metals are collected in Pb in the usual way and the button is cupelled without addition of Ag; the resulting leady bead is heated at 1300° in a gentle current of air for 1—2 hr. to remove the remaining Pb. The bead is

then dissolved in 1:3 aqua regia, the solution evaporated repeatedly with HCl to remove HNO<sub>3</sub>, any insol. material collected (A), and the filtrate treated with solid NH<sub>4</sub>Cl to ppt. the Pt. The (NH<sub>4</sub>)<sub>2</sub>PtCl<sub>6</sub> ppt. is washed with saturated NH<sub>4</sub>Cl and dissolved in hot H<sub>2</sub>O, the solution boiled with formic acid and NH<sub>4</sub> formate, and the Pt ppt. collected, ignited, and weighed. The NH<sub>4</sub>Cl filtrate is diluted and treated with 1% nitroso-β-naphthol to ppt. Au and Pd; the ppt. is washed with 5% AcOH, ignited, weighed (Au + Pd), and cupelled with Ag. The bead is parted in HNO<sub>3</sub> to obtain Au, Pd being obtained by diff. The filtrate from the Au + Pd is treated with HCl, Pb(OAc)<sub>2</sub>, and Zn dust, the ppt. is scorified with residue A and cupelled with Ag, and the bead parted in 1:1 HNO<sub>3</sub> to obtain a residue of "other Pt metals." A. R. POWELL.

**Rumanian ores and improved extraction of metals.** N. METTA (An. Minelor România, 1931, 14, 57—61; Chem. Zentr., 1931, i, 1896).—A discussion of Transylvanian mines and minerals. A. A. ELDRIDGE.

**Buffer action in nickel-plating solutions.** K. PITSCNER (Metal Ind., 1931, 29, 119—120).—In the usual plating range of pH 6—5 the buffer effect of H<sub>3</sub>BO<sub>3</sub> and NH<sub>4</sub>Cl is greater when alkali is added, whilst about pH 2.5 the reverse is the case. The NH<sub>4</sub>Cl content of Ni solutions is kept high enough for the anode corrosion to keep the trend of the pH val. towards the upper limit of the range. Addition of acid only is necessary. CHEMICAL ABSTRACTS.

**[Ore] flotation.** B. KAMIEŃSKI (Przemysł Chem., 1931, 15, 201—202).—The separation of galena by flotation with oil is due to adsorption of positively-charged galena particles on the oil-water interface. The addition of CuSO<sub>4</sub> to a blende suspension changes the negative charge of the particles to a positive one, as a result of the formation of a coating of CuS. R. TRUSZKOWSKI.

**Effect of particle size on flotation.** A. M. GAUDIN, J. O. GROH, and H. B. HENDERSON (Amer. Inst. Min. Met. Eng. Tech. Pub., 1931, No. 414, 3—23).—There is a time sequence of flotation with regard to size, the medium coarse particles floating first. Extremely fine pulps were not successfully floated. Recovery is optimal in one range and selective in another. CHEMICAL ABSTRACTS.

**Furnaces.**—See I. Coal seams [for blast furnaces]. Steel tubes for N determinations. Core oils for foundries.—See II. Ferrum reductum and pulveratum.—See XX. Protecting water-conduits.—See XXIII.

See also A., July, 791, Single crystals of Ag. Allotropism of Rh. 794, Solubility of Au in Hg, and of Cu in Ag. 805, Prep. of α- and β-W, and of La and Nd. 813, Determination of Cu, of CuSeO<sub>3</sub> in presence of Cu<sub>2</sub>Se, and of Al<sub>2</sub>O<sub>3</sub> in Al and its alloys. 814, Assaying of Pt and Pd. Determination of Os. 816, Ni-Fe alloys for instruments.

#### PATENTS.

**Manufacture of coated iron or steel articles.** PARKER RUST PROOF CO. (B.P. 350,560, 14.3.30. U.S., 22.4.29).—The articles are boiled in a dil. solution

containing  $\text{H}_3\text{PO}_4$ ,  $\text{Fe}_3\text{P}_2\text{O}_8$ , and a small amount of  $\text{Cu}_3\text{P}_2\text{O}_8$ , whereby they become coated with an insol. phosphate film impregnated with metallic Cu. After drying, the surface is heated with an oxidising flame to convert the Cu into  $\text{CuO}$ . A. R. POWELL.

**Manufacture of coated iron and steel articles.** W. W. TRIGGS. From PARKER RUST PROOF CO. (B.P. 350,559, 14.3.30).—The articles are boiled in a dil. acid phosphate solution containing colloidal  $\text{H}_4\text{SiO}_4$ . A. R. POWELL.

**Case-hardening of metals [iron].** W. W. TRIGGS. From AMER. CYANAMID CO. (B.P. 349,547, 21.11.29).—The case-hardening bath comprises a fused mixture of 2 pts. of  $\text{CaCl}_2$  and 1 pt. of  $\text{NaCl}$  to which is added occasionally 0.5–1% of  $\text{Ca}(\text{CN})_2$ . [Stat. ref.] A. R. POWELL.

**Manufacture of ferro-metal magnetic alloys.** INTERNAT. NICKEL CO., INC., Assees. of A. S. SHOFFSTALL and A. E. KAYES (B.P. 350,603, 21.3.30. U.S., 19.4.29).—Fe and Ni in the desired ratio are melted together with enough C to give an alloy containing 0.02–0.03% C, the liquid is covered with a refining flux consisting of  $\text{CaO}$ ,  $\text{CaF}_2$ , and a little tar coke, and 0.25% Mn is added to assist in removing P and S. The refined alloy is then deoxidised with a Ca–Si or Zr–Si alloy when the Ni content is 45–55% and with a 25 : 5 : 70 Mg–Si–Ni alloy when the Ni content is 70–80%. The resulting ingots have a high ductility and are readily forged or rolled hot. A. R. POWELL.

**Manufacture of [iron] alloys from pulverulent initial materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,587, 23.12.29).—Finely-divided Fe or  $\text{Fe}_2\text{O}_3$  from  $\text{Fe}(\text{CO})_5$  is mixed with a solution containing a compound of the alloying metal and the mixture is heated under conditions which result in the reduction of the mass to a porous sinter, which is then consolidated by pressure and worked into sheet by sintering in a reducing atm. and hot-working. *E.g.*, 4 pts. of Fe powder are made into a paste with 1 pt. of a 20% solution of  $\text{Cr}(\text{NO}_3)_3$  and the mixture is slowly heated to 500° until  $\text{N}_2\text{O}_4$  ceases to be evolved, then reduced in  $\text{H}_2$  at 1000° for 24 hr. to obtain a sintered Fe–Cr alloy. An alloy of Fe with Co and Ni is made by heating  $\text{Fe}_2\text{O}_3$  from  $\text{Fe}(\text{CO})_5$  with a solution of  $\text{Ni}(\text{CO})_4$  and  $\text{Co}(\text{CO})_4$  in  $\text{C}_6\text{H}_6$  at 400° under pressure and reducing the resulting powder with  $\text{H}_2$  at 1000°. A. R. POWELL.

**Manufacture of [chromium] steel [for boiler tubes].** D. COLVILLE & SONS, LTD., and A. McCANCE (B.P. 350,048, 12.3.30).—Steel containing 4–10 (7.36)% Cr, 0.2–0.7 (0.36)% Si, 0.05–0.20 (0.105)% C, and 0.2–0.45 (0.33)% Mn is claimed. The Brinell hardness remains const. at 121 between 15° and 760°. A. R. POWELL.

**Treatment of wire and rods to be drawn with simultaneous use of trisodium phosphate lyes as neutralisation means and drain oils as lubricant.** R. W. MOLL and H. VOM BRUCK (B.P. 350,275, 2.7.30. Ger., 27.11.29).—The material to be prepared for drawing is first pickled in an acid bath, then washed in  $\text{H}_2\text{O}$ , dipped in  $\text{Na}_3\text{PO}_4$  lye, and subsequently dried in air; as lubricants for cold-drawing, drain oils (*i.e.*, oils which

have already been used as lubricant and drained off) are employed. H. ROYAL-DAWSON.

**Protection of iron or steel.** H. SUTTON and A. J. SIDERY (B.P. 350,469, 3.3.30).—A thin film of Zn is electrodeposited on the cleaned surface of the iron (*etc.*) before the latter is immersed in a bath of molten Al or Al alloy maintained at 600–800° for 1–2 min. [Stat. ref.] H. ROYAL-DAWSON.

**[Copper–zinc–manganese] alloy.** J. N. WHITE (U.S.P. 1,785,372, 16.12.30. Appl., 12.11.29).—An alloy comprising at least 88 (93–95)% Cu, 0.5–4 (1–1.25)% Mn, and the remainder Zn is suitable in the construction of flexible tubes *etc.* H. ROYAL-DAWSON.

**Roasting and sintering of pulverulent ores having a high sulphur content.** METALLGES. A.-G. (B.P. 350,828, 1.9.30. Ger., 28.9.29).—A furnace of the powdered-coal-burning type is arranged above the hearth of a blast-roasting apparatus and fired with these ores instead of with coal dust. The burning ore thus falls on to the ore charge on the hearth and the two are roasted *etc.* together. Additional fuels may be necessary. H. ROYAL-DAWSON.

**Froth-flotation concentration of [zinc–lead] minerals.** MINERALS SEPARATION, LTD., H. LAVERS, and A. H. HIGGINS (B.P. 349,591, 31.1.30).—The addition of small quantities of  $\text{Na}_3\text{AsS}_4$  or of a mixture of  $\text{Na}_2\text{S}_x$  and  $\text{As}_2\text{S}_3$  to the ore pulp to prevent flotation of ZnS in the selective flotation of PbS from complex Pb–Zn ores is claimed. A. R. POWELL.

**Treatment of materials [old accumulator plates] containing lead.** C. R. HAYWARD (B.P. 349,607, 28.2.30).—The material is smelted at 800° with a flux consisting of  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7$ , and  $\text{CaF}_2$  and air is blown through the mass to remove As, Sb, and Sn. The flux is removed and regenerated by heating with carbonaceous material to reduce its metal content to an Sb–As–Sn–Pb alloy. A. R. POWELL.

**Electrodeposition of zinc.** U. C. TAINTON (B.P. 349,432, 26.11.29).—Electrolysis of  $\text{ZnSO}_4$  solutions is improved by the use of Pb anodes containing 1% of Ag, Bi, or As and by adding to the bath  $\text{Na}_2\text{SiO}_3$  equiv. to 5 lb. of  $\text{SiO}_2$  and 1–2 lb. of gum arabic per ton of Zn deposited. A. R. POWELL.

**Extraction of metals from ores and other materials.** NAT. PROCESSES, LTD., and A. R. GIBSON (B.P. 349,563, 29.11.29).—The valuable metal is leached out by means of a cold solution containing  $\text{NH}_3$  and  $\text{SO}_2$ . *E.g.*, ZnO is extracted from Waelz flue dust with a solution containing 7.5%  $\text{NH}_3$  and 9.2%  $\text{SO}_2$ ; Sn is removed from tinplate with a solution containing 7.5%  $\text{NH}_3$  and 27%  $\text{SO}_2$ ; Ni, Co, and Cu from hydroxide residues with a solution containing 6%  $\text{NH}_3$  and 8%  $\text{SO}_2$ ; and Cu from roasted material containing Sb and As with the same solution. Sb and As are removed from the residues from the last-named operation by extraction with a solution containing 7.5%  $\text{NH}_3$  and 27%  $\text{SO}_2$ . A. R. POWELL.

**Aluminium alloy.** H. C. HALL (B.P. 350,110, 1.4.30).—The alloy consists of Fe 1.5–3.0%, Mg 0.7–3.0%, Cu 0.3–2.5%, Si 1.0–4.5%, and remainder Al with <0.2% Ni. H. ROYAL-DAWSON.

**Electrolytically depositing aluminium.** ALUMINIUM-IND. A.-G. (B.P. 349,842, 30.7.30. Ger., 24.8.29).—Articles may be electroplated with Al in a bath comprising a molten 1:3 mixture of NaCl and AlCl<sub>3</sub> containing a small quantity of PbCl<sub>2</sub>, using an Al anode and a c.d. of < 9.3 amp./sq. ft. Better adhesion is obtained by making the article the anode for a short time before reversing the current.

A. R. POWELL.

**Production of self-improving aluminium alloys.** A.-G. DER EISEN- U. STAHLWERKE VORM. G. FISCHER, and MESSINGWERK SCHWARZWALD A.-G. (B.P. 349,463, 24.2.30. Ger., 27.2.29).—An alloy of Al with 1–25% Cu and 0.1% Ni is treated in the ladle, just prior to casting, with 0.2–5.0% of a binary alloy of Mg with Ni, Cu, Zn, or Sn so that the resulting casting may contain 0.1–0.25% Mg.

A. R. POWELL.

**Surface treatment of aluminium and its alloys.** R. W. CARTER (B.P. 349,596, 21.2.30).—The surface of rolled Al having been rubbed with a mixture of fine pumice powder and Ca(OH)<sub>2</sub>, the metal is immersed in a hot bath containing 2 oz. of NaOH and 0.5 oz. of Na<sub>2</sub>HPO<sub>4</sub> per gal. and then washed with a solution containing 0.5 oz. of HNO<sub>3</sub> and 2 oz. of alum per gal.

A. R. POWELL.

**Aluminium alloys.** H. C. HALL and T. F. BRADBURY (B.P. 350,446—7, 8.3.30. Addn. [A] to B.P. 323,353; B., 1930, 198; [B] to B.P. 334,430; B., 1930, 1034).—The alloys previously claimed are modified by replacing 0.01–5% of the Al content with a corresponding amount of Zn.

A. R. POWELL.

**Aluminium alloys.** H. C. HALL and T. F. BRADBURY (B.P. 350,021—2, 8.3.30. [A] Addn. to B.P. 323,353; B., 1930, 198; [B] to B.P. 334,430; B., 1930, 1034).—The alloys of the prior patents are altered so that, in (A), the Ni content is 2.5–4.5% and the Si 2.8–5%, and in (B) the alloy contains 2.5–5.5% Ni.

H. ROYAL-DAWSON.

**[Aluminium-bronze] alloys.** H. C. ANSTAY and T. BOLTON (B.P. 349,734, 23.4.30).—Al bronze containing 4–11% Al, 0.02–0.25% V, about 2% Ni, and about 1% Fe is claimed. Alloys with 4–7.35% Al and free from Fe and Ni may be worked readily cold, but for hot-working alloys with 7.35–11% Al, 0.1% V, 2% Ni, and 1% Fe are preferred.

A. R. POWELL.

**Bessemerising of nickel-containing mattes.** H. WADE. FROM INTERNAT. NICKEL CO., INC. (B.P. 349,700, 28.3.30).—Ni or Ni-Cu matte containing 18–20% S is blown in a converter at 1330–1500° with a 9:1 mixture of steam and air preheated to 500° until the S is reduced to 2–4%. The temp. is then raised to about 1600° and the material blown with superheated steam without air until the resulting metal contains 0.005–0.02% S. The charge is then deoxidised with C, Si, Mg, Al, or Ca. The two-stage blowing operation is preferable to the known single-stage process in that it prevents excessive corrosion of the refractory lining.

A. R. POWELL.

**Tank for chromium plating.** C. R. GLEASON (U.S.P. 1,784,987, 16.12.30. Appl., 3.6.27. Renewed 14.3.30).—Electrolyte is contained in a Pb-lined wooden container having an inner lining of glass.

J. S. G. THOMAS.

**Control of electroplating.** R. J. PIERSOL (U.S.P. 1,785,389, 16.12.30. Appl., 14.1.29).—An undulating d.c. is employed to reduce the effects of polarisation, e.g., in Cr-plating.

J. S. G. THOMAS.

**Union of [tungsten-nickel wire] metal surfaces by welding.** WESTINGHOUSE LAMP CO., ASSEES. OF J. B. FITZPATRICK (B.P. 350,296, 16.7.30. U.S., 17.7.29).—W filaments are joined to Ni lead-in wires by spot-welding with 75 amp. at 0.6 volt for 10 sec. so that the molten Ni covers the whole of the portion of recryst. W wire and part of the surrounding non-recryst. portions of the wire.

A. R. POWELL.

**Recovery of molybdenum from mixtures of molybdenum or molybdenum compounds with other metals or metal compounds.** J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 350,135, 14.4. and 24.10.30).—MoO<sub>3</sub> catalysts which have been used for the hydrogenation of oils are extracted with a solvent for the oil, heated at 350° to remove the solvent, and roasted at 400–600°, preferably 480°, in a current of air or of N oxides, any sol. metal oxide, e.g., ZnO, is removed by heating with a mixture of 12% HCl and 25% HNO<sub>3</sub>, and the MoO<sub>3</sub> extracted from the residue with aq. NH<sub>3</sub> and sufficient (NH<sub>4</sub>)<sub>2</sub>S to ppt. any other sol. metal as sulphide. The Mo is recovered from the filtered solution by addition of a slight excess of HCl.

A. R. POWELL.

**Solder [for aluminium etc.].** F. J. LENSSENS (B.P. 350,649, 11.4.30).—An alloy melted in the presence of a small quantity of resin and tallow containing, approx., 40–60 pts. each of Zn and Sn, 1–10 pts. Cd, and 0.5–5 pts. Fe is claimed.

H. ROYAL-DAWSON.

**[Soft-]soldering fluxes.** GRASSELLI CHEM. CO., ASSEES. OF (A) H. B. DYKSTRA, (B) H. J. BARRETT (U.S.P. 1,785,115 and 1,785,173, 16.12.30. Appl., 7.5.30).—The fluxes claimed are, in (A), chlorinated rosin and, in (B), polymerised vinyl esters of aliphatic acids, e.g., vinyl formate, acetate, propionate, and butyrate.

H. ROYAL-DAWSON.

**Soldering materials.** GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 349,873, 17.9.30. Ger., 28.9.29).—Moulded pellets, comprising a mixture of the powdered soldering metal with an org. amine, acetamide, urea, or a cyanamide derivative and a flux, e.g., NH<sub>4</sub>Cl, ZnCl<sub>2</sub>, or (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, are claimed.

A. R. POWELL.

**Casting of ingots.** T. D. STAY and W. HOLZHAUER (B.P. 348,531, 10.2.30. U.S., 11.2.29).—See U.S.P. 1,777,657; B., 1931, 639.

**Producing a phosphate coating on metal.** PARKER RUST PROOF CO. (B.P. 350,565, 14.3.30. U.S., 29.4.29).—See U.S.P. 1,755,391; B., 1930, 1034.

**Protectively coating the external surface of pipes and other externally cylindrical bodies.** SOUTH DURHAM STEEL & IRON CO., LTD., and D. ROSS (B.P. 350,601, 20.3.30).

Zr ores.—See VIII. Magnetic separators.—See XI.

## XI.—ELECTROTECHNICS.

**Examinations with ultra-violet light.** M. GUYOT (Ann. Falsif., 1931, 24, 196–207).—A résumé of the various methods which have been used for examining



both solids and liquids in ultra-violet light. Recommended procedure is given for various classes of materials. The characteristic colours of the fluorescence exhibited by various types of artificial silk are tabulated. A method for determining the fluorescence of liquid extracts of drugs and also of oils is given; it is stated to be applicable to the analysis of mixtures.

E. B. HUGHES.

Measurement of smoke.—See I. Switch oils.—See II. Felspar.—See VIII. Steel production. Detecting Ni and Co in steel. Evans pile and protection of Fe. Ni-plating.—See X.

See also A., July, 1935, Photo-electric cells. 790, Magnetisation-temp. curves of Fe, Co, and Ni. 795, "Getters." 805, Prep. of  $\alpha$ - and  $\beta$ -W, and of La and Nd. Method of rapid electrolysis. (CN)<sub>2</sub> synthesis. Cathodic projection of elements. Light sources for photochemical reactions. 809, Formation of N fluorides.

#### PATENTS.

Electric furnaces [for glass]. BRIT. HARTFORD-FAIRMONT SYND., LTD., and E. MEIGH (B.P. 350,474 and 350,476, 6.3.30).—(A) The distance between opposed electrode surfaces making contact with the material to be heated and forming the resistance is greater where the material is more fluid. (B) Continuous electrodes extending along substantially the whole length of the melting chamber are arranged along the direction of motion of the material being treated.

J. S. G. THOMAS.

Manufacture of crucibles [for electric induction furnaces etc.]. W. R. WHITNEY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,784,647, 9.12.30. Appl., 3.11.26).—Refractory material, *e.g.*, fireclay, mixed with C or graphite is moulded and fired in a reducing atm. and finally heated in the air to burn out the C or graphite from the outer layer of refractory material.

J. S. G. THOMAS.

Electromagnetic separators. Magnetic separators. H. H. THOMSON, A. E. DAVIES, and W. E. BOX (B.P. [A] 350,281, 7.7.30, and [B] 350,049, 13.3.30).—(A) Transverse magnetic bars having upper surfaces of convex or other form alternating with spaced armature bars having lower surfaces of similar form are arranged on the floor of an inclined trough or tray, to form a sinuous passage of substantially uniform cross-section for passage of material under treatment. (B) A pair of spaced magnet plates provided with staggered or interspaced projections are arranged within a recess in a non-magnetic bench for testing scrap metal, and an electromagnet having its poles in contact with the plates is arranged beneath the bench. [Stat. ref. to (B).]

J. S. G. THOMAS.

[Copper oxide-caustic alkali] galvanic cell. G. W. HEISE and C. W. BROKATE, Assrs. to NAT. CARBON Co., Inc. (U.S.P. 1,786,406, 23.12.30. Appl., 16.2.23).—Org. materials, *e.g.*, the product obtained by treating wood with NaOH, are added to the electrolyte of a cell comprising a container, a metallic anode, caustic electrolyte, and a layer of cryst. material substantially insol. in the electrolyte, *e.g.*, sand or alundum, which

presents a surface favourable to crystallisation of compounds of the anode metal. J. S. G. THOMAS.

Means for reinforcing dry-battery electrolyte. G. W. HEISE and E. A. SCHUMACHER, Assrs. to NAT. CARBON Co., Inc. (U.S.P. 1,784,592, 9.12.30. Appl., 28.5.28).—The caustic alkali gel forming the electrolyte in a Zn-Cu cell with CuO depolariser is reinforced with fibrous material, *e.g.*, glass wool, asbestos, or "excelsior."

J. S. G. THOMAS.

[Gas-filled] light-sensitive cells. L. KECSKEMETI-KAYE (B.P. 350,032, 17.11.30).—The envelope contains a mixture of He, Ne, Xe, Kr, and H<sub>2</sub>, about equal vols. of the gases being employed. H. ROYAL-DAWSON.

[Hot-cathode] electronic-discharge apparatus. RADIOTECHNIQUE (B.P. 350,605, 21.3.30. Fr., 9.4.29).—An indirectly heated cathode comprising at least one layer containing Co, *e.g.*, Ni-Co alloy, is, in operation, heated below the Curie point of that layer.

J. S. G. THOMAS.

[Gas-filled] electric glow-discharge lamps. WESTINGHOUSE ELECTRIC & MANUFACTURING Co., Assees. of D. D. KNOWLES (B.P. 349,013, 5.2.30. U.S., 5.2.29).—A gas-filling composed of about 90% of Ne and about 10% of H<sub>2</sub>, or other gas having a critical energy level for a change of physical state which is less than the energy level for excitation of Ne, is claimed. [Stat. ref.]

J. S. G. THOMAS.

Electric-discharge tubes. N.V. PHILIPS GLÖHLAMPENFABRIK (B.P. 350,273, 1.7.30. Holl., 18.7.29).—The wall of the tube consists, at least in part, of one or more windows of Zr metal, which permit the X-rays or cathode rays to pass from the vacuum space into that surrounding the tube. H. ROYAL-DAWSON.

Arc-quenching material. G. STEERUP, Assr. to ELECTRICAL ENGINEERS EQUIPMENT Co. (U.S.P. 1,786,581, 30.12.30. Appl., 19.3.28).—A mixture composed of CCl<sub>4</sub> (85–97%) and 15–3% of a mixture of Al stearate (70%) and Al palmitate (30%) is claimed.

J. S. G. THOMAS.

Incandescence body for electrical incandescence lamps, vacuum tubes, etc. L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 350,204, 20.5.30).—The body throughout consists of Re or Re-W alloy. [Stat. ref.]

H. ROYAL-DAWSON.

Film formation and operation of electrolytic condensers etc. R. D. MERSHON (U.S.P. 1,784,674, 9.12.30. Appl., 14.7.23).—A filmed anode is immersed in an electrolyte contained in a Cu vessel and means are provided for electroplating injurious metals, *e.g.*, Ni, Sn, Ag, contained in the electrolyte on to the Cu vessel.

J. S. G. THOMAS.

Electrical sealing compositions. BAKELITE CORP., Assees. of M. E. DELANEY (B.P. 349,982, 5.3.30. U.S., 6.3.29).—A composition composed of about 39% of a chlorinated C<sub>10</sub>H<sub>8</sub> (especially C<sub>10</sub>H<sub>4</sub>Cl<sub>4</sub>), 27% of gilsonite, and 34% of montan wax is claimed.

J. S. G. THOMAS.

Formation of an insulating coating on parts of electrical apparatus. P. BRABANT (B.P. 315,798, 10.11.30).—Apparatus made of Al, Mg, or their alloys is immersed in an oxidising bath and heated electrically until the liquid in the bath boils. J. S. G. THOMAS.

**Manufacture of electric insulating material.** INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS-GES. (B.P. 350,429, 4.3.30. Ger., 4.3.29).—Strips of paper, impregnated with a solution of an artificial resin and subjected to heat treatment which renders the resin less readily fusible and insol., are coated on one side with the same or another artificial resin and wound under pressure and heat treatment.

J. S. G. THOMAS.

**Insulators for sparking plugs.** I. G. FARBENIND. A.-G. (B.P. 344,256, 3.4.30. Ger., 22.4.29. Addn. to B.P. 340,865).—The head piece of the insulators, which is exposed to high temp. in the combustion chamber, is made of corundum, the surface of which is made smooth by treatment with fused borax.

J. S. G. THOMAS.

**Electric gas purifier.** H. F. J. NOLZE (U.S.P. 1,785,592, 16.12.30. Appl., 24.5.26).—Apparatus comprising electrodes between which the gas is subjected to an electric discharge, an exhaustor having baffles which can be moved into or out of the exhaustor according to the temp. of the gas from the purifier, nozzles for injecting water in the exhaustor, and means for controlling the amount of spray according to the temp. of the plant is claimed.

J. S. G. THOMAS.

**Treatment of colloids.** A. M. HERBSMAN (U.S.P. 1,783,471, 2.12.30. Appl., 15.4.29).—A stream of colloid is exposed to radiation of short wave-length, e.g., to the radiation from a quartz-Hg-vapour lamp or X-ray tube, and the various phases are separated gravitationally.

J. S. G. THOMAS.

**[Electrical] testing of gas.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF G. E. INMAN (B.P. 349,204, 22.3.30. U.S., 23.3.29).—The purity of a gas which is being supplied for a manufacturing operation, e.g., for filling electric incandescence lamps, is controlled by passing the gas through a vessel through which a high-frequency electric discharge is also being passed, and measuring or recording the variation in current in the high-frequency circuit.

A. B. MANNING.

**[Cell vessels for] electrolytic cells.** ELEKTRIZITÄTS A.-G., FORM. SCHUCKERT & Co., and A. FISCHER (B.P. 350,639, 8.4.30. Ger., 11.4.29).

**[Synthetic resin-covered carrying frames for] electrolytic processes.** BIRMINGHAM SMALL ARMS CO., LTD., and S. C. WILSDON (B.P. 350,072, 18.3.30).

**[Holding devices for] electrolytic etching apparatus.** E. G. HARTEL (B.P. 350,658, 17.4.30).

**Photoelectric cells.** L. D. J. A. DUNOYER (B.P. 351,142, 14.4.30. Fr., 13.4.29).

**Luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 344,072, 5.12.29. Addn. to B.P. 343,053).

**[Automatic switch for extinguishing back-arc-ing in] metallic-vapour rectifiers.** A.-G. BROWN, BOVERI & Co. (B.P. 350,005, 3.3.30. Ger., 20.3.29. Addn. to B.P. 279,045).

**Cathodes of vacuum electric tube devices.** E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 350,545, 13.3.30).

**Pure H<sub>2</sub>O<sub>2</sub>. O<sub>3</sub>.**—See VII. Protection of Fe. Magnetic alloys. Welding of wires. Zn. Old accumulator plates. Al. Cr-plating. Control of electroplating.—See X. Rubber compositions. Articles containing rubber.—See XIV. Tobacco etc.—See XX. Blasting cap.—See XXII.

## XII.—FATS; OILS; WAXES.

**Structure viscosimetry of vegetable oils.** Y. NISIZAWA (Kolloid-Z., 1931, 55, 343—347).—Measurements with the overflow viscosimeter show that linseed oil and sunflower oil alone or dissolved in C<sub>6</sub>H<sub>6</sub> or COMe<sub>2</sub> exhibit structure viscosity. The phenomenon is specially marked after the addition of resin. The results point to an isocolloidal nature of the oils.

E. S. HEDGES.

**Polymerisation and isomerisation of tung oil.** H. DECKERT (Farbe u. Lack, 1931, 36, 307—308).—A summary of a paper by R. Semmig (Diss., Techn. Hochschule, Dresden, 1923). The extent of formation of  $\beta$ -elæostearin in tung oil by successive irradiations and filtrations, the variation of time of gelatinisation with temp. of heating of the oil, the decrease of  $\beta$ -elæostearin formation with increase in polymerisation, and the relation between these factors and "frosting" of films etc. are discussed.

S. S. WOOLF.

**Technical emulsions of oils and waxes with water.** H. BENNETT (Oil & Fat Ind., 1931, 8, 219—221).—Excellent emulsions, e.g., of carnauba wax, are produced by mixing oils or waxes into an aq. solution (8—12%) of NH<sub>4</sub> linoleate. Formulæ and technique of preparation are described.

E. LEWKOWITSCH.

**Oil of sumac.** H. P. TREVITHICK (Oil & Fat Ind., 1931, 8, 220).—Light petroleum extracted 17.54% of a viscous dark oil having:  $d_{15.5}^{25}$  0.9256,  $n_{25}^{25}$  1.4726, acid val. 21.4, I val. (Wijs) 96.1, saponif. val. 183.2, unsaponifiable matter 2.38%. The total fatty acids (92.72%) had titer 23.7°, neutralisation val. 193.2.

E. LEWKOWITSCH.

**Hydrolysis of fats in alkaline alcoholic solution in relation to the determination of unsaponifiable matter.** J. DAVIDSOHN and E. J. BETTER (Z. angew. Chem., 1931, 44, 565—567).—The residue from the light petroleum extract of a fat after saponification with alcoholic KOH solution contained a mixture of Et esters, to the presence of which the frequent irregularities amongst determinations of unsaponifiable matter are ascribed. A refractometric test of the petroleum extract is recommended.

H. F. GILLBE.

**Aq. dispersions.**—See I. Treatment of textiles.—See VI. Examinations with ultra-violet light.—See XI.

See also A., July, 820, Palmitostearoazelain from cacao butter. 822, Separation of fatty acids. Catalytic decomp. of olive oil etc. Tetracosic acid of peanut oil. 861, Acid formation in wool fat. 867, Nutritive values of hardened oils. 881, Vitamins of fish oils.

PATENT.

**Mineral oil sulphonates.**—See II.

**XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.****Reflectance measurements in the paint industry.**

G. F. A. STUTZ (J. Opt. Soc. Amer., 1931, 21, 323—335).—A discussion of current practice in the measurement of colour, and of the report of a Sub-Committee of the American Society for Testing Materials.

C. W. GIBBY.

**Measurement of obliterating power of paints.**

E. TICHONOV (Farben-Ztg., 1931, 36, 1469—1470).—The thickness of layer of a known vol. of paint is adjusted to the crit. val. at which the light from a standard light source just passes through the layer, and the area covered by unit wt. is calc. The apparatus is illustrated by photograph and diagram.

S. S. WOOLF.

**Paint resistant to flue gases.**

W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1931, 36, 1690—1691).—Experiments are described in which the interval between the time of application of the priming ( $Pb_3O_4$ ) and finishing coats and the time allowed for the system to harden before submission to chimney tests was increased in comparison with the author's previous tests (cf. B., 1929, 254). Paint based on tung oil stand oil again proved inferior when applied over red-lead primer to that when applied on bare metal, and ageing achieved improved fume-resistance in the latter case, but not in the former. Red-lead paint alone is readily broken down and the problem is largely one of vehicle.

S. S. WOOLF.

**Durable red-lead paint.** J. SCHEIBER (Farbe u. Lack, 1931, 36, 303—304).—The use of solutions of "Supraresen," a proprietary resinous material derived from dammar, as vehicle for red-lead paints achieves improved storage properties without decreased durability in comparison with the normal  $Pb_3O_4$ -linseed oil paints.

S. S. WOOLF.

**Pigment and oil.** E. KLUMPP (Kolloid-Z., 1931, 55, 348—351).—A discussion of the relations of pigment and oil in paints leads to the conclusion that the amount of oil required depends on the shape of the pigment particles rather than on their surface or size. For spherical particles the oil required is 66% of the vol. of pigment particles, but angular particles require more oil.

E. S. HEDGES.

**Poisonous action of new lead pigments.** M. RAGG and A. RIEDEMANN (Farben-Ztg., 1931, 36, 1647—1649).—The solubilities of white lead, and red leads of normal and "disperse" type in pepsin hydrochloride and in blood-serum are greater than those of the new "grey-red lead" pigments, e.g., "Arcanol," "Subox." These various Pb pigments when administered to mice showed that Subox and Arcanol were  $\frac{1}{3}$ — $\frac{1}{2}$  as poisonous as red and white leads.

S. S. WOOLF.

**[German] specification for titanium-white.** ANON. (Farben-Ztg., 1931, 36, 1464—1465).—Four grades of titanium-white are detailed, that "for paint use" containing at least 18%  $TiO_2$  and 25%  $ZnO$ , whilst three industrial grades contain 96—100%, 50%, and 25%  $TiO_2$ , respectively, the remainder being  $BaSO_4$  in every case, with the exception of a max. allowance of 3% of total  $CaO$ ,  $Al_2O_3$ , and  $SiO_2$ . An oil paste of the paint-type pigment ground in refined bleached linseed oil is also defined. Fineness, sampling, and packing are

specified and details of analysis of the pigment and oil paste are given.

S. S. WOOLF.

**"Blooming" of oil varnishes.** A. W. C. HARRISON and E. FONROBERT (Farben-Ztg., 1931, 36, 1467—1469, 1512—1514, 1554—1556, 1601—1603, 1645—1646).—"Bloom" is considered as a surface effect to be clearly distinguished from such causes of loss of gloss as surface distortion, water deposition, opalescence, incipient disintegration, etc. The possible factors in varnish making that cause or support bloom are driers, free resin or fatty acids, thinners (i.e., normal constituents), and badly refined or cooked oil, badly run gum, free glycerin, neutral oils, high-boiling solvent or diluent residues (i.e., abnormal constituents). The influences of film thickness, priming and undercoats, atm. conditions, etc. are discussed. Typical formulations and making instructions for non-blooming varnishes are quoted.

S. S. WOOLF.

**Light sensitivity of rosin paper-sizing materials.**

A. E. KIMBERLY and J. F. G. HICKS (Bur. Stand. J. Res., 1931, 6, 819—827).—Rosin darkens on exposure to the light from a C arc, both bleached and unbleached rosin behaving similarly. Ferrous rosinate is also sensitive to light, probably owing to photochemical oxidation to the ferric state. A mixture of rosin with an amount of ferrous rosinate even less than is likely to occur in a rosin size is more sensitive than rosin alone, an observation which probably has a bearing on the yellowing of papers on exposure to light.

R. CUTHILL.

**Resins. XI. Donath's reaction.** E. STOCK (Farben-Ztg., 1931, 36, 1473).—The Donath rosin reaction is given by ester gum (cf. B., 1931, 553) to an extent dependent on the acid val. of the latter; thus a pale yellow coloration only was given with an ester gum of acid val. < 5.

S. S. WOOLF.

**Philippine turpentine from *Pinus insularis*, Endlicher.** I. DE SANTOS and A. P. WEST (Philippine J. Sci., 1931, 45, 233—238).—Benguet pines gave variable amounts of resin, yielding 85—83 wt.-% of rosin and 15—17% of colourless turpentine which differed considerably in composition. The pinene content was high, but the physical properties of the pinene fraction from different trees varied appreciably.

E. LEWKOWITSCH.

**Oxidation of turpentine oils.** J. TERPOUGOV (Bull. Inst. Pin, 1931, 103—108; cf. B., 1929, 218).—Further examination of Portuguese turpentine after long keeping confirms the previous results. In presence of air resins are formed, the oil-sol. resins decomposing into formic, acetic, and butyric acids. The acidity of a Swedish turpentine from dead wood rose in 3 yrs. from 0.49 to 45.05; distillation of the oil gave an aq. and an oily layer, both containing formic and acetic acids in quantity much greater than corresponded with the higher acid val. French, Greek, and Siamese turpentine oils showed a similar production of acid on keeping.

C. HOLLINS.

**Protecting Fe.**—See X. **Tung oil.**—See XII.

See also A., July, 846; **Orange skin pigment.** 847, **Red and yellow paint pigments.** Prep. of Pb rosinate. **Euphorbium resin.**

## PATENTS.

**Impregnating and coating compositions.** IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, and H. M. BUNBURY (B.P. 349,903, 27.12.29).—Aq. emulsions of "glyptal" type resins, initially dissolved in org. solvents if desired, and rubber or substitutes therefor, *e.g.*, "Gloria" aq. rubber-substitute emulsion, together with an emulsifying agent (a sulphonated oil) and colloidal clay (bentonite), giving matte or semi-matte finishes, are claimed. S. S. WOOLF.

**Manufacture of compounds of manganese and rosin.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF P. S. MILLER (B.P. 350,735, 20.6.30. U.S., 20.6.29).—Rosin, melted at 250°, is heated in contact with dry  $\text{MnO}_2$  up to 315°; the resulting resinates contains at least 4.5 wt.-% Mn and is sol. in mineral oil up to 20 wt.-% of solution. Palmitic acid aids the dissolution of the resinates. H. ROYAL-DAWSON.

**Emulsions of resins of the polyhydric alcohol-polybasic acid type, and application thereof.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. BAIRD (B.P. 349,988, 27.12.29).—A solution of a "glyptal"-type resin in an org. solvent is emulsified with  $\text{H}_2\text{O}$ , with or without the addition of an emulsifying agent and/or a protective colloid, *e.g.*, bentonite, soaps, gelatin. A flattening agent, *e.g.*, Al stearate, which may also act as an emulsifier, is added if desired, whilst colouring matter, plasticisers, driers, and an acid catalyst may also be incorporated. S. S. WOOLF.

**Composition of matter [from hardenable phenol-aldehyde resin].** BAKELITE GES.M.B.H. (B.P. 349,931, 25.2.30. Ger., 25.2.29).—A potentially active phenol-aldehyde resin in the resol (fusible, sol.) stage is mixed with oxidised (*e.g.*, blown) drying oils in a suitable mutual solvent, yielding varnishes etc. which dry to clear resistant products without any separation of the oil and resin. E. LEWKOWITSCH.

**Manufacture of nitrocellulose [plastic] products.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. RENFREW (B.P. 349,984, 28.11.29).—Nitrocellulose (35 pts.) of viscosity 1—100 c.g.s. units in a solution of 20 g. in 100 c.c. of 95%  $\text{CO}_2$  at 20°, having been prepared in a mechanical dipper plant by nitrating cellulose in at least 40 times its wt. of mixed acid until the product contains 11.5—12.5% N, is gelatinised with a non-volatile gelatiniser (50 pts.), *e.g.*, tolyl phosphate. The nitrocellulose may be pretreated in known manner to reduce the viscosity to the above range. Floor coverings are obtained by incorporating fillers ( $1\frac{1}{2}$ —2 $\frac{1}{2}$  pts.) with the above plastic (1 pt.). S. S. WOOLF.

**Plastic material.** N. M. MNOKIN (B.P. 350,456, 10.3.30. U.S., 6.5.29).—The plastic product from the chemical interaction of olefine-halogen additive compounds, *e.g.*,  $\text{C}_2\text{H}_4\text{Cl}_2$ , and a sol. polysulphide, *e.g.*, of Na or Ca, particularly the soft type containing 74—85% S, is stabilised against loss of flexibility and development of brittleness by heating, *e.g.*, at 138° for 1 hr. (Cf. B.P. 302,270; B., 1929, 826.) D. F. TWISS.

**Thermoplastic cements.** BAKELITE CORP., ASSEES. OF M. E. DELANEY (B.P. 349,981, 5.3.30. U.S., 6.3.29).—Natural resins, ester gum, etc. (70 pts.) are melted with

a solid halogenated polycyclic hydrocarbon, *e.g.*, trichloronaphthalene (30 pts.); 5% of rubber latex and plasticisers may be added as desired.

E. LEWKOWITSCH.

**Aq. dispersions.**—See I.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Chemistry of [rubber] latex.** III. P. SCHOLZ and K. KLOTZ (Kautschuk, 1931, 7, 110—115; cf. B., 1931, 554).—The relative magnitude of the figures for "total solids" and crêpe-rubber yield of latex is of important physiological significance. With latex obtained by tapping on alternate days, the difference between the two figures, *i.e.*, the proportion of non-rubber constituents, is approx. 2.9%, but it tends to be a little lower for latex containing more than 43% of rubber and a little higher for latex below 37% rubber. For latex from *Ficus*, gutta-percha, and *Euphorbia*, as well as from *Hevea* fruits, the difference is between 6% and 8%. For *Hevea* trees at their first tapping or at the opening of a new tapping cut, the proportion of non-rubber constituents in the latex is about 3.5%; latex from over-tapped trees gives a figure below the normal. During the wintering period of the trees the proportion of non-rubber constituents in latex is much affected, whilst the rubber content is substantially unaltered.

D. F. TWISS.

**Crystallised caoutchouc.** R. PUMMERER and G. VON SUSICH (Kautschuk, 1931, 7, 117—119).—Spontaneous crystallisation occurs more readily in crêpe rubber than in smoked sheet and many samples of the former under X-ray examination reveal a cryst. structure without being stretched. The X-ray diagram of the cryst. rubber obtained by Pummerer and Koch by purification and separation from solution is closely similar to that of the  $\alpha$ -modification of gutta-percha, and it is believed that the so-called cryst. rubber was identical with gutta-percha. The origin of this gutta-percha in the rubber is not certain, but the formation from the rubber is unlikely. It is improbable that rubber has ever been caused to separate from solution in a cryst. condition; cryst. caoutchouc occurs only in stretched and in "frozen" rubber. D. F. TWISS.

**Piperidine accelerators [of vulcanisation of rubber].** P. SCHIDROWITZ and M. W. PHILPOTT (India-rubber J., 1931, 82, 25—30).—Piperidine piperidinocarbothionolate is probably the most active ultra-accelerator known. Thus using 1% in a mixture containing also rubber 100, ZnO 2.5, and stearic acid 0.5 lb., good results are obtained with as little as 0.75% of S; alternatively, with 2% S, excellent results are obtained with 0.35% of accelerator. Zinc piperidinocarbothionolate, unlike the piperidine salt, is insol. in  $\text{H}_2\text{O}$ ; it is slightly less active, but again there is no great advantage in increasing the proportion of S beyond 2%. Lead piperidinocarbothionolate approaches the Zn salt in activity, and, indeed, if judged by equimol. proportions, is distinctly the more active; it shows, however, marked resistance to scorching and prevulcanisation. It is further activated by  $\text{PbO}$ . D. F. TWISS.

**Identifying rubber quantitatively in compositions of rubber and asphalt.** New "ebonite method."

F. C. VAN HEURN and M. A. BEGHEYN (India-rubber J., 1931, 81, 847—851).—Rubber reacts rapidly with S at 150° to give vulcanite, whereas asphalt and S begin to react appreciably at about 175°. For the analysis of asphalt-rubber mixtures about 2½ g. are extracted with xylene for about 8 hr. The extract after evaporation to dryness and weighing is heated with twice its wt. of S in a pyrex tube for 4—5 hr. at 160° (or lower if H<sub>2</sub>S is developed) with stirring. After extracting this product in an alundum thimble with xylene the insol. residue of vulcanite is dried at 150°. The S content of the impure vulcanite is then determined by direct analysis. The rubber content of the original material is then 0.02125*ab*, where *a* is the % of impure vulcanite obtained and *b* its content of S. If the proportion of rubber in the material is large, it is advisable to reduce it by adding asphalt before proceeding with the analysis. D. F. TWISS.

## PATENTS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., E. A. MURPHY, A. NIVEN, and D. F. TWISS (B.P. 350,450, 10.3.30).—Articles to be provided with a matte or granular surface finish are coated, *e.g.*, by spraying or dipping, with a granular dispersion obtained by effecting coagulation of an aq. rubber dispersion such as latex by precipitation therein of one or more compounding ingredients, *e.g.*, Mg silicate or carbonate, by double decomp. For this purpose one of the sol. reagents, *e.g.*, Na<sub>2</sub>CO<sub>3</sub> or Na silicate, normally has no coagulating effect, whilst the other, *e.g.*, sulphate of Mg or Al, may incidentally possess coagulating influence. D. F. TWISS.

**Manufacture of articles containing rubber or similar material.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., D. F. TWISS, and R. G. JAMES (B.P. 350,106, 31.3.30).—Semi-permeable material such as fabric is coated with rubber by contracting one side with a normally alkaline aq. dispersion of electronegative rubber and the other with an acid aq. dispersion in which the particles have assumed a positive electric charge. The mutual coagulation of the two dispersions at the surface of the separating material may be aided by electrophoresis with the anode and cathode in the acidic and alkaline dispersions, respectively. D. F. TWISS.

**Manufacture of rubber articles.** SOC. ITAL. PIRELLI (B.P. 350,227, 2.6.30. Italy, 7.6.29).—The article to be manufactured is formed on a core of a water-dispersible inorg. colloid such as a smectic clay or bentonite, which may be covered with an adherent protective coating, *e.g.*, of paper or thin linen. The core is eventually removed after disintegration with the aid of H<sub>2</sub>O. D. F. TWISS.

**Manufacture of rubber compositions.** J. C. PATRICK (B.P. 350,472, 5.3.30).—The plastic product from an olefine dihalide and a metal polysulphide, preferably the soft type of product (*cf.* B.P. 350,456; B., 1931, 769), is mixed with rubber and heated without the addition of free S. The vulcanised products so obtained contain no free S and are of value for electrical insulating purposes. D. F. TWISS.

**Manufacture of vulcanisable compounds.** J. C. PATRICK and N. M. MNOOKIN (B.P. 350,549, 13.3.30).—

A mixture of rubber or plastic olefine polysulphide (*cf.* preceding abstract) and a proportion of S in excess of that normally required for vulcanisation of the rubber in the mixture is vulcanised by heat. The products, which may contain 50% or more total S, resemble soft vulcanised rubber in mechanical properties and do not exhibit "blooming." D. F. TWISS.

**Manufacture of cellular compositions [from rubber].** H. L. FISHER, Assr. to B. F. GOODRICH CO. (U.S.P. 1,786,563, 30.12.30. Appl., 28.12.26).—A porous or cellular product is obtained by treating rubber at a raised temp., *e.g.*, at 150°, with an isomerising agent such as H<sub>2</sub>SO<sub>4</sub> or an org. sulphonic acid or sulphonyl chloride. Blowing agents may additionally be employed if products are required of very low apparent sp. gr. D. F. TWISS.

**Preservation of natural varieties of rubber and artificial rubber-like masses.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 350,563, 14.3.30).—The OH-derivatives of diphenyl and of its substitution products have a marked preservative influence on rubber and do not cause discoloration even on exposure to light. D. F. TWISS.

**Reclaimed rubber.** C. H. CAMPBELL, Assr. to PETER COOPER CORPS. (U.S.P. 1,786,149, 23.12.30. Appl., 9.8.29).—Rubber scrap is reclaimed by subjecting it to a hydrolytic devulcanising process after mixing it with tanned leather. The cleavage products from the tanned leather assist the softening of the harder particles of the scrap. D. F. TWISS.

**Reclaimed rubber.** L. MELLERSH-JACKSON. From AMER. GLUE CO. (B.P. 350,269, 27.6.30).—Reclaimed rubber, produced by a process involving hydrolytic action, is improved in plasticity by the addition of 2—5% of cleavage products of tanned leather or tanned leather itself, before the devulcanising operation. D. F. TWISS.

**Manufacture of vehicle tyres comprising artificial rubber.** I. G. FARBENIND. A.-G. (B.P. 350,490, 12.2.30. Ger., 12.2.29).—Tyres are manufactured from products obtained by polymerisation of mixtures of  $\beta$ -dimethylbutadiene with butadiene and/or isoprene, to which a finely-divided form of soot is added before vulcanisation. D. F. TWISS.

**Rubberising of fibrous material.** E. HOPKINSON (U.S.P. 1,784,523, 9.12.30. Appl., 21.6.24).—An aq. dispersion of rubber such as latex is rendered more viscous, *e.g.*, by concentrating or by adding thickening agents, and, after being compounded, is used for the manufacture of rubberised fabric by application to a normally penetrable fabric, possibly in a single coating operation, without substantial penetration. The latex can conveniently be of 50% concentration or higher. The finally vulcanised textile material retains a high proportion of its original flexibility. D. F. TWISS.

**Rubberised sheet material.** I. H. ROBINSON (B.P. 350,589, 18.3.30).—Waterproof sheet material suitable for shoe insoles or for rainproof coverings is made by mixing a granular substance such as cork, asbestos, wood powder, or leather buffings with rubber latex, preferably with the temp. raised gradually up to 80° and then drying. The material is then rolled into sheet

and sandwiched between two layers of fabric, such as linen, one at least of which has been previously rubberised.

D. F. TWISS.

**Production of rubber substitute.** M. BANDLI (B.P. 349,901, 20.12.29).—A precipitant liquid ( $H_2O$ ) is added to a solution of cellulose nitrate in a mixture of solvents of which at least one is readily miscible with the precipitant and at least one other is not readily miscible. The solution contains also a plasticiser (which may itself be one of the essential solvents, *e.g.*, tolyl phosphate) in a dissolved or suspended condition, which is precipitated with the cellulose nitrate.

D. F. TWISS.

**Reclaiming or improving of gum exudates [*e.g.*, gutta-percha].** C. MARTELL, Asst. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,786,075, 23.12.30. Appl., 6.5.25).—Inferior gum exudate, such as gutta-percha, is masticated with rubber latex; the resulting removal of foreign matter and absorption of rubber from the latex cause the gutta to emerge with all the characteristics of a good grade.

D. F. TWISS.

**Manufacture of rubber-like compositions.** F. C. ZOBEL (U.S.P. 1,786,281, 23.12.30. Appl., 23.1.26).—Gum copal is heated with hard-wood creosote oil and the solution is poured into petroleum naphtha. The resulting amorphous ppt. is kneaded and washed with  $H_2O$  and yields a rubber-like mass, which is sol. in the usual rubber solvents and can be used for erasing pencil marks.

D. F. TWISS.

**Vulcanising and treating of rubberised materials or the like and apparatus therefor.** PIONEER VENTILATING & DRYING Co., LTD., J. H. WILD, and J. LLOYD (B.P. 350,648, 11.4.30).

**Rubber tubing.** GAS LIGHT & COKE Co., J. G. CLARK, and C. A. MASTERMAN (B.P. 350,385, 3.3.30).

**Impermeable cloth.**—See VI. Coating compositions.—See XIII.

## XV.—LEATHER; GLUE.

**Sex differences of hide powder.** II. T. TADOKORO and K. YOSHIMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 175 B; cf. B., 1931, 504).—Hide powder of female animals differs from that of male animals in that (a) it is more easily digested by trypsin, (b) the I absorption of the alkali-sol. portion is smaller, (c) the alkaline solution of both crude and refined varieties has a lower absorptive power for light of wave-length  $> 2740 \text{ \AA}$ , (d) the glutin has greater rotatory power in alkaline solution, (e) the glutin solution attains a max. viscosity after a shorter time of boiling, (f) the glutin has a higher Ac val. and its  $NI_2$ -acids contain more diamino- and lysine-N, but less histidine-N.

E. H. SHARPLES.

**Analyses of salt used for curing hides and skins.** R. C. BOWKER and J. BEEK, JUN. (J. Amer. Leather Chem. Assoc., 1931, 26, 312—317).—Complete analyses of various mined and evaporated salts are tabulated.

D. WOODROFFE.

**Dermatophytes as the cause of leather damage.** M. BERGMANN, W. HAUSAM, and E. LIEBSCHER (Collegium, 1931, 248—254).—A description, with photographs, of the damage caused by dermatophytes, the

spores of which were found in the grain of half-tanned calfskins.

D. WOODROFFE.

**Determination of moisture in vegetable-tanned leather.** D. BURTON (J. Soc. Leather Trades' Chem., 1931, 15, 273—281).—The difficulties of obtaining concordant moisture figures for leather are discussed. Loss of wt. was observed after heating some leathers for 200 hr. An increase in wt. of 0.3% was observed after heating several samples of leathers for 12—16 hr., followed by a decrease on prolonged heating. This increase was smallest in leathers low in  $H_2O$ -sol. matter. The continuous loss in wt. on heating leather was accentuated by the presence of mineral oil in the leather. About 0.3% more moisture was shown by use of a vacuum oven than when an air oven was used for drying purposes. Immediately the oven is opened the lid or stopper should be placed in the weighing bottle, which should be immediately transferred to the desiccator and weighed after 20 min.  $H_2SO_4$  should be used in the desiccator. A vac. should be maintained in the oven throughout the heating period.

D. WOODROFFE.

**Analysis of vegetable-tanned leather.** [Report of a Committee of the French Section of the Society of Leather Trades' Chemists.] G. PARSY (J. Soc. Leather Trades' Chem., 1931, 15, 251—256).—In determining moisture, the leather should be heated for 11 hr. at  $103^\circ$ , or for 8 hr. at  $108^\circ$ , or for 5—8 hr. at  $113^\circ$ . Some volatile matter is lost by greasy leathers at  $103^\circ$ . The fat should be determined by extraction with  $CCl_4$  or  $C_6H_6$  and the fat extract should be heated for 7—8 hr. at  $108^\circ$  before weighing. Greasy leathers should be degreased before the moisture determination.

D. WOODROFFE.

**Determination of moisture in vegetable-tanned leather.** P. CHAMBARD (J. Soc. Leather Trades' Chem., 1931, 15, 258—261).—Satisfactory results have been obtained on vegetable-tanned sole leather if the sample was planed, heated for 5 hr. in an oven at  $110^\circ$ , cooled, weighed, heated for a further 2 hr. at  $110^\circ$ , and reweighed, and the result calc. therefrom, provided the difference between the weighings did not exceed 15 mg.

D. WOODROFFE.

**Average moisture content of vegetable-tanned heavy leathers.** F. STATNER (Collegium, 1931, 254—256).—The moisture in vegetable-tanned heavy leathers is not affected by the amount of  $H_2O$ -sol. matter, but is influenced by the fat content. The average fat and moisture contents of various leathers were, respectively, (%):  $< 10.0$ , 12.6; 10—20, 12.4;  $> 20$ , 10.9. The moisture content was not a direct function of the fat content, but depended on the method which had been used to incorporate the fat with the leather.

D. WOODROFFE.

**Report of a Committee [of the French Section of the Society of Leather Trades' Chemists] on the analysis of bating materials.** A. BODIN (J. Soc. Leather Trades' Chem., 1931, 15, 257—258).—Bating materials should be evaluated by the amount of a standard protein, *e.g.*, casein, fibrin, or edestin, hydrolysed by them. The Fuld-Gros, Kubelka and Wagner, Schneider and Ulcek, "Pouvoir Présure," Film, and Bergmann methods are suggested.

D. WOODROFFE.

**Lesser-known tropical tanning materials.** F. STATHER (Collegium, 1931, 256—262).—The tannin, non-tans, insol. matter, and  $H_2O$  contents of the following materials are given (%): Angico wood (*Viptadenia rigida*, Benth.) 4.5—10.9, 1.7—2.3, 75.3, 14.5; Angico bark 16.0—22.0, 5.9—9.5, 59.0, 14.5; Aroeiro bark (*Schinus areira*, L. [*S. molle*, L.]) 23.0, 8.8, 53.6, 4.6; Acajou bark 21.5, 10.5, 55.0, 13.0; Capororoca oak (*Rapanea guyanensis*, Aubl., and *R. umbellata*, Mez.) 8.75, 2.9, 73.9, 14.5; copaiba bark (*Copaiba Langsdorfii*, Desf.) 14.5, 2.0, 69.0, 14.5; Jatahy bark (*Hymenaea courbaril*, L.) 19.2, 5.75, 60.0, 14.5; inhacaperana bark 19.75, 1.75, 64.0, 14.5; mangue bark (*Laguncularia racemosa*, Gaertneri, or *Conocarpus racemosus*, Linn.) 17.0—29.9, 6.0—13.5, 43.0—62.0, 14.5; murta bark (*Eugenia lucida*, Camb.) 15.9, 8.2, 61.4, 14.5; cassia bark (*Cassia fistula*, Linn.) 18.2, 8.6, 58.7, 14.5. Cassia is found in all tropical regions; the other barks are derived from South America. All are pyrocatechol tans.

D. WOODROFFE.

**Sulphiting of quebracho [tannin].** M. BERGMANN and G. POJARLIEFF (Collegium, 1931, 239—243).—When quebracho tannin is sulphited, equal parts of two compounds are formed, from one only of which the sulphurous acid can easily be split off. From the known constitution of quebracho tannin it is evident that there is a fundamental change in its nature during sulphiting. The resorcinol part of its mol. loses its phenolic character and is converted into a hydrogenated aromatic alcohol with properties different from those of the original unsulphited phenol. Sulphiting is mainly a chemical process followed by colloid-chemical changes, thus distinguishing it from mere solubilising which reduces the particle size.

D. WOODROFFE.

**Chemico-technical properties of Rumanian spruce bark as a tanning material.** C. OTIN and G. HUIDOVICI (J. Soc. Leather Trades' Chem., 1931, 15, 264—273).—Analyses of 40 samples of Rumanian spruce bark (*Abies excelsa*) showed (%): tannin 5.26—17.58 (aver. 11.7); sol. non-tans 3.89—18.38 (aver. 9.3). The tannin content is fairly uniform throughout the whole length of the trunk. A satisfactory bark is obtained from those trees which yield the best timber. Large quantities are available in Rumania, and it compares favourably with that from other parts of Europe.

D. WOODROFFE.

**Filtration of tannin solutions for the determination of insoluble [matter].** Report of Committee of the American Leather Chemists' Association. J. S. ROGERS (J. Amer. Leather Chem. Assoc., 1931, 26, 272—297).—Slightly higher amounts of insol. matter were obtained at 18° (International method) than at 20° (Official American). A turbid filtrate was obtained when quebracho extracts were filtered by these two methods, but clear filtrates were given by all extracts filtered by the Riess method. Too rapid filtration was shown by the latter method on valex extract, slow filtration by chestnut and quebracho extracts, and very slow filtration by hemlock and pine bark. Good agreement can, however, be obtained by this method, which is preferable to the International and American methods in principle, since the kaolin mat is of even

thickness. Variations in results are often caused by the use of different papers and by pipetting solutions at different temp. Slightly higher total solids were obtained by evaporating 50 c.c. of solution instead of 100 c.c.

D. WOODROFFE.

**Photographic gelatin.**—See XXI.

See also A., July, 812, **Measurement of OH and SH in  $Na_2S$  solutions.**

PATENTS.

**Unhairing of hides or skins.** G. D. McLAUGHLIN, G. E. ROCKWELL, F. O'FLAHERTY, and J. H. HIGBERGER, Assrs. to TANNERS COUNCIL OF U.S.A. (U.S.P. 1,785,092, 16.12.30. Appl., 15.3.27).—Hides or skins are treated with a saturated  $Ca(OH)_2$  liquor to which has been added about 1% of a primary, secondary, or *tert.* amine, or an amide, or mixtures of these for 2—3 days.

D. WOODROFFE.

**Tanning of chamois, buckskins, or other leather.** A. ERNST (U.S.P. 1,784,828, 16.12.30. Appl., 27.3.30).—The suitably prepared pelts are drummed with a solution of 1—1.5%  $CH_2O$  and 0.5—0.75% MeOH for 45 min., after which 0.5% of soap, 2.25% of moellon degreas, 2.25% of cod oil, 2% of Glauber's salt, and 2.75% of  $NaHCO_3$  are added and drumming is continued until tannage is complete.  $MgSO_4$  is then added to the mixture and the skins are again drummed, washed, and dried.

D. WOODROFFE.

**Treatment of tanning solutions.** A. G. WACKENREUTER (U.S.P. 1,786,880, 30.12.30. Appl., 30.4.28).—Tanning liquors in which hides have been suspended for some time are centrifuged to remove moulds, solids, and harmful fungi, and the separated liquor is conc. for further use.

D. WOODROFFE.

**Manufacture of a vegetable adhesive.** I. F. LAUCKS and G. DAVIDSON, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,786,209, 23.12.30. Appl., 31.8.28. Cf. U.S.P. 1,689,732; B., 1929, 141).—A vegetable meal containing protein, e.g., soya bean, linseed, or cottonseed flour, is incorporated with an alkaline medium [e.g.,  $NaOH$ ,  $Ca(OH)_2$ ] and  $ZnSO_4$  (0.1 pt./30 pts. of meal); other substances, e.g.,  $NaCl$ ,  $CaCl_2$ ,  $Na_2Cr_2O_7$ , may also be added.

L. A. COLES.

**Impregnated articles.**—See V. **Reclaimed rubber.** **Reclaimed leather.** **Rubberised sheet.**—See XIV.

## XVI.—AGRICULTURE.

**Soils of the Nile and Gash.** I. P. VAGELER and F. ALTEN (Z. Pflanz. Düng., 1931, 21A, 47—57).—Discussion of appropriate methods of examining these soils.

A. G. POLLARD.

**Soils of the Havelland plains.** B. TACKE (Mitt. Ver. z. Förd. Moork. i D.R., 1931, Nos. 1, 2, 3—7, 25—29; Bied. Zentr., 1931, 60A, 151).—The response of alkaline low-moor soils to various types of manurial treatment is recorded. Physiologically neutral or acid fertilisers are preferable. Bone meal is non-effective in these soils. Applications of  $MnSO_4$  counteracted, to some extent, the ill effects of soil alkalinity. The conjoint use of milling waste and S produced good results.

A. G. POLLARD.

**Comparison of methods for determining the volume-weight of soils.** A. S. CURRY (J. Agric. Res.,



1931, 42, 765—772).—A cylinder method is described. Existing methods proved less satisfactory for the soils examined.

A. G. POLLARD.

**Catalytic power of soils.** I. F. RADU (Landw. Versuchs-Stat., 1931, 112, 45—54).—The catalytic power bears no relationship to the  $p_H$  or exchange acidity (cf. B., 1929, 182), but increases with the content and degree of dispersion of colloidal clay and humus. Correlation between catalytic power, nutrient content, and bacterial numbers is examined.

A. G. POLLARD.

**Dynamics of the absorbing complex of soils.** M. A. VINOKUROV (Pochvoved., 1928, 23, No. 3—4, 46—92).—In a chernozem soil the Ca was higher in the autumn and lower in the spring; the reverse held for the Mg. The total bases were lower in the spring. During the first part of the growing season the total exchange capacity ( $BaCl_2$  method) decreased slightly, whilst the Ca increased. In some cases the total Ca and Mg absorbed was greater than the base-exchange capacity. Increase in the absorption capacity during summer and winter is attributed to change in  $p_H$  and possibly to mechanical absorption. The quantity of highly dispersed particles of the absorption complex falls from the beginning of spring until the middle of summer.

CHEMICAL ABSTRACTS.

**Determination of adsorption capacity of the mineral and organic portions of the adsorption complex of soil.** B. MALÁČ (Věstn. Českoslov. Zem., 1930, 6, 1063—1065; Chem. Zentr., 1931, i, 1964—1965).—The soil was treated with  $BaCl_2$  followed by 0.1N- $HNO_3$  until no further reaction was obtained, the  $Ba^{++}$  and  $Cl^-$  in the filtrate being determined. By pretreatment of the soil with a cation ( $NH_4^+$ ,  $K^+$ ,  $Na^+$ ) of which the humate is sol. in pyridine, followed by extraction of the complex with pyridine, only the mineral portion remained.

A. A. ELDRIDGE.

**Determination of soil organic matter.** C. T. SCHOLLENBERGER (Soil Sci., 1931, 31, 483—486).—The author's original method (B., 1927, 662) is modified by the addition of  $H_3PO_4$  or HF to the dil. solution prior to titration with  $Fe(NH_4)_2(SO_4)_2$  in the presence of  $NHPh_2$ . The end-point is sharp. A blank determination should be made.

A. G. POLLARD.

**Base exchange in soils rich in organic matter.** L. SMOLIK (Věstn. Českoslov. Akad. Zem., 1930, 6, 912—915; Chem. Zentr., 1931, i, 1963).—The base-exchange capacity is greater in soils rich in C than in those poor in humus. No connexion with N content was observed.

A. A. ELDRIDGE.

**Carbon : nitrogen ratio in relation to the accumulation of organic matter in soil.** F. J. SALTER (Soil Sci., 1931, 31, 413—430).—The micro-org. decomposition of plant material in soil depends primarily on the C : N ratio, there being a general tendency for the latter to stabilise at 10 : 1. With wider ratios losses of C occur, and with narrower ratios there is an accumulation. Nitrate formation is rapid where the C : N ratio is narrow, but is depressed for several months where the ratio is wide. The N fixed as a result of energy supplies carried by wide-ratio materials does not become available until losses of C have reduced the C : N ratio to 10 : 1. Fixation of N is favoured by a

C : N ratio of 15 : 1, but is not appreciably increased by applications of phosphates. Addition of N to soils is necessary in order to increase the accumulation of C.

A. G. POLLARD.

**Relations between the hydrolytic acidity of soils and the degree of saturation with lime.** A. GEHRING and O. WEHRMANN (Z. Pflanz. Düng., 1931, 21A, 1—32).—The CaO requirement of soil as determined by the authors' method (B., 1930, 256) is that requisite to ensure a sufficiency of replaceable Ca to maintain a good physical condition in addition to removing acidity. Methods based on measurements of hydrolytic acidity are not concerned with the nature of the active soil bases. In determining the degree of saturation of soils with Ca, due proportionality between the wt. of sample and vol. of  $Ca(OH)_2$  solution used and vol. of filtrate collected must be maintained. The hydrolytic acidity as determined by the authors' method is in close agreement with the degree of saturation with Ca if the value " $y_1$ " be multiplied by the factor 4. Relationships between this value and Kappen's measurements of hydrolytic acidity are discussed.

A. G. POLLARD.

**Replaceable cations in the soil and the plant.**

K. K. GEDROIZ (Udobr. Urozhai, 1930, 2, 464—475).—When a chernozem soil was saturated with Mg, Ba, Mn, Co, Ni, Cu, or H ions, no crop (mustard) was obtained with or without a complete fertiliser; when  $CaCO_3$  was added, no crop (oats) was obtained with Ba, Ni, or Co, small crops with Cu, Mg, or Mn, and a normal crop with H. In another series (Mg, Ca, Sr, Cd,  $Fe^{++}$ ,  $Fe^{+++}$ , Al, H), Cd and  $Fe^{++}$  gave zero, and Mg, Al, and H a small yield in absence of  $CaCO_3$ ; Sr gave a normal crop. With  $CaCO_3$  present Cd gave no,  $Fe^{++}$  and  $Fe^{+++}$  small, and Al and Mg 50% normal, crops.

CHEMICAL ABSTRACTS.

**Phosphorus assimilation by soil micro-organisms.** L. G. THOMPSON, JUN., F. B. SMITH, and P. E. BROWN (Soil Sci., 1931, 31, 431—436).—The  $H_2O$ -sol. P of soils is affected to different extents by various species of soil micro-organisms. Some species render sol. more P than they can assimilate; with others the reverse is the case. *Azotobacter chroococcum* and various species of *Aspergillus* are examined in this respect.

A. G. POLLARD.

**Transformation of *Azotobacter*-nitrogen in soils.**

H. ENGEL (Z. Pflanz. Düng., 1931, 21A, 32—46).—The N of *Azotobacter* cultures, whether alive or dead, was rapidly nitrified on admixture with soil. The results are discussed in relation to decom. of straw and cattle manures in soil (cf. B., 1930, 73).

A. G. POLLARD.

**Chemical methods for determining the availability of soil phosphate.** P. L. HIBBARD (Soil Sci., 1931, 31, 437—466).—Extraction of soils with dil. acids (1 : 5 equilibrium mixtures) offers the simplest and quickest method for determining the availability of soil P, but several extractions with acids of different concentrations yield more satisfactory results. Strict comparisons of different soils should be made with extracts of similar  $p_H$  (e.g., 4.0). Simple extraction methods which indicate conditions at the time of sampling only are inadequate to characterise the supply

of P throughout the plant's growth. The "relative solubility" method (Lemmermann) is more satisfactory from this viewpoint. Methods involving extraction of soils with  $\text{CO}_2$  and  $\text{CaCO}_3$  (Dirks and Scheffer), citric acid (Dyer),  $\text{HNO}_3$  (Fraps),  $\text{NaCl}$  in  $\text{H}_2\text{SO}_4$  (Arrhenius) or  $\text{H}_2\text{O}$  are not generally applicable to soils of all types. Percolation methods are superior to equilibrium extracts in that they resemble more closely the action of the growing plant, and give a general but not accurate interpretation of the ability of soils to maintain a supply of P.

A. G. POLLARD.

**The phosphate question [in soils]. V. Phosphate and nitrate concentrations and plant growth.** O. ARRHENIUS (Z. Pflanz. Düng., 1931, 10B, 289—292).—In sand-culture experiments with oats and barley in which both the N and P contents of the nutrient were varied, insufficient supplies of either nutrient completely nullified the effect of increasing the concentration of the other. With clover (inoculated) additions of P were effective in all proportions used.

A. G. POLLARD.

**Portable field apparatus for the determination of chlorides in soils.** R. J. BEST (J. Coun. Sci. Ind. Res., Australia, 1931, 4, 122—123)—Apparatus described previously (B., 1929, 731) is adapted for field work.

A. G. POLLARD.

**Effect of humic acid on the *Aspergillus* method [for soil-nutrient determination].** L. E. KIESSLING (Z. Pflanz. Düng., 1931, 21A, 86—104).—The Niklas-Poschenrieder-Trischler method for determining the available K in soils by means of *A. niger* gave unduly high values in low-moor soils. Similar results were obtained on mineral soils to which humic preparations ( $\text{H}_2\text{O}$ -sol. or -insol. or K humate) were added. Humic acid not only stimulates mycelium production, but causes modifications in its structure and in conidia formation.

A. G. POLLARD.

**Neubauer method for determining mineral nutrient deficiencies in soils.** S. F. THORNTON (J. Amer. Soc. Agron., 1931, 23, 195—208).—The Neubauer method gives results most closely in accord with the pot and field tests; extraction with 0.2N- $\text{HNO}_3$  and the Illinois phosphate test frequently give high values for  $\text{P}_2\text{O}_5$ . Nutrient absorption by seedlings is greatly affected by selection of seed and temp. control, and to a smaller extent by light intensity, moisture content, soil reaction, and the presence of other nutrients.

CHEMICAL ABSTRACTS.

**Availability of nitrogen in farm manure under field conditions.** A. F. HECK (Soil Sci., 1931, 31, 467—481).—Little, if any, of the  $\text{H}_2\text{O}$ -insol. N of farmyard manure is recovered in the first crop after application. The recovery of the N of liquid manure is greater if applied with dung than if used alone. If ploughed in immediately after spreading, no difference is apparent in the availability of the N of fermented and fresh manure. Nitrification of manure is more rapid when applied as a top dressing and harrowed in than when ploughed in. Losses of N from manure in the interval between spreading and ploughing in are greatest when the manure has become ammonified but not fermented, less after complete fermentation, and least in fresh

manure. Admixture of straw with manure decreases the amount of N recovered in the first crop.

A. G. POLLARD.

**Changes in stall manures during storage and their action in soil.** H. C. VON SEYDEWITZ (Landw. Versuchs-Stat., 1931, 112, 55—102).—Inadequate heating in manure stacks ( $< 60^\circ$ ) results in incompletely rotted straw, greater losses of N and dry matter during fermentation, and a final product of lower dry-matter content. Losses of N from hot-fermented manure stacks did not increase after the 4th month. Drainage from hot-fermenting stacks decreased with the age of the stack and contained much less  $\text{NH}_3$ -N than did ordinary liquid manure. Hot-fermented manure was nitrified more rapidly and more completely than yard manure and produced greater crop increases.

A. G. POLLARD.

**Comparison of various methods of storing manure.** J. P. MAMCHENKOV (Udobr. Urozhai, 1930, 2, 284—291).—Loose piling, compacting, and Krantz' method (compacting at  $55^\circ$ ) were compared, combined,  $\text{NH}_3$ -, and total N being determined. Packing down is preferable, since mineral-N is preserved.

CHEMICAL ABSTRACTS.

**Effect of drying manure on the nitrogen losses and crop yields.** J. P. MAMCHENKOV and J. F. ROMASHKEVICH (Udobr. Urozhai, 1930, 2, 394—397).—Piled manure lost more N than scattered manure; the latter lost N chiefly as  $\text{NH}_3$  in the first 2 days and was slightly inferior to fresh manure with oats.

CHEMICAL ABSTRACTS.

**Availability of nitrogen of green manure for rice and the supplementary value of various fertilisers.** H. MISU and H. SHIMOHIRA (Ann. Agric. Exp. Sta. Gov.-Gen. Chosen, 1929, 4, 65—94).—The availability of N was lower in dried than in fresh green manure. The yield was better with fresh than with fermented green manure.

CHEMICAL ABSTRACTS.

**Effect of phosphate manuring on the yield and quality of malting barley.** C. DREYSPRING, H. KURTH, and F. HEINRICH (Z. Pflanz. Düng., 1931, 10B, 265—289).—In pot cultures with many varieties of barley, applications of easily-sol. P definitely accelerated growth and caused increased tillering and earlier flowering and maturing, the latter being particularly apparent in the "green-ripe" and "yellow-ripe" stages. Phosphate manuring produced greater yield increases in grain than in straw, there being an increase both in the wt. and number of ears. The length of straw was not definitely affected. The protein content of the grain decreased with increasing applications of superphosphate. This effect was greatest in varieties having a high protein content when grown in untreated soil. In no case was the protein content  $< 8.9\%$ . There was a direct relationship between the proportion of high-mol. protein and the P content and stage of maturity of the grain. Phosphate fertilisers did not increase the starch content of the grain. The P content of the grain was directly proportional to the grain yield, the number of tillers and ears, to the wt. per ear, the acceleration of growth, and the increase in grain : straw ratio, but was inversely proportional to the total protein content.

A. G. POLLARD.

**Comparative value of nitrogenous fertilisers on the growth of autumn and spring barley.** I. OMAI (Ann. Agric. Exp. Sta. Gov.-Gen. Chosen, 1930, 3, 347—354).—Of the materials tested,  $\text{NaNO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$  gave the highest recovery of N (60–66%) and the best yield. The efficiency of the N in pig and cow manure is small. CHEMICAL ABSTRACTS.

**Fertiliser experiments with citrus seedlings.** I. TAKAHASHI (J. Okitsu Hort. Soc., 1930, 25, 38—50).—The  $\text{P}_2\text{O}_5$  and K requirements are, respectively, 50 and 66% of the N requirement. CHEMICAL ABSTRACTS.

**Efficiencies of phosphoric acid of various fertilisers for spring- and autumn-sown barley.** I. OMAI (Ann. Agric. Exp. Sta. Gov.-Gen. Chosen, 1930, 3, 394—402).—Application of  $\text{Na}_2\text{HPO}_4$ , bone meal, and superphosphate gave about 190% greater yield and 130–140% more absorbed  $\text{P}_2\text{O}_5$ ; rice polishings and  $\text{AlPO}_4$  gave 150% (yield), 60–70% ( $\text{P}_2\text{O}_5$ ). Autumn-sown barley showed higher absorption and higher yield than spring-sown barley. CHEMICAL ABSTRACTS.

**Organic fertilisers for oats and flax.** Z. V. LOGVINOVA and A. P. SHCHERBAKOV (Udobr. Urozhai, 1930, 2, 476—482).—Various org. sources of N were compared with  $\text{NaNO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$ . With oats intestinal slime gave good results; meat scrap, horn meal or shavings, burned horn, blood meal, feathers and down, and oil meal gave good results when larger quantities were compared. The residual effects on the succeeding crop were better for org. than for inorg. N. With flax, org. N was superior to inorg. N. CHEMICAL ABSTRACTS.

**Ammonification of nitrogenous substances by pure cultures of micro-organisms.** H. C. PULLEY (J. Agric. Res., 1931, 42, 791—800).—Ammonification of the following N compounds in soil by pure cultures of various organisms occurred in the order: asparagine (most rapid), peptone, gluten, urea, casein, gelatin, dried blood, uric acid, egg albumin, hippuric acid. Acetanilide and caffeine depressed ammonification, which was entirely inhibited by  $\text{NiPh}_2$ .  $\text{CaCN}_2$  was not ammonified in the soil examined, nor was any urea formed. In general, amino-N is readily ammonified, but the N groups of purine bases are more resistant. A. G. POLLARD.

**Effect of salt on the microbial heating of alfalfa [lucerne] hay.** L. S. STUART and L. H. JAMES (J. Agric. Res., 1931, 42, 657—664).—Lucerne hay heated most rapidly when its moisture content was 30%. Retardation of heating following the addition of NaCl varied inversely with the moisture present. Small amounts (1–2%) of NaCl tend to increase the growth of moulds and reduce that of bacteria. With larger proportions microbial activity may be delayed sufficiently to permit the curing of the hay. A. G. POLLARD.

**Cotton plant, with special reference to its nitrogen content.** G. M. ARMSTRONG and W. B. ALBERT (J. Agric. Res., 1931, 42, 689—703).—A generous supply of soil-N is associated with a succulent growth and high N contents of leaves, stalk, and fruit. The N supply did not affect the N content of seed and lint from

7 to 21 days old. The N content of all tissues, with the possible exception of seed and fruit, decreased with maturity. Close spacing of plants tended to accelerate N absorption, to produce bolls of higher N content, and to increase the proportion of stem to dry wt. The general intake and distribution of N during growth is recorded. A. G. POLLARD.

**Fertilising citrus trees.** J. J. THERON (Farming in S. Afr., 1931, Reprint No. 29, 5 pp.).—In the area examined, citrus trees responded to applications of  $\text{NaNO}_3$  and phosphates, but K had little effect. Appropriate manurial treatments are specified. A. G. POLLARD.

**Percentage dry matter and field weight of ear maize from unlimed and limed plots.** A. W. BLAIR (J. Agric. Res., 1931, 42, 773—774).—Maize grown on acid soil does not mature and dry out so quickly as when the same soil is adequately limed. Small but not very definite increases in grain yield after liming are recorded. A. G. POLLARD.

**Sugar and catalase determinations of seeds of varying viability.** A. NIETHAMMER (Z. Pflanz. Düng., 1931, 21A, 69—86).—A general parallelism exists between the catalase activity and germinative capacity of many seeds, the effect being more definite among seeds of cultivated crops than of wild plants. Differences are apparent among certain seeds of the same species and strain. Seeds of sufficient age to lose all germinative power usually had no catalase activity. A. G. POLLARD.

**Analysis of seeds.** L. BUSSARD (Ann. Falsif., 1931, 24, 211—220).—Tests are made for identification, degree of purity ( $P$  = % genuine seeds), presence and nature of foreign material, dryness, and germinating faculty ( $G$  = % genuine seeds germinating in given time). Cultural value =  $PG/100$ . Methods of sampling are given, together with data for 58 kinds of seeds of good quality. E. B. HUGHES.

**Progressive changes in the wax-like coating on the surface of the apple during growth and storage.** K. S. MARKLEY and C. E. SANDO (J. Agric. Res., 1931, 42, 705—722).—With advancing growth, the waxy covering of apples shows a general increase in  $\text{Et}_2\text{O}$ -sol., light petroleum-sol (“oily fraction”), and ursolic acid contents. The oily fraction increases more rapidly than the ursolic acid. Changes in the proportions of these constituents are associated with differences in the physical properties of the coating, and its permeability to gases, in the effectiveness of spray applications and the ease of removal of spray residues. The amounts of ursolic acid and  $\text{Et}_2\text{O}$  extract are greater in the shaded than in the lighted side of the apple, but the oily fraction is equally distributed. The proportions of the 3 named constituents vary with season and variety. A. G. POLLARD.

**Effect of arsenic on the composition of citrus fruits.** I. TAKAHASHI (J. Okitsu Hort. Soc., 1930, 25, 153—163).—Spraying citrus trees with arsenates caused a decrease in acidity and an increase in sugar content of fruit juice, with diminished keeping quality. Excessive use of arsenate affects the yield and taste. CHEMICAL ABSTRACTS.

**Chemical eradication of *Ribes*.** H. R. OFFORD (U.S. Dept. Agric. Tech. Bull., 1931, No. 240, 24 pp.).—Of numerous chemicals examined,  $\text{NaClO}_3$  proved most successful. One application of 10–50% solution sufficed for *R. petiolase*, but with *R. lacustre* and *R. inerme* 3 applications of 25% solution were necessary. Additions of fish-oil soap and flaked glue (0.01–0.05% of the dry chemical used) improved the spreading and adhesive properties of the solutions. The fire risk in handling solutions of >10% of  $\text{NaClO}_3$  in the field is considerable.

A. G. POLLARD.

**$\text{Ca}_3(\text{PO}_4)_2$  and phosphate rock.**—See VII.

See also A., July, 877, **Influence of the soil on microbiological phenomena. C cycle in sphagnum, an alder-bush soil, and a garden soil.** 882, **Transpiration etc. of the cotton plant. Ripening of oranges.**

## PATENTS.

**Production of non-caking fertilisers.** A. M. CLARK, W. K. HALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,566, 29.1.30).— $\text{NH}_4\text{NO}_3$  is mixed with 1–5% of finely-ground phosphate rock to prevent caking in bulk.  $(\text{NH}_4)_2\text{HPO}_4$  solution, prepared from phosphate rock,  $\text{H}_2\text{SO}_4$ , and  $(\text{NH}_4)_2\text{SO}_4$ , is separated from the  $\text{CaSO}_4$  and treated with aq.  $\text{NH}_3$  to ppt.  $\text{AlPO}_4$  and  $\text{FePO}_4$ . The ppt. is collected, washed, dried, ground, and mixed with  $\text{NH}_4\text{NO}_3$  or with the  $(\text{NH}_4)_2\text{HPO}_4$  obtained by crystallising the mother-liquor.

A. R. POWELL.

**Production of ammonium nitrate-ammonium sulphate mixed fertilisers.** RUHRCHEMIE A.-G. (B.P. 349,998, 24.2.30. Ger., 23.2.29).—The combustion gases from the  $\text{NH}_3$  oxidation process are treated with liquid or gaseous  $\text{SO}_2$ , or  $\text{SO}_2$  may be mixed with the  $\text{NH}_3$  during oxidation, the mixture of  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  produced being then neutralised with  $\text{NH}_3$  and the product mixed with other fertiliser salts.

W. J. WRIGHT.

**Thermal process for production of a fertiliser consisting of lime [calcium] phosphate.** A. BUCHLEITNER (B.P. 350,167, 30.4.30. Austr., 30.4.29).—Phosphorites are continuously reduced in an Fe bath containing P by means of C, so as to enrich the P content of the bath, the P being then partly removed by oxidation and the  $\text{P}_2\text{O}_5$  combined with CaO.

W. J. WRIGHT.

**Production of organic iron compounds [fertilisers].** H. BLUMENBERG, JUN., and J. H. BLUMENBERG (U.S.P. 1,783,694, 2.12.30. Appl., 31.8.26).—Cellulosic material, e.g., sawdust, is steeped in 20%  $\text{FeSO}_4$  or  $\text{Fe}_2(\text{SO}_4)_3$  solution, removed, dried, and heated at 120° until dark brown or black.

L. A. COLES.

**Manufacture of disinfectant [dusting] compositions [containing mercury].** M. ENGELMANN, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,783,377, 2.12.30. Appl., 27.2.28).—A  $\text{Hg}^{++}$  salt, an alkyl or aryl derivative of a metal higher than Hg in the electromotive series (e.g.,  $\text{PbMe}_4$ ,  $\text{SnEt}_4$ ,  $\text{PbMe}_3\text{Cl}$ ,  $\text{PbPh}_4$ ), and an inert diluent (e.g., tolanite, kieselguhr) are ground together.

L. A. COLES.

**Fungicide and insecticide.** W. C. O'KANE (U.S.P. 1,786,125, 23.12.30. Appl., 18.5.27).—"White" hydro-

carbon oil which has been used for extracting the active constituents of pyrethrum, derris, etc., or which has been mixed with extracts from these, is emulsified with  $\text{H}_2\text{O}$ , and the emulsion is mixed with a colloidal Cu prep. obtained, e.g., by heating CuO or  $\text{CuSO}_4$  with waste molasses or sulphite liquor and NaOH. L. A. COLES.

**Manufacture of [fungicidal] sulphur compositions.** H. BLUMENBERG, JUN. (U.S.P. 1,783,695, 2.12.30. Appl., 14.2.27).—A 9:1 mixture of S and  $\text{H}_3\text{BO}_3$  is sublimed at 130–300°.

L. A. COLES.

**Seed fungicide and disinfectant.** H. ADLER and G. A. McDONALD, ASSRS. to VICTOR CHEM. WORKS (U.S.P. 1,785,472, 16.12.30. Appl., 10.12.27).— $\text{CuSO}_4$  solution is added slowly to a hot aq. suspension of Ca oxalate, and the ppt. is removed and dried.

L. A. COLES.

**Insecticide [for spraying fruit trees etc.].** J. H. LAMBERT, ASSR. to STANDARD OIL CO. (U.S.P. 1,785,451, 16.12.30. Appl., 6.12.24).—An emulsion, which is diluted with  $\text{H}_2\text{O}$  for use, comprises straw oil, Na salts of sulphonated mineral oils, soap, and alcohol. L. A. COLES.

## XVII.—SUGARS; STARCHES; GUMS.

**Influence of temperature of storage of raw [beet] sugars on their affining quality.** O. SPENGLER and A. TRAEGL (Z. Ver. deut. Zucker-Ind., 1931, 81, 289–299).—Two raw beet sugars, light and medium dark in colour and containing 0.9% and 1.5%  $\text{H}_2\text{O}$ , were stored at const. temp. of 20–60° for periods of 8 hr. to 3 weeks, in bottles of 200 c.c. capacity hermetically closed, laid on their sides, and turned at intervals to prevent draining. Their colour was measured before and after the "Zucker-Institut" affining test. At 30° no detectable darkening occurred in 3 weeks, but at 40° darkening was perceptible after 1–2 weeks, and at higher temp. it was more rapid and pronounced, being detectable after 8 hr. at 60° both before and after the affining test. The light sample deteriorated to a relatively greater extent than the darker one. None of the stored samples contained more than a trace of invert sugar. Raw beet sugars should be cooled at least to 30° before storage, especially if stored in bulk, for in sacks there is greater opportunity for subsequent cooling. J. H. LANE.

***Termobacterium mobile*, a Mexican bacterium as new acidifying bacterium for beet slices.** P. LINDNER (Z. Ver. deut. Zucker-Ind., 1931, 81, 25–36).—Isolated from the juice of the agave, collected for the production of Mexican *pulque*, this organism is an effective agent for the conservation of exhausted beet slices etc. by lactic acidification, no AcOH nor butyric acid being formed. It ferments sucrose, dextrose, and levulose, but not maltose or lactose; in presence of air the EtOH formed is completely assimilated.

J. H. LANE.

**Alkalisiation of the diffusion in working dried beet slices.** O. SPENGLER and N. LOGINOV (Z. Ver. deut. Zucker-Ind., 1931, 81, 107–118).—Extracting dried beet slices containing 64.8% of sucrose and 7.6% of moisture with tap-water of  $p_H$  7.6, at 69–76°, in a 5-unit laboratory diffusion battery, with 50 g. of slices per unit of 250-c.c. capacity, the authors obtained juice of 42.1° Brix, purity 89.72%,  $p_H$  5.2, and an invert

sugar : sucrose ratio of 2.1% compared with 1.4% in the dried slices. This increase in invert sugar ratio was almost entirely prevented by addition of 0.4% CaO or 0.8%  $\text{Na}_2\text{CO}_3$ , calc. on dried slices, the juices obtained having  $p_{\text{H}}$  6.9. The purities were substantially the same as without alkali, but the viscosities were 14% and 8% higher with CaO and  $\text{Na}_2\text{CO}_3$ , respectively. Excessive addition of alkali may render the exhausted slices unfit for fodder.

J. H. LANE.

**Continuous indication of alkalinity in the saturation of beet sugar juices by conductivity recording.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1931, 81, 1—12).—In a trial of a Lindner continuous conductometric alkalinity indicator, for the first carbonatation, throughout one campaign in the Genthin sugar factory, with frequent checks of alkalinity by titration and true conductivity, the automatic indications were consistent within the degree of precision usual in the determination of the alkalinity by titration. Subject to confirmation with other juices in other factories, the automatic indicator is considered practicable for the first carbonatation, but not for the second, the irregularities being too large in relation to the very small alkalinity in the second carbonatation.

J. H. LANE.

**Improved utilisation of carbon dioxide in carbonatation [of beet juice].** I. O. SPENGLER, G. BARTSCH, and J. WIGAND (Z. Ver. deut. Zucker-Ind., 1931, 81, 173—182).—An experimental form of carbonatation apparatus, with a packed scrubbing column, enabled the proportion of the total  $\text{CO}_2$  utilised to be increased from 50% to 85%, with a saving of 20% of time, but the juice was not purified to the normal extent, the scum-cake contained excessive amounts of free CaO and "bound" sugar, and the packing in the scrubbing column became badly encrusted with  $\text{CaCO}_3$ .

J. H. LANE.

**New magnesia process [for beet-juice purification].** F. W. MEYER (Z. Ver. deut. Zucker-Ind., 1931, 81, 378—390).—After the usual double carbonatation beet juice is sulphited to about  $p_{\text{H}}$  7.2 and passed through filter presses containing MgO mixed with 10% of powdered wood charcoal, by which it is decolorised and brought to an alkalinity of 0.01—0.02% CaO, according to its rate of flow. The working of the process in the Wismar factory, where it has been employed for 3 campaigns in white sugar manufacture, is described. Technical MgO powder, as used for (Steinholz) flooring, is employed, but dried above  $110^\circ$  before being mixed with  $\text{H}_2\text{O}$  and 10% of wood charcoal and charged into the presses. With a layer 10 mm. thick, a juice pressure of 1 atm. produces a suitable rate of flow. The decolorising effect is about equal to that of vegetable carbons at one seventh of the cost. When exhausted the material may be partly revived twice by passing  $\text{H}_2\text{O}$  through the presses for  $\frac{1}{2}$  hr. The filters are repacked every 24 hr., but during the last 12 hr. are used only for their alkalisising effect. With sound beets the final thin juice is colourless and the thick juice has a pale lemon colour. The thick juice is sulphited, limed, and carbonated. High-grade white sugar requiring no blue is obtained. In a short note SPENGLER confirms the claims made for the process.

J. H. LANE.

**Diatomaceous earths produced in Japan as the filtration medium of sugar solutions.** I, II. T. TANNO and M. ODAGIRI (J. Soc. Chem. Ind., Japan, 1931, 34, 157—158 B).—Japanese-coast diatomaceous earths are similar in properties to the American Supercells used for sugar solutions and if suitably refined are superior to the American refined earths, which they may replace for sugar filtration.

E. H. SHARPLES.

**Solubility of lime in sucrose solutions according to van Aken.** E. LANDT and E. SAALMANN (Z. Ver. deut. Zucker-Ind., 1931, 81, 361—377).—An account of recent work by van Aken (Diss., Delft, 1930) who confirms the irregular solubility of CaO in sucrose solutions noted by Claassen and, after further investigation by fractional filtration, interprets the results in terms of adsorption, peptisation, and coagulation, denying that CaO forms definite compounds with sucrose.

J. H. LANE.

**Comparative experiments on the purification of raw [beet] juices unfiltered and filtered through the Seitz deposited-fibre filter in the Genthin refinery.** O. SPENGLER and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1931, 81, 300—311).—The Seitz filter, consisting of a closed tank containing vertical gauze cells on which a mixture of fine asbestos and vegetable fibres is deposited from suspension (cf. B., 1926, 807), has proved effective in removing very fine pulp particles from raw juice which has to be made alkaline with CaO for transport from one factory to another. Studying the possibility of its application to juice purification in general, the authors found that filtration of diffusion juice, before the usual liming and carbonatation, removed about 50% of the coagulable matters (AcOH test), but did not markedly affect the purity and ash content of the final thin juice. A saving of 75% of the usual consumption of CaO could be effected by treating raw juice with 0.1% of CaO (calc. on beets) at  $80^\circ$ , then filtering through a Seitz filter, liming further with 0.6% of CaO, and carbonating once to an alkalinity of 0.018%. This method of working yields thin juice of normal quality, but it would probably not be economical owing to the low output of the Seitz filters, which was found to be only 100 litres per hr. per sq. m. of surface for the limed juice.

J. H. LANE.

**Movement of nitrogenous substances in sugar manufacture, Usinskii, 1929—1930.** B. A. LYASKO (Nauk. Zapiski Tzuk. Prom., 1930, 10, 23—38).—The melassigenic N represents about 33% of the total N of the beet. At the diffusion battery about 50% of the total, 80% of the protein, and 10% of the melassigenic N are eliminated, whilst  $\text{NH}_3$ - and  $\text{NH}_2$ -N pass entirely into the juice. 96.5% of the N in syrup is melassigenic.

CHEMICAL ABSTRACTS.

**Experiments on raw sugar with Russian activated carbon.** M. P. KOTLYARENKO and N. K. KONIBOLTZKI (Nauk. Zapiski Tzuk. Prom., 1930, 10, 102—111).—A comparison with Norit. Max. regeneration was obtained by heat and HCl treatment.

CHEMICAL ABSTRACTS.

**Quality of the sweet (wash) water from bone-black filters.** M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauk. Zapiski Tzuk. Prom., 1930, 10, 51—74).—With

decrease in sp. gr. the proportion of non-sugars increases; washing should cease at 2.5 Brix (1.5—1.6% of sugar).

## CHEMICAL ABSTRACTS.

**Saccharification of flaked rice with acids.** T. KOMURO (J. Soc. Chem. Ind., Japan, 1931, 34, 166—167 B).—Hydrolysis of flaked rice is slower than that of potato starch, due to the different properties of the starch granule and to the presence of an amphoteric protein, oryzenin. This substance is deposited when the hot filtrate from the hydrolysed rice is kept (optimum  $p_H$  4.1). The colour intensity of the hydrolysed solution increases, and the efficiency of decolorisation decreases, with increase of  $p_H$ , the former changing rapidly at  $p_H$  3—6.

E. H. SHARPLES.

**Surface tension of solutions of different sugars.** E. LANDT (Z. Ver. deut. Zucker-Ind., 1931, 81, 119—124).—A collection of data published by various workers, relating to sucrose, maltose, dextrose, levulose, and galactose. None of these sugars is surface-active, i.e., each increases the surface tension of its aq. solution.

J. H. LANE.

**Polarimetric determination of potato starch and the relation between dry matter and starch contents in potatoes.** C. VON SCHEELE and G. SVENSSON (Landw. Versuchs-Stat., 1931, 112, 1—43).—The rasped sample is washed on a filter with  $H_2O$  and then with  $N/285$  HCl, heated on a water-bath with more acid to form a paste, autoclaved for 15 min., and subsequently the polarimetric value is taken. The starch-free dry-matter content of potatoes is not a const., but increases with the total dry matter. The Maercker table for calculating the dry matter and starch content from the sp. gr. of potatoes needs rectification. A. G. POLLARD.

**Size of the cells and of the starch granules in potatoes.** G. BREDEMANN and W. SCHULZE (Z. Spiritus-ind., 1931, 54, 157).—The mean diam. of the plant cells in different varieties of potatoes cultivated in the same area varies markedly. The size of the cells is related neither to the size of the starch granules nor to the starch content of the potatoes.

C. RANKEN.

**Spectral colorimeter.**—See I.

See also A., July, 794,  $H_2O$  adsorption [from sucrose solutions] by  $SiO_2$  gel. 802, Hydrolysis of sucrose by strong acids.

## PATENT.

**Insecticides.**—See XVI.

## XVIII.—FERMENTATION INDUSTRIES.

**Alsatian wines.** LOBSTEIN and SCHMIDT (Ann. Falsif., 1931, 24, 220—229).—From the analyses of 19 samples it is concluded that the accepted œnological ratios do not apply, owing to differences of climate and method of manufacture, which are described. The presence of lactic acid and methods of determination of tartaric acid are discussed.

E. B. HUGHES.

**Determination of sorbitol [in wine].** G. FIESSELMANN (Chem.-Ztg., 1931, 55, 490—491).—Adulteration of wine by fruit wine is indicated by the presence of sorbitol. To determine the content of sorbitol, the wine is decolorised with charcoal and conc. under

decreasing pressure at 4—5°. After condensation of  $PhCHO$  for 12 hr. with the sorbitol in the syrup, the benzylidenesorbitol is precipitated by adding  $H_2O$ , filtered, dried in vac. over  $H_2SO_4$ , and weighed. The ppt. should give an Ac derivative having m.p. 98°.

C. RANKEN.

**Production of absolute alcohol in converted distillery plant.** FRITZWEILER and K. R. DIETRICH (Z. Spiritusind., 1931, 54, 155—157).—By the introduction of additional valves and conductors the Barbet and the Guillaume plants, which are used for the rectification of EtOH in ordinary distillery practice, can be economically converted for the production of abs. EtOH by the azeotropic method.

C. RANKEN.

**Gasification of water hyacinth.**—See II. **Thermobacterium mobile.**—See XVII. **Sterilisation of enzymes.**—See XIX.

See also A., July, 794, Adsorption of  $H_2O$  from aq. EtOH. 811, Colorimeter for determining  $[H^+]$  of solutions. 873, Activity of peroxidase. Action of ultra-short electromagnetic waves on amylase. 875, Influence of catalytic elements on alcoholic fermentation. Action of poisons on yeast. Components of activator-Z. 877,  $COMe_2$ -BuOH fermentation.

## PATENTS.

**Manufacture of yeast of high enzymic activity.** M. MOSKOVITS, and KRAUSZ-MOSKOVITS EGYESÜLT IPARTELEPEK RÉSZVÉNYTÁRSASÁG (B.P. 349,201, 20.3.30. Hung., 22.3.29).—After completion of the reproduction of yeast in a dil. mash with strong aeration, assimilable and acidified nitrogenous substances are added so that the mash contains 0.05—0.25% N and 0.3% of acid (calc. as lactic acid), whilst the content of carbohydrates is restricted to 1%. After some hr. the content of carbohydrates in the mash is increased to 6% and the enzymes of the cells are caused to develop by vigorous fermentation.

C. RANKEN.

**Yeast manufacture.** J. H. BARRINGTON, ASSR. to STANDARD BRANDS, INC. (U.S.P. 1,784,618, 9.12.30. Appl., 16.11.23. Renewed 29.4.30).—Yeast is propagated in a nutrient solution the acidity of which is controlled by the addition during the first stages of fermentation of an alkaline substance innocuous to yeast and containing yeast-assimilable N, and by the addition of an inorg.  $NH_4$  salt during the latter stages of fermentation.

C. RANKEN.

**Purification of yeast.** G. T. REICH (U.S.P. 1,783,521, 2.12.30. Appl., 8.3.29).—The yeast, which is removed from the fermenters by mechanical means, is diluted with  $H_2O$  at 40° and purified by fractional gravity separation.

C. RANKEN.

**Preservation of hops.** G. SCHLICK (B.P. 350,780, 18.7.30. Ger., 29.7.29).—The air entering the preserving chambers is loaded with moisture by means of an atomiser so that drying of the stored hops is prevented.

C. RANKEN.

**[Production of] aldehyde-free alcoholic liquids.** H. HEUSER (U.S.P. 1,785,447, 16.12.30. Appl., 28.6.26).—Fusel oil and  $CH_2O$  are removed by evaporating the EtOH-containing liquid until the content of EtOH is

reduced by 25—40%. The boiling residue is transferred to a container, in which it is cooled either in contact with gas free from  $O_2$  or in the completely filled and sealed container.

C. RANKEN.

**Peptonised food.**—See XIX.

### XIX.—FOODS.

**Sterilisation of flours and enzymes in the powdered state [with carbon disulphide].** III, IV. A. J. J. VANDE VELDE (Bull. Soc. chim. Belg., 1931, 40, 237—242, 247—253).—III. Treatment of amylase and pepsinase with cold  $CS_2$  for some days, and subsequent removal of the solvent with a current of air, sterilises without loss of activity.

IV. The method is effective with taka-diastrase, trypsinase, pancreatin, and rennin.

A. A. LEVI.

**Detection of persulphate, bromate, and benzoyl peroxide in flour.** AXON. (Ann. Falsif., 1931, 24, 232—233).—Flour is treated on a glass plate with drops of reagents as follows: for persulphate the test is made with 1% KI solution; for bromate, with 0.25*N*- $H_2SO_4$  containing  $NaHSO_3$ , after addition of iodide reagent, or, if persulphate is present, with rosaniline decolorised with  $NaHSO_3$ ; for benzoyl peroxide, with 1% benzidine.

E. B. HUGHES.

**Adulteration of milk by the addition of sugar solution.** A. SABATIE (Ann. Falsif., 1931, 24, 208—211).—Discrepancies between the solids-not-fat and the sum of the lactose, protein, and ash were found to be due to the presence of added sucrose.

E. B. HUGHES.

**How the cream layer forms on milk.** A. C. DAHLBERG and J. C. MARQUARDT (N.Y. State Agric. Exp. Sta. Bull., 1931, No. 591, 11 pp.).—A theory, found to fit the observed facts, is advanced that the positive charges on the Ca ions neutralise the negative charges on the fat globules. This permits the formation of fat clusters, which rise in the milk much more rapidly than would the individual fat globules.

E. B. HUGHES.

**Utilisation of dry skim milk in the manufacture of ice cream and cream cheese.** J. C. MARQUARDT (N.Y. State Agric. Exp. Sta. Bull., 1931, No. 174, 24 pp.).—Dried skim milk stored in unsealed packages for more than 60 days made unsatisfactory ice cream and cheese. The insol. portion of poor-quality dried skim milk is generally composed of casein, but may be a combination of casein and lactose.

E. B. HUGHES.

**Effect of pasteurising and homogenising temperatures on certain properties of ice-cream mixes.** C. D. DAHLE and G. S. BARNHART (J. Agric. Res., 1931, 42, 675—688).—The processing of ice-cream mixtures at 77—82° decreases the degree of "fat clumping," reduces viscosity and freezing time, and increases protein stability. These advantages are most apparent when pasteurisation and homogenisation are carried out at the high temp. Reduction of the temp. from 170—180° to 150° for homogenising gives less satisfactory results. High pasteurising temp. affects principally the viscosity, overrun, and protein stability, whereas a high homogenising temp. has the greater effect on fat clumping.

A. G. POLLARD.

**Chemical changes in the fat of frozen and chilled meat.** I. Frozen mutton and lamb. II. Chilled beef.

C. H. LEA (J.S.C.I., 1931, 50, 207—213 T, 215—220 T).—I. Changes in chemical composition, appearance, and flavour of the fat of carcasses of lamb were followed during precooling, cold storage at  $-5^\circ$ ,  $-10^\circ$ , and  $-20^\circ$  for periods up to 7 months and during hanging at room temp. subsequent to storage. Free acidity changes were comparatively slight, the max. values recorded being of the order of 1.2% (as oleic acid) for external fat, and somewhat less for kidney fat. In no case were any signs of superficial oxidation of the fat observed during 24 hr. precooling at room temp., and no definite increase in susceptibility to oxidation during subsequent storage could be detected as a result of the precooling treatment. Oxidation of the fat of carcasses held at  $-5^\circ$  or below for periods up to 7 months was very slight, and in no case did oxidation sufficient to affect the flavour of the fat occur when such carcasses were held for 3 days at room temp. in not too powerful light. "Sweating" in the presence of light exerted a deleterious effect on the keeping properties of the fat when held subsequently at room temp. The effect is largely one of light action, so that sweating for short periods in the absence of light might be expected to produce considerably less change. "Sweating," however, even for short periods, produced a marked deterioration in appearance of the carcasses. In these experiments a temp. of  $-5^\circ$  failed to prevent the growth of moulds and yeasts on the carcasses. No visible growth occurred at  $-10^\circ$  or  $-20^\circ$ , and the fat of the meat stored at all three temps. did not appear to be attacked by micro-organisms when held for three days at a mean temp. of  $12^\circ$  subsequent to storage.

II. With regard to the chilling of beef, the implications of the experiments carried out are as follows. In none of these experiments involving either (a) storage at  $0^\circ$  or  $-1.6^\circ$  for 42 days, followed by hanging for 4 days at  $8.6^\circ$  or  $10^\circ$ , or (b) storage at  $0^\circ$  for 60 days, was atm. oxidation of the fat sufficient to render it unpalatable, provided that undue exposure to strong light (sunlight or direct skylight) was avoided. Portions of the fat, particularly the front edge of the breast (or brisket), were, however, found to acquire on prolonged storage a "tainted" flavour which was apparently due to the action of micro-organisms, and was always accompanied by a high free acidity in the fat. The free fatty acids, though not themselves responsible for the flavour, may be used as an index to the activity of the taint-producing organisms. Measures, such as the use of air circulation, or of a storage temp. of  $-1.6^\circ$  in place of  $0^\circ$ , which produced conditions less favourable to the growth of micro-organisms were found to extend the storage life of the fat and hence of the meat.

**Growth of micro-organisms on chilled and frozen meat.** R. B. HAINES (J.S.C.I., 1931, 50, 223—227 T).—Plate counts of the nos. of micro-organisms on frozen lamb stored at  $-5^\circ$  for 238 days showed that on freezing there was a decrease in the nos., followed by an increase on certain portions of the carcase due mainly to the growth of yeasts and moulds. Similar counts on quarters of chilled beef stored at  $0^\circ$  indicated that



bacterial growth may occur in certain cases, and an instance of spoilage by organisms of the *Pseudomonas* group is considered. Definite changes in the "sol. N" were obtained at 0°.

**Distinction between natural and artificial honey.** S. A. SCHOU and J. ABILDGAARD (Dansk Tidsskr. Farm., 1931, 5, 89—105).—The presence of artificial honey in bee honey and *vice versa* may be detected by examination of the ultra-violet absorption spectrum of a 5% solution of the material. The absorption curve for natural honey ascends regularly, but that for artificial honey shows a strong absorption band at 2825 Å., due to the presence of hydroxymethylfurfuraldehyde. The quantity of this latter substance present in artificial honey can be determined accurately by this means, the average content of a number of samples examined being 1%, but the method, although it will reveal with certainty the presence of an admixture of artificial honey in natural honey, does not permit an accurate determination of the amount present.

H. F. HARWOOD.

**Determining N in  $\text{NH}_4$  salts. Ca phosphate.**—See VII. **Citrus fruits.**—See XVI. **Dried beet slices. Saccharification of flaked rice. Starch in potatoes.**—See XVII.

See also A., July, 811, **Colorimeter for determining [H] of solutions.** 858, **Metallised food.** 867, **Nutritive values of hardened oils.** 868, **Nutritive value of caseinogen and autoclaved albumin, and of potato protein and gelatin.** 880—1, **Vitamins (various).** 884, **Org. acids of spinach, broccoli, and lettuce.** 885, **Carotenoids of fruits and vegetables. Micro- and histo-chemistry of fruits and leaves.** 886, **Trigonelline in Gautemala coffee.**

#### PATENTS.

**Leavening preparation.** A. H. FISKE, Assr. to RUMFORD CHEM. WORKS (U.S.P. 1,787,193, 30.12.30. Appl., 2.7.29).—The keeping quality of phosphate baking powders is improved by addition of 2—3% Fe (calc. as  $\text{Fe}_2\text{O}_3$ ) during manufacture. The added ferruginous material should be dissolved in the  $\text{H}_3\text{PO}_4$  from which the phosphate is manufactured prior to its incorporation in the leavening preparation.

E. B. HUGHES.

**Production of dietetic foods.** E. DAHO (B.P. 350,777, 16.7.30).—Aq. extracts of tubers, rhizomes, etc. of edible plants, especially of the *Compositae* and *Synanthereae* families, containing inulin, inositol, etc., replace part or all of the  $\text{H}_2\text{O}$  used in the prep. of bread, biscuits, etc. for diabetics.

E. B. HUGHES.

**Manufacture of peptonised food.** W. R. B. ST. J. GATES and J. TAVROGES (B.P. 350,831, 3.9.30).—Milk predigested at 49° with pancreatic extract, and starch (wheat flour) similarly predigested, are cooled to 7°, mixed in stated proportions, and then condensed or desiccated.

E. B. HUGHES.

**Butter colouring.** K. J. MONRAD, Assr. to C. HANSEN'S LABORATORY, INC. (U.S.P. 1,786,256, 23.12.30. Appl., 13.4.27).—The red component of an orange-coloured butter is neutralised by mixing with the butter an oil-sol. green colouring matter. H. ROYAL-DAWSON.

**Preservation of eggs.** D. E. SEAWARD (U.S.P. 1,785,461, 16.12.30. Appl., 31.5.28).—The eggs are wrapped in paper pretreated with 1—2% aq.  $\text{AgNO}_3$ , so that air, light, and bacteria are excluded. Containers may be treated similarly.

E. B. HUGHES.

**Preservation of meat, carcasses, or the like.** H. WATKINS-PITCHFORD (B.P. 350,758, 7.7.30).—Meat brushed or sprayed with a protective covering of blood-serum will retain its fresh appearance for a considerable time; mould and bacterial growth upon its surface is restrained and loss of wt. due to drying is retarded.

E. B. HUGHES.

**Vitaminised food seasonings.** E. MAYBURY (B.P. 350,684, 14.5.30).—Vitamin concentrates from vegetable sources are coated with powdered rice, yeast, or egg to give a free-running powder, which is blended with dried spices.

E. B. HUGHES.

**Rapid pickling of meat [in airtight containers].** M. NEGELE-FISCHER (B.P. 351,355, 2.10.30).

[Case for] **preservation by cooling of foodstuffs and the like exposed for sale.** A. E. SIMCOCK (B.P. 350,620, 29.3.30).

**Treatment of tea etc.**—See XX.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Ipecacuanha preparations and their stability.** H. ESCHENBRENNER (Pharm. Ztg., 1931, 76, 694—696).—Notes on the stability of infusions and fluid extracts of ipecacuanha root prepared under various conditions are given. Double infusions, 1 : 400, with the addition of small quantities of HCl gave extracts of most const. composition and highest alkaloid activity. A fluid extract, 1 : 20, prepared with dil. alcoholic HCl suffered no loss in alkaloid activity over a period of nearly 2 years.

E. H. SHARPLES.

**Testing of ferrum reduction D.A.B. VI and distinction between this and ferrum pulveratum D.A.B. VI.** M. SIEGERT (Pharm. Ztg., 1931, 76, 624—625).—The methods of testing ferrum reductum described in D.A.B. VI do not distinguish between reduced Fe and powdered Fe. These two products may be distinguished from one another by shaking with 20 times the wt. of cold  $\text{H}_2\text{O}$ ; in 1 hr. all the powdered Fe has settled, whereas the reduced Fe forms a colloidal suspension; in 48 hr. the walls of the vessel are covered with a brown layer if the prep. is powdered Fe, whereas reduced Fe has settled completely and the deposit is full of gas bubbles, but no brown flakes remain floating. If the prep. is heated in the air to dull redness, reduced Fe becomes rapidly covered with a red layer of  $\text{Fe}_2\text{O}_3$ , whereas powdered Fe is converted into black  $\text{Fe}_3\text{O}_4$ . The preps. can also be distinguished from one another under the microscope, reduced Fe appearing matt and lustreless, whereas powdered Fe shows sharp edges and irregularly sized particles which are more or less burnished.

A. R. POWELL.

**Preparation of bismuth oxyiodide tannates.** A. MIHALOVICI and G. SPECH (Pharm. Zentr., 1931, 72, 401—402).—The method given is a modification of that described in G.P. 101,776.

E. H. SHARPLES.

"**Enfleurage**" of **jasmin**. TRABAUD (Parfum. Mod., 1930, No. 2, 84—85).—Me anthranilate and indole, which improve the odour, are obtained in extraction by fat. The method affords a higher yield than ordinary extraction. CHEMICAL ABSTRACTS.

**Bactericidal efficiency of essential oils**. R. E. MILLER (Amer. J. Pharm., 1931, 103, 324—328).—The bactericidal efficiency of numerous essential oils and synthetics has been tested by their action on *B. typhosus* and *S. albus* and by a determination of their PhOH coeffs. When two or more of the oils or active principles are mixed in a solution of 31 pts. each of EtOH, glycerin, and H<sub>2</sub>O, and 6.6 pts. of soap, the resulting mixture has a greater bactericidal efficiency than when each substance is used separately and in larger units. E. H. SHARPLES.

**Bergamot industry**. F. LA FACE (Boll. Uff. Staz. Sperim. Ind. Ess. Agrumi Calabria, 1931, 6, 46—67).—A review of the Calabrian bergamot industry, with suggestions for its technical improvement. The content of essential oil does not vary regularly as the gathering season (Dec.—Feb.) advances, although generally higher yields are given in the early, and lower ones in the later, periods. Improved extraction of the essential oil is realisable by the use of the machine now employed in Sicily for the treatment of lemons, the fruit being subjected to centrifugation and sprayed with H<sub>2</sub>O, which is returned to process after the oil has been separated. Addition of salts to the H<sub>2</sub>O, in accordance with Cusmano and Bennett's patent, enhances the separation of the oil, which is of excellent odour and paler than that obtained with the ordinary presses, and contains less stearoptene and more esters. T. H. PORE.

**Some abnormal aniseed oils and B.P. requirements**. W. M. SEABER and S. MARSHALL (Perf. Ess. Oil Rec., 1931, 22, 163—165).—The abnormally low *n* vals. (< 1.5520 at 25°) of some otherwise quite satisfactory star-anise oils, these values bearing no relationship to the f.p. or m.p., are not due to deficiency of anethole. There is present in such an oil a fraction of low b.p. and *n* val., but otherwise the oil is quite genuine. It is suggested that the B.P. limit should be lowered to *n*<sub>D</sub><sup>25</sup> 1.5510. Distillation ranges of several oils are given, and it is considered that the B.P. requirements are too strict. E. H. SHARPLES.

**Essential oil of *Eucalyptus rariflora* (Bailey)**. A. R. PENFOLD, C. B. RADCLIFFE, and W. F. SHORT (J. Proc. Roy. Soc. New South Wales, 1930, 64, 101—114).—*E. rariflora*, on steam-distillation, gave 2.05—2.94% (2 samples) of oil having, respectively: *d*<sub>4</sub><sup>15</sup> 0.9072, 0.9154; *z*<sub>D</sub><sup>20</sup> —8.5°, —10°; *n*<sub>D</sub><sup>20</sup> 1.4872, 1.4909; ester val. 10.0, 15.4; ester val. after acetylation 58.0, 75.4; solubility in 80% EtOH 0.8 vol., partly in 10 vols. The oil contained Δ<sup>4</sup>-carene, β-phellandrene, *l*-α-pinene, β-pinene, cymene, cineole (10—15%), sesquiterpenes (principally aromadendrene), sesquiterpene alcohols, with small quantities of cuminal, phellandral, and cryptal, unidentified phenols, and dehydroangustione (β-diketone). E. H. SHARPLES.

**Essential oils from some cultivated Eucalypts**. II. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1931, 64, 210—223; cf. B.,

1927, 28).—Particulars of oils from the following trees grown from seed in New South Wales are given: *E. Australiana* (1.8% yield, *d*<sub>4</sub><sup>15</sup> 0.9191, *z*<sub>D</sub><sup>20</sup> +4.6°, *n*<sub>D</sub><sup>20</sup> 1.4651; contained 50—56% of cineole, no phellandrene), *E. Macarthuri* (0.2—0.55%, *d*<sub>4</sub><sup>15</sup> 0.9248—0.9326, *z*<sub>D</sub><sup>20</sup> +2° to +4.75°, *n*<sub>D</sub><sup>20</sup> 1.4705—1.4753; geranyl acetate 62.5—71.5%), *E. Macarthuri* from Kenya Colony (0.22%, *d*<sub>4</sub><sup>15</sup> 0.9247, *z*<sub>D</sub><sup>20</sup> +2.2°, *n*<sub>D</sub><sup>20</sup> 1.4702; geranyl acetate 69.5%), *E. Smithii* (0.63%, *d*<sub>4</sub><sup>15</sup> 0.9235, *n*<sub>D</sub><sup>20</sup> 1.4657, cineole 65%), *E. citriodora* (0.6—1.4%, 5 samples grown at different times of the year described), *E. Dives* (3.4—3.8%, *d*<sub>4</sub><sup>15</sup> 0.8891—0.9037, *z*<sub>D</sub><sup>20</sup> —64.8° to —72.5°, *n*<sub>D</sub><sup>20</sup> 1.4794—1.4812, piperitone 52%), *E. Dives*, var. *B.* (3.1%, *d*<sub>4</sub><sup>15</sup> 0.9149, *z*<sub>D</sub><sup>20</sup> —7.2°, *n*<sub>D</sub><sup>20</sup> 1.4660, cineole 45—48%, piperitone 8%, much phellandrene), *E. Dives*, var. *A.* (3%, *d*<sub>4</sub><sup>15</sup> 0.8803, *z*<sub>D</sub><sup>20</sup> —55.25°, *n*<sub>D</sub><sup>20</sup> 1.4793, piperitone 3%). *E. bicostata* (Maiden, Blakely, and Simmonds). Oils from different types (5) of leaves of this form of *E. globulus* were obtained in 1.23—2.4% yield and had *d*<sub>4</sub><sup>15</sup> 0.9194—0.9336, *z*<sub>D</sub><sup>20</sup> +8° to +20°, *n*<sub>D</sub><sup>20</sup> 1.4646—1.4804, ester val. 6.0—24.6, ester val. after acetylation 36.3—103.7, solubility in 70% EtOH 1 in 1.1 to 1 in 1.5. The principal constituents were cineole 38—65% (o-cresol method), *d*-α-pinene, eudesmol, and isovaleraldehyde. E. H. SHARPLES.

**Essential oils of three species of *Geijera* and the occurrence of a new hydrocarbon**. I. A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1931, 64, 264—297).—Leaves and terminal branchlets were steam-distilled in each case. *G. parviflora*, var. *A.* (N.S.W.), gave 0.17—0.53% of essential oil having *d*<sub>4</sub><sup>15</sup> 0.8676—0.8942, *z*<sub>D</sub><sup>20</sup> +22.8° to +50.85°, *n*<sub>D</sub><sup>20</sup> 1.4721—1.4886, ester val. 1.8—15.2, ester val. after acetylation 14.5—48.9, solubility in 80% EtOH, insol. in 10 vols. (11 samples) and sol. in 10 vols. (1 sample), and containing *d*-α-pinene, camphene, limonene, dipentene, cineole, sesquiterpenes, and phloracetophenone dimethyl ether. *G. parviflora* from Queensland: five samples grown at Eidsvold gave 0.6—1.18% of oil having *d*<sub>4</sub><sup>15</sup> 0.9070—0.9199, *z*<sub>D</sub><sup>20</sup> —6° and —7° (2 samples), *n*<sub>D</sub><sup>20</sup> 1.4987—1.5042, ester val. 4.5—15.1, ester val. after acetylation 71.2—103.0, and containing linalool, a hydrocarbon, C<sub>11</sub>H<sub>18</sub> (b.p. 198—199°/774 mm., *d*<sub>4</sub><sup>15</sup> 0.8788, *z*<sub>D</sub><sup>20</sup> 0°, *n*<sub>D</sub><sup>20</sup> 1.4914), constituting about 50% of the oil, and unidentified terpenes, together with small amounts of azulene and octoic acid. Oil from material grown at Rockhampton was similar to the Eidsvold oil with the exception that a considerable quantity of phloracetophenone dimethyl ether was present. Plants from Dalby gave an oil resembling those from New South Wales and containing pinene and camphene in quantity. *G. Muellieri* (Bentham) yielded only 0.09% of oil having *d*<sub>4</sub><sup>15</sup> 0.9132, *z*<sub>D</sub><sup>20</sup> +14.8°, *n*<sub>D</sub><sup>20</sup> 1.4920, ester val. 12.9, ester val. after acetylation 57.0, solubility in 80% EtOH, insol. in 10 vols., and containing *d*-α-pinene, *d*-camphene, cadinene, sesquiterpene alcohol, and an unidentified alkali-sol. substance (0.3%). *G. salicifolia* (Schott) from Eidsvold gave 0.02—0.1% of essential oil and 0.2—1.27% of solid oil, the latter being phloracetophenone dimethyl ether (m.p. 82°). The constituents of the oil (*d*<sub>4</sub><sup>15</sup> 0.9139—0.9344, *z*<sub>D</sub><sup>20</sup> —4.8° to +6.4°, *n*<sub>D</sub><sup>20</sup> 1.4839—1.5006, ester val. 8.7—32.0, ester val. after acetylation 105.6—134.3) could not be identified. E. H. SHARPLES.

**Essential oils of *Zieria Smithii* (Andrews) and its various forms.** I. A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1930, 64, 83—89).—Fresh and air-dried leaves of *Z. Smithii* gave 0.4% and 0.9% of oil, respectively, and the respective constants were:  $d_{4}^{20}$  1.0588, 1.0449;  $\alpha_D^{20}$  +5.2°, +4.95°;  $n_D^{20}$  1.5264, 1.5232; ester val. 18.8, 4.6; ester val. after acetylation 28.7, 26.1; solubility in 80% EtOH 1 in 4 vols., 1 in 1.4 vols. The main constituents of the oils were phenol ethers (70—80%), mainly safrole and a little methyleugenol, with small quantities of *d*- $\alpha$ -pinene, sesquiterpenes, eugenol, stearic acid, etc.

E. H. SHARPLES.

**MgO<sub>2</sub>.**—See VII. Examinations with ultra-violet light.—See XI.

See also A., July, 808, Millon's reagent. 813, Determination of Hg<sub>2</sub>Cl<sub>2</sub>. 847, Rotenone from *Derris* root. Perfumes in fungi. 850, Cinchona alkaloids. 854, Sparteine from *Chelidonium majas*. *Corydalis* alkaloids. 855, *Strychnos* alkaloids. Org. derivatives of Si. Org.-Sb compounds. 856, Reagent for free phenolic OH groups in org. compounds. Determination of cocaine and its salts. 857, Microchemical identification of alkaloids. 870, Toxic principle from Nao-Yang-Hua. 871, Alkaloids of gelsemium. 872, Pantocaine, a new local anaesthetic. 876, Decomp. of nicotine in tobacco. 877, Diphtheria antitoxin. Germicidal efficiency of *o*-phenylphenol. 878, Posterior pituitary prep. Separation of sexual hormones. 879, Prep. of Theelol. 880—1, Pure carotene and vitamin-A. Vitamins (various). 881, Cryst. antineuritic substance. Vitamins of fish oils. 886, Oil and alkaloids in seeds of *Papaver somniferum*. Benzylmethylamine in Ma Huang extract.

## PATENTS.

**Mercury derivatives of tetraiodofluorescein.** L. M. ROEG, Assr. to BREWER & Co., Inc. (U.S.P. 1,786,172, 23.12.30. Appl., 11.11.27).—An aq. suspension of erythrosin is treated with Hg(OAc)<sub>2</sub> and AcOH, the mixture diluted with water, boiled, filtered, and the ppt., after washing, dissolved in *N*-NaOH to give a neutral, non-poisonous, stable alkali salt of the mono-Hg derivative of tetraiodofluorescein.

E. H. SHARPLES.

**Manufacture of benziminazolonestibinic acids.** I. G. FARBENIND. A.-G. (B.P. 343,744, 19.2.30. Ger., 30.3.29. Addn. to B.P. 343,072; B., 1931, 612).—An *o*-arylenediaminestibinic acid is treated with 1 mol. of a chloroformic ester; e.g., 4-nitro-*o*-phenylenediamine is diacetylated, reduced, diazotised, and converted into the diacetylstibinic acid, which is hydrolysed and condensed with ethyl chloroformate. C. HOLLINS.

**Extraction of pyrethrum.** W. J. TREVILLIAN, Assr. to W. T. RAWLEIGH Co. (U.S.P. 1,786,967, 30.12.30. Appl., 14.4.29).—Pyrethrum flowers are extracted with a petroleum fraction and the marc is expressed with a twisting motion, i.e., in a screw conveyor having a diminishing pitch. The residue is re-extracted and re-expressed.

E. H. SHARPLES.

**Manufacture of a thyroid gland preparation.** I. G. FARBENIND. A.-G. (B.P. 349,966, 28.2.30. Ger.,

28.2.29).—An aq. extract of thyroid glands is treated with a limited quantity (40—45 pts. by vol.) of saturated aq. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> or other very sol. salt. The ppt. is rejected and the aq. extract completely or fractionally precipitated by further addition of the saturated solution, the total of which added does not exceed 80% of the original aq. extract. The ppts., which have the character of I-albumoses (0.6—0.8% I), are purified by dialysis and reprecipitation. E. H. SHARPLES.

**Manufacture of lens extract.** E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 1,787,179, 30.12.30. Appl., 11.10.26).—Lenses of the eyes of animals, e.g., hogs or cattle, are extracted, preferably several times and both hot and cold, with EtOH. The combined extracts are filtered, evaporated with the addition of H<sub>2</sub>O until all the EtOH is removed, and the residue is defatted, conc., filtered, and preserved.

E. H. SHARPLES.

**Treatment of tobacco, tea, petals of flowers, and the like aromatic vegetable products.** MONTIS A.-G., and S. ROSENHOCH (B.P. 349,992, 27.1.30).—The products, preferably at 30—70°, are treated electrically in the presence of metals having a bactericidal action. The Cu, Ag, etc. ions may be dispersed throughout the material or form the electrodes (in the form of rollers, bands, etc.) or the substances may be treated between two metallised travelling bands which form the electrodes. The material may be ozonised either before, during, or after treatment.

E. H. SHARPLES.

**Manufacture of vaccine for hog cholera.** M. DORSET (U.S.P. 1,784,928, 16.12.30. Appl., 1.9.28).—Hog cholera virus is treated at below 41° (37.5° for about 48 hr.) with CH<sub>2</sub>O in an amount sufficient to destroy its disease-producing property without affecting its immunising power.

E. H. SHARPLES.

**Perfumery. [Adsorption of perfumes.]** E. E. REID (U.S.P. 1,786,630, 30.12.30. Appl., 31.10.22).—Flowers are treated with powdered adsorbent mineral gel or other adsorbent, either directly or in counter-current direction in a specified apparatus and, if desired, in a tenuous atm. The impregnated adsorbent may be used as such or the odorous material recovered by a counterflow solvent-extraction process.

E. H. SHARPLES.

**Insecticides.**—See XVI. **Dietetic foods.**—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Measurement of the sensitivity of [photographic] emulsions. Comparative results by different methods.** L. LOBEL and M. DUBOIS (Bull. Soc. franç. Phot., 1931, 18, 52—61).—From comparisons with practical exposure tests, the Hurter and Driffield (inertia) method is more accurate than are the methods of Jones and Russel, Labussière, or Scheiner. Practical results are on the average 25% higher than those given by the first method.

J. LEWKOWITSCH.

**Effect of environment on photographic sensitivity** III. **Effect of sodium sulphite.** S. E. SHEPPARD and E. P. WIGHTMAN (Phot. J., 1931, 71, 281—287; cf. B., 1929, 303).—KBr, except at strong concentrations,

lowers the speed of an emulsion only when present during exposure;  $\text{Na}_2\text{SO}_3$  is effective when plates are treated either before, during, or, to a lesser degree, after exposure. Gamma is decreased only by  $\text{Na}_2\text{SO}_3$ .

J. LEWKOWITSCH.

**Additive and subtractive effect in two successive photographs.** R. LANDAU (Sci. Ind. phot., 1931, [ii], 2, 1—4; Chem. Zentr., 1931, i, 1868).—The desensitising action on a photographic film of short exposure to light of high intensity is discussed. The effect was not observed with positive film, and hence appears to be connected with the sensitisation of the emulsion.

A. A. ELDRIDGE.

**Photographic gelatin.** R. SPYCHALSKI and J. TOMASZEWSKI (Przemysl Chem., 1931, 15, 202—213).—The higher the initial viscosity of gelatin solutions, and the smaller the temp. change of this value, the more suitable is the given gelatin for photographic purposes. The presence of salts used in photography, such as bromides etc., considerably affects the viscosity of gelatin solutions. The m.p. and setting point of gelatin jellies are also useful criteria of their technical suitability. All salts examined lower the m.p. and f.p., the action of anions being in the order  $\text{Cl} > \text{Br} > \text{NO}_3$ .

R. TRUSZKOWSKI.

**Composition of developer in mechanical development of kinematographic positive film.** J. I. CRABTREE and C. E. IVES (Sci. Ind. phot., 1931, [ii], 2, 26—31; Chem. Zentr., 1931, i, 1868).—Changes during development, and additions necessary to counter-balance them, are discussed.

A. A. ELDRIDGE.

**Photographic reducers.** G. ROBIN (Sci. Ind. phot., 1931, [ii], 2, 24—26; Chem. Zentr., 1931, i, 1868).—A study of the action of  $\text{KMnO}_4$ ,  $\text{Ce}(\text{SO}_4)_2$ ,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , and Farmer's reducer in relation to contrast.

A. A. ELDRIDGE.

**Photochemistry on paper and glass.** A. REYCHLER (Bull. Soc. chim. Belg., 1931, 40, 254—258).—The latent image produced on filter paper soaked in an aq. fine suspension of  $\text{AgBr}$ , or  $\text{AgNO}_3$  solution, or a  $\text{Ag}$  sol, then partly dried, exposed, and washed, can be developed physically. An emulsion on glass, exposed while still fluid, through the back of the plate, and washed off, leaves a developable latent image adhering lightly to the glass.

J. LEWKOWITSCH.

See also A., July, 805, **Cathodic projection of elements. Light sources for photochemical reactions.** 806, **Separate processes in ripening. Theory of the latent image. Fastness to light of azo dyes. Photographic effects of vitamins-A and -B.**

#### PATENTS

**Manufacture of photographic pictures on chromated gel layers.** N. LEBEDENKO (U.S.P. 1,785,635, 16.12.30. Appl., 28.4.28. Ger., 4.5.27. Cf. G.P. 455,046; B., 1930, 121).—The briefly exposed, sensitised layer is treated before staining with an atm. supersaturated with steam, below  $40^\circ$ ; some  $\text{AcOH}$  or  $\text{CO}_2$  may be added to the vapour. A uniform light exposure may be imposed after short treatment in the dark, and, when on both the back and front of the gel layer, prevents wrinkling.

J. LEWKOWITSCH.

**[Production of kinematographic pictures of long narrow type in] colour photography.** TECHNICOLOR MOTION PICTURE CORP., Assecs. of J. A. BALL (B.P. 351,306, 14.8.30. U.S., 21.8.29).

#### XXII.—EXPLOSIVES; MATCHES.

**Determination of the maximum detonation velocity of nitroglycerin and nitroglycol.** P. NAOÛM and A. BERTHMANN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 188—190).—The tests were carried out by Dautriche's method in seamless steel tubes of various thicknesses and diameters, the explosive being initiated by a No. 8 detonator, with or without a picric acid primer. The average value for nitroglycerin was 8500, and for nitroglycol 8250 m./sec. Dserschkovitch and Andreev's contention (B., 1930, 1170) that the alleged low sensitiveness of nitroglycerin is due to the labile isomeride cannot be maintained on either practical or theoretical grounds. Loss in sensitiveness of stored gelatinous explosives is caused by increase in sp. gr. due to diminution in the air content (cf. B., 1931, 464).

W. J. WRIGHT.

**Determination of velocity of detonation by Dautriche's method with niperyt (pentaerythritol tetranitrate) detonating fuse.** W. FRIEDERICH (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 184—187).—The detonation velocity of various portions from a length of fuse varied over the ranges 0.16—3.76, 0.16—1.30, and 0—0.37% with fuse filled with trotyl, fulminate, and niperyt, respectively. If trotyl fuse is bent the density of the filling is considerably decreased and a reduced velocity is obtained. With fulminate and niperyt fuse this does not occur. The velocities of trotyl, fulminate, and niperyt fuse are 4500—5000, 5000—5300, and 6300—7300 m./sec., respectively. The results of velocity determinations on liquid and solid stable nitroglycerin, blasting "gelatine," dynamites, and trinitrotoluene, initiated by a compressed charge of niperyt and paraffin wax, are tabulated. The highest value obtained for liquid nitroglycerin was 8242 m./sec., and for the solid stable material 8287 m./sec.

W. J. WRIGHT.

#### PATENTS.

**Propellant powder and method of colloiding same.** C. H. KECK (U.S.P. 1,784,189, 9.12.30. Appl., 28.6.27).—Nitrocellulose is rendered colloidal by incorporating it with one or more nitro-aromatic compounds and only sufficient solvent to dissolve the latter, the product being subjected to pressure and heat. A suitable composition consists of nitrocellulose 74.5, trinitrotoluene 15, dinitroxyline 10, and  $\text{NHPh}_2$  0.5%.

W. J. WRIGHT.

**Explosive.** C. H. WATERS, Assr. to DUMOV NAT. CHEM. CO. (U.S.P. 1,786,046, 23.12.30. Appl., 26.3.27).—A chlorate blasting explosive contains liquid mononitrotoluene and liquid dinitrotoluene together with other constituents. A suitable composition is:  $\text{NaClO}_3$  29.84,  $\text{NaNO}_3$  45.86,  $\text{CaCO}_3$  3.31, crude mononitrotoluene 2.62, crude dinitrotoluene 10.91, and sawdust 7.45%. (Cf. U.S.P. 1,073,279; B., 1913, 992.) W. J. WRIGHT.

**Manufacture of explosives and blasting charges.** DYNAMIT A.-G. (B.P. 350,293, 14.7.30. Ger., 22.11.29).—Explosives capable of being cast are produced by forming

eutectic mixtures of monoethanolamine dinitrate and diethanolamine trinitrate or of either of these with methylamine nitrate, other ingredients being added, if desired.

W. J. WRIGHT.

**Manufacture of detonators.** C. A. WOODBURY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,783,372, 2.12.30. Appl., 3.5.28).—The base charge consists of a compound, melting below 100° and having high sensitiveness, *e.g.*, erythritol tetranitrate, which may be mixed with another solid explosive. The charge is melted and allowed to solidify, an initiating composition being then pressed on the top of it.

W. J. WRIGHT.

**Electric blasting cap and ignition material for the same.** E. I. DU PONT DE NEMOURS & Co. (B.P. 350,036, 11.3.30. U.S., 18.3.29).—In electric detonators, the loose igniting composition above the main charge comprises a thiocyanate, an oxidising compound, and a material containing not more than one nitric ester and having a low ignition temp. A suitable composition consists of  $\text{Pb}(\text{CNS})_2$  30–50%,  $\text{KClO}_3$  10–30%, and ground smokeless powder ("Pyro-powder") 30–50%.

W. J. WRIGHT.

### XXIII.—SANITATION; WATER PURIFICATION.

**Effect of iron on the anaërobic decomposition of sewage sludge.** L. R. SETTER (Sewage Works' J., 1930, 2, 504–520).—Gasification is not affected by  $\text{FeCl}_3$  up to 5 p.p.m.; at 5–20 p.p.m. the digestion period is doubled or trebled. Retardation is partly eliminated by neutralisation of the hydrolytic acidity; 10 p.p.m. is not toxic to the organisms, but 20 p.p.m. reduced the microbial population. Fe, or Fe oxide or hydroxide, has no effect;  $\text{FeCl}_2$ ,  $\text{FeSO}_4$ , or Fe acetate or citrate cause slight retardation.

CHEMICAL ABSTRACTS.

**Correlation between biochemical oxygen demand and suspended solids of activated sludge effluent.** H. G. SWOPE (Sewage Works' J., 1930, 2, 500–503).—The demand increases with increase in the solids. The relative increase in the demand is the greater as the period of incubation is prolonged.

CHEMICAL ABSTRACTS.

**Water purification processes at Cleveland, Ohio.** W. C. LAWRENCE (J. Amer. Water Works' Assoc., 1931, 23, 896–902).—Plant studies show that Great Lake  $\text{H}_2\text{O}$  requires a detention period of 2–8 hr. and not more than 1.1 grains of coagulant per gal.; that sand filters remain clean if a 50% expansion with wash-water is available; and that  $\text{NH}_3\text{--Cl}_2$  is preferable to activated C for odour and taste removal. Laboratory tests suggest that  $\text{FeCl}_3$  with a slow rate of mixing will give the most rapidly settling floc.

C. JEPSON.

**Formation of artificial and natural protective layers in water conduits.** L. W. HAASE (Gas- u. Wasserfach, 1931, 74, 572–576. Cf. B., 1929, 266).—The theory of the formation of natural protective layers of oxide or Ca salts on Fe water conduits is outlined. Waters rich in alkaline-earth carbonates and containing dissolved  $\text{O}_2$  are able to form protective layers; waters poor in such carbonates may form such layers, if other conditions are favourable. Acid waters of low carbonate

content form a layer of rust which does not protect from further attack. Some protection is afforded by coating the Fe with Zn or bitumen, but neither method is entirely satisfactory. The protective action of the Zn coating fails after the deposition thereon of a layer of Ca salts. Pb pipes should be used only with waters rich in alkaline-earth carbonates and  $\text{O}_2$ , which rapidly form a protective layer. Strongly corrosive waters may be submitted to deacidification, or should be distributed through conduits of more resistant metal. In hot-water systems two types of deposit are possible, *viz.*, a hard adherent layer and a sludge. The latter is not dangerous, whilst the former is beneficial if not allowed to become too thick. This danger may be avoided by controlling the  $\text{O}_2$  content of the water.

A. B. MANNING.

**Tests for control of water-softening plant.** L. O. NEWTON (Fuel Econ. Rev., 1931, 10, 59–61).—A review of known processes.

D. K. MOORE.

**Fumigation with formaldehyde: an attempt to improve Storm's method.** D. W. HORN and L. E. HUNTER (Amer. J. Pharm., 1931, 103, 332–335).—The action of formalin on a mixture of  $\text{KMnO}_4$  and  $\text{KClO}_3$  is directly proportional to the amount of  $\text{KMnO}_4$  present, *i.e.*, as measured by the % of total  $\text{CH}_2\text{O}$  evolved as gas by the heat generated during oxidation. The need for external heating to initiate the oxidation is not obviated by the use of  $\text{KMnO}_4$  (cf. B., 1930, 122).

E. II. SHARPLES.

**Water for steam plant.**—See I. Corrosion of water-pipes.—See X. Poisonous action of lead pigments.—See XIII. Essential oils as bactericides.—See XX.

See also A., July, 801, Preparation of "equilibrium" and conductivity waters. 877, *o*-Phenylphenol as germicide.

#### PATENTS.

**Water softening and purification.** A. R. MOBERG, Assr. to F. O. PAIGE, JUN. (U.S.P. 1,786,501, 30.12.30. Appl., 15.4.29).—A conc. solution of  $\text{FeCl}_3$  containing peptised  $\text{Al}(\text{OH})_3$  is used to clarify  $\text{H}_2\text{O}$ , and this reagent may be combined with CaO and soda-ash for softening purposes and to coagulate the ppt. W. G. CAREY.

**Purification of swimming-bath water.** R. ADLER (B.P. 350,540, 12.3.30. Czechoslov., 13.3.29).— $\text{H}_2\text{O}$ , continuously withdrawn from the bath, is treated with excess of  $\text{Cl}_2$  and is then passed through carbonaceous material,  $\text{NH}_3$  or an  $\text{NH}_4$  salt being added at any desired point in the cycle of operation. The  $\text{H}_2\text{O}$  may be filtered through a sand, or Mn-removing, filter.

W. G. CAREY.

**Warning means for poison gases.** [Fumigant.] H. LEHRECKE, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,786,623, 30.12.30. Appl., 24.6.26. Austr., 18.7.25).—90–95% of HCN and 10–5% of chloropicrin are absorbed in, *e.g.*, active charcoal or kieselguhr.

L. A. COLES.

**Structures for protection against poisonous gases.** E. KRAEMER and K. HÖRRMANN (B.P. 350,832, 4.9.30).

**Preventing boiler scale.**—See I.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

AUG. 28 and SEPT. 4, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Heating value of superheated and saturated steam.** C. KIRSCHBAUM (Chem. Fabr., 1931, 287—288).—Experiments with two heat exchangers containing, respectively, straight and spiral steam coils showed that the difference between heat transfer with and without superheat is inappreciable. In the calculation of heating surfaces it is sufficient to take the heat-exchange number and temp. fall, referring to saturated steam at the appropriate pressure. C. IRWIN.

**Effect of scrapers on heating, cooling, and mixing.** F. E. HUGGINS, JUN. (Ind. Eng. Chem., 1931, 23, 749—753).—Curves are produced indicating the increased rate of heating or cooling of liquid or semi-liquid masses when efficient scrapers remove the insulating layer formed on the container. The use of scrapers was not justified for thin liquids, *e.g.*, water, but much time was saved with the more viscous liquids as represented by heavy oils. C. A. KING.

**Huillard drying furnaces.** J. HAMON (Bull. Soc. Ind. Mulhouse, 1930, 96, 484—490).—The usual type of Huillard dryer comprises a tower with several superposed perforated hearths with larger central apertures; on a vertical shaft are rabbles rotating on each hearth, working the material inwards, and rotating distributors over each central aperture, upon which the material is worked outwards by fixed rabbles, gases from the furnace passing upwards, and salt or other material to be dried, downwards. On account of its great economy of fuel (1.3% of standard coal against 3.4% for a rotating cylinder) it was formerly adopted for drying KCl; a further economy is possible if waste gases are available. The formation of crusts in the furnace and corrosion in the exhaust fan and cyclone collector (through which some damp salt will pass) were both very serious. The trouble was due to the formation of a NaCl-KCl mother-liquor provided by the first moisture evaporated, and was known to disappear when 3—4% out of the total 8—10% of moisture had been removed; therefore the top hearth was heated more intensely. One method of doing this is to admit about one third of the fresh gases to the top hearth direct, and to exhaust them from above the second while the remaining two thirds pass from the bottom to the same exhaust. A more complicated flow is also described in which all the gases pass countercurrent to the material through at least one hearth before being exhausted. The rotating distributors are also made conical and the fixed rabbles abolished, and provision has been made for controlled admission of cold air to various points. B. M. VENABLES.

**Tank insulation.** ANON. (Ind. Eng. Chem., 1931, 23, 776).—The insulation of the working tanks in a creosoting installation from which the creosote at a temp. of 93° is circulated through the treating cylinders effected a fuel saving of 12.3%. C. IRWIN.

**Multiple-bush hot air and gas pyrometer.** W. GILBERT (Engineering, 1931, 132, 63).—The thermocouple, in this case used for measuring the temp. of the exit gases from a rotary kiln, is surrounded by a number of concentric tubes between which the hot gases pass. This minimises loss of heat from the couple by radiation if the walls of the pipe are at a lower temp. than that of the gases. C. W. GIBBY.

**Weight-%.-mol.-% nomograph.** H. WATERMAN (Ind. Eng. Chem., 1931, 23, 803).—In a binary system of which *A* and *B* are the mol. wts. of the components, a pivot point is obtained on the nomograph where the straight line joining *A* and *B* cuts the centre line. Then any line drawn through this point will join equiv. mol.-% and wt.-% of *A*. Three-component systems may be dealt with by taking them in pairs. The component scales are logarithmic, whilst the mol.- and wt.-% scales are plotted as  $\log(100 - x)/x$ . C. IRWIN.

**Anhyd. liquid SO<sub>2</sub>.**—See VII. **Corrosion prevention.**—See X. **Purification of gases.** **Flue gas purification.**—See XI.

### PATENTS.

**Chemical furnace.** F. L. PRESTON (U.S.P. 1,784,109, 9.12.30. Appl., 7.7.28).—The apparatus comprises two heated pans in line and a single hood which is placed over the pan in action and removed from that being discharged and charged. One side of the sloping roof of the hood is double and is continued over the edge of the pan into a trough, forming an outlet passage and liquid seal for gases, in the latter of which noxious or desired constituents may be absorbed.

B. M. VENABLES.

**Processes for heating material to medium and high temperatures with utilisation of heat of condensation.** E. DAUB (B.P. 349,923, 27.11.29).—Oils are distilled or cracked by utilising the latent heat of condensation of stable cyclic org. compounds of b.p. above 100°, *e.g.*, C<sub>10</sub>H<sub>8</sub>, C<sub>14</sub>H<sub>10</sub>, PhOH, diphenol, quinoline, etc. The temp. in different parts of the apparatus may be regulated as required by suitably controlling the pressure. A. B. MANNING.

**Treatment of liquid furnace material.** C. H. SCHOL (B.P. 350,035, 11.3.30. Ger., 28.1.30).—Liquid slag is poured on to the top of a wheel which is provided

\* The remainder of this set of Abstracts will appear in next week's issue.

with shallow cavities in which  $H_2O$  or other vapour-producing material has been previously charged. The resulting porous mass is allowed to form a continuous band overflowing the cavities and is removed by a rotating scraper at the lower part of the wheel. The  $H_2O$  or other fluid may be supplied through radial pipes attached to the wheel and provided with valves which are automatically open through about  $\frac{1}{4}$  of a revolution and closed for  $\frac{3}{4}$ . B. M. VENABLES.

**Recuperative furnace construction.** A. E. WHITE. From MORGAN CONSTRUCTION CO. (B.P. 350,278, 4.7.30).—Vertical recuperator tubes supported on an apertured plate are provided with means for locking the tubes to the plate so that they cannot creep upwards owing to repeated expansion and contraction.

B. M. VENABLES.

**Heat-exchanging apparatus.** C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 350,190, 12.5.30).—In a heater or cooler having a thermostat in the outgoing stream of fluid that is to be discharged at const. temp., if the flow is irregular (e.g., water-gas) a single thermostat will be insufficient owing to lag. In this invention claim is made for the use of an additional thermostat in the inlet stream or at some intermediate point in the path of the desired fluid, to effect a preliminary regulation of the heating or cooling agent.

B. M. VENABLES.

**Grinding mills.** HARDINGE CO., INC., ASSEES. OF H. HARDINGE (B.P. 350,785, 21.7.30. U.S., 22.7.29).—A screening device situated inside the outlet of a cylindro-conical mill is described. Scoop-like members formed of a grid backed by screening material are attached to the shell near the outlet; these permit undersize to pass out through the trunnion and deliver oversize and balls to shoots which, when they are in an upper position, are inclined back into the interior of the mill. At the same time a central passage is left for the discharge of any abnormal overload of material. B. M. VENABLES.

**Grinding of materials.** J. F. GODDARD, and SUPER CEMENT, LTD. From J. LUNDTEIGEN (B.P. 350,538, 12.3.30).—Material, e.g., cement clinker or gypsum, being ground in the dry state has mixed with it a segregating agent which when rubbed against the particles being ground generates static electricity and causes repulsion of the particles. B. M. VENABLES.

**Pulveriser.** G. H. KAEMMERLING, Assr. to ERIE CITY IRON WORKS (U.S.P. 1,783,717, 2.12.30. Appl., 16.7.29).—A pulveriser of the type having a beater and fan on the same shaft is rendered usable on damp or clogging material by the provision of extra air inlets on the side of the fan casing remote from the beater; the inlets are normally closed by plugs, but in the event of clogging they are opened and an additional rush of air is permitted through the fan. B. M. VENABLES.

**Apparatus for crushing, granulating, or grinding minerals, rock, slags, etc.** J. F. WAKE (B.P. 349,995, 6.2.30).—The apparatus comprises a number of nested rings, of progressively different diameters, which are caused to approach and recede from each other by the action of a central driving cylinder or roll which is either (a) eccentric; and squeezes the intermediate

rings at a rotating point against a resiliently fixed outer ring, or (b) concentric, and squeezes the rings at one point between itself and an outer roll.

B. M. VENABLES.

**Single-roll crusher.** G. W. BORTON, Assr. to PENNSYLVANIA CRUSHER CO. (U.S.P. 1,783,373, 2.12.30. Appl., 9.5.24).—A long single-roll crusher has the breaker plate divided into sections which can yield individually to uncrushable pieces.

B. M. VENABLES.

**Hammer crusher.** G. W. BORTON, Assr. to PENNSYLVANIA CRUSHER CO. (U.S.P. 1,786,694—5, 30.12.30. Appl., 6.6.29).—Crushers of the type described in U.S.P. 1,772,533 (B., 1931, 370) are provided with means for hindering large pieces from entering the disintegrating zone, while permitting them to be struck by the hammers projecting through the grid forming the bottom of the hopper.

B. M. VENABLES.

**Pulverising mill.** F. J. BULLOCK, Assr. to PAPEC MACHINE CO. (U.S.P. 1,785,435, 16.12.30. Appl., 21.1.30).—Hammer heads for a disintegrator are constructed of laminated metal, the outer sheets being harder than the inner ones; alternate sheets may be wider than the others.

B. M. VENABLES.

**Automatic temperature control in pulverising mills.** J. CRITES and W. A. KOREN, Assrs. to RAYMOND BROTHERS IMPACT PULVERIZER CO. (U.S.P. 1,783,358, 2.12.30. Appl., 11.1.29).—In an air-borne grinding system operating in a closed circuit and effecting drying of the material by withdrawal of moist air and supply of a corresponding quantity of fresh preheated air, thermostats are provided both in the pulveriser and in the fresh-air supply pipe either of which will cut off the fuel from one of the burners effecting the air heating should the set temp. be exceeded. For coal the former temp. may be about  $45^\circ$  to remove part of the moisture, and the latter about  $180^\circ$  to prevent ignition.

B. M. VENABLES.

**Separation of dry materials.** C. W. H. HOLMES, and BIRTLEY CO., LTD. (B.P. 349,899 and 350,485, [A] 22.11.29, [B] 3.12.29).—Shaking tables with air currents upwards through the decks are described. In (A) the deck slabs of a twin table are independently adjustable; in (B) a table as described in B.P. 332,291 (B., 1930, 886) has both the spillage edge and the banking bar inclined to the line of reciprocation, the latter at the greater angle.

B. M. VENABLES.

**Separation of dry materials.** C. W. H. HOLMES, and BIRTLEY CO., LTD. (B.P. 350,497, 7.12.29).—The materials are allowed to slide down a shoot having adjustable inclination and to impinge upon a roller of glass or other resilient material; the substance having the lower coeff. of friction, e.g., coal, acquires a greater velocity and bounces further. The roller is rotated at an adjustable speed, but in no case fast enough to produce centrifugal effect, and is provided with a brush or other means to remove dust.

B. M. VENABLES.

**Mechanical [air-]separator.** W. M. COOK, Assr. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,783,357, 2.12.30. Appl., 23.12.29).—A separator embodying a rotating distributor plate and upward and rotating air currents is described.

B. M. VENABLES.



**Apparatus for extracting liquid from solid or semi-solid matter by pressure.** R. T. BINNIE and W. MILLER (B.P. 350,509, 12.2.30).—The material is squeezed between chain conveyors. Two separate chains are used: the inner one is guided in a polygonal path by internal rollers; the outer is bent back on itself and almost embraces the inner chain, the part not so embraced serving for the feeding and withdrawal of material.

B. M. VENABLES.

**Classifying and washing apparatus.** H. W. NEWTON, Assr. to DORR Co. (U.S.P. 1,784,246, 9.12.30. Appl., 9.7.25).—A combination of a trommel and a rake classifier is described. The former may be set either longitudinally or transversely to the latter and is provided with a scoop feed and trunnion discharge having provision for draining the oversize.

B. M. VENABLES.

**Continuous weighing of sediment [as precipitated].** A. SEMLER (B.P. 348,459, 14.7.30. Austr., 12.7.29).—Sediments are weighed as they are formed from suspensions etc. by means of a torsion balance with submerged pan. The arm which adjusts the torsion of the spring may either be continually adjusted to follow the wt. of sediment, or it may be set to a predetermined increment in wt. (the pan arm being prevented from following by a stop) and the time taken for the pan to acquire that wt. noted.

B. M. VENABLES.

**Centrifugal machines for separation of materials.** H. KAMMERL (B.P. 348,806, 1.4.30. Ger., 23.11.29).—A sieveless centrifuge is provided with a number of ring weirs which form pockets for the collection of heavy material, *e.g.*, impurities in kaolin or asbestos. Elastic bolsters may be formed in the pockets by the admission of material such as cotton fibre. [Stat. ref.]

B. M. VENABLES.

**Centrifugal bowls.** AKTIEB. SEPARATOR (B.P. 349,880, 26.9.30. Swed., 28.9.29).—In a separator having outlets for heavy matter together with an additional carrier liquid, the original material and the carrier liquid are supplied to separate chambers in the upper part of the bowl hood and led therefrom through closed passages to suitable points in the separating and discharge zones, respectively.

B. M. VENABLES.

**System of filtration.** J. PUGLIESE (U.S.P. 1,783,341, 2.12.30. Appl., 31.8.29).—Material such as sewage is passed through a tank with a bottom inclined in both directions and provided with a series of weirs to entrap heavy solids. The effluent then passes in order through a series of sand and charcoal filters arranged in cascade and finally through a second sloping tank. Closable apertures are provided in the weirs at different levels to discharge retained liquid and solids separately.

B. M. VENABLES.

**Filtration system.** C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,784,132, 9.12.30. Appl., 9.7.28).—The material, *e.g.*, sewage, is supplied downwards through an axial pipe to an intermediate level of a vertical vessel; from this zone the heavy solids settle out and the fluid rises through a filter bed supported on a foraminous shelf and kept clean by a squeegee or other device rotating against its lower

surface. At the top of the vessel the clear liquid is aerated by injected air, and by allowing it to flow over and through perforated shelves. The liquid may finally be allowed to fall through a considerable head and provide the energy to work the squeegee and air compressor.

B. M. VENABLES.

**Cake-discharging means for pressure filters.** J. A. McCASKELL (U.S.P. 1,784,372, 9.12.30. Appl., 7.10.25).—In a filter of the rotating-disc type the tank for prefill is formed of deep corrugations inside the pressure-tight casing. The top edges of the tank on the down-going side are provided with scrapers which remove the cake from the discs and cause it to fall between the corrugations into a longitudinal worm conveyor which, in the receiving portion of its length, has a single worm, but in the delivery portion the worm is double, is entirely surrounded by its casing, and compresses the cake through a restricted outlet, discharging it without allowing pressure medium (air) to escape. Excess moisture is allowed to escape to the prefill tanks through perforations in the worm casing.

B. M. VENABLES.

**Filter-cake discharger.** J. V. ZENTHOEFER, Assr. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,785,237, 16.12.30. Appl., 15.2.27).—A rotary brush or scraper for the removal of filter-cake is constructed of a number of U-shaped strips of rubber or similar material. In the construction of the hub care is taken, by the use of suitably rounded parts, to avoid severe bending stresses in the rubber.

B. M. VENABLES.

**Liquid filter.** SVENSKA ACKUMULATOR AKTIEB. JUNGNER (B.P. 336,122, 29.10.29. Swed., 30.10.28).—A filter is constructed of thin metal plates without distance pieces or projections from the surface, a pack of peculiarly shaped plates is assembled with odd numbers one hand and even numbers the other hand, and the filtering interstices can be cleaned by merely rotating the odd numbers relatively to the even.

B. M. VENABLES.

**Centrifuge.** G. B. PETSCHKE and A. E. DRISSNER, Assrs. to NAT. ACME Co. (U.S.P. 1,783,546, 2.12.30. Appl., 7.9.29).—The bowl comprises a number of concentric cylinders, passage of fluid being permitted only round their ends; the fluid is passed first through the inner compartment in a thin layer on the wall, then through the outer, and finally through the middle compartment. The apparatus is convertible from a clarifier into a machine capable of separating a larger quantity of heavy material, by altering the radius of discharge of the heavier component by effecting rotation of plugs with eccentric apertures.

B. M. VENABLES.

**Mixer [for stiff materials].** K. ADAMS (U.S.P. 1,783,553, 2.12.30. Appl., 21.6.28. Renewed 6.1.30).—A mixer for stiff concrete and other materials comprises a rotating but non-tilting bowl with stirring blades, and a vertical shaft, also with stirring blades, rotated from the top through a screw device and connected at the bottom to a discharge door. For mixing, the bowl and shaft are rotated in such opposite directions that the shaft screws itself upwards and closes the door; afterwards the whole is reversed, the shaft screws itself downwards, the door is opened, and the material pushed out.

B. M. VENABLES.

**Feeder and disintegrator or mixer.** R. V. ARNOLD, E. E. FLICKINGER, and S. E. WANTZ (U.S.P. 1,786,556, 30.12.30. Appl., 3.7.29).—The apparatus comprises a cylindrical casing with apertures in the upper part at each end; above one aperture is a feeding hopper and below it a worm conveyor which extends towards the other (outlet) aperture. Under the latter are stirring blades or rods which effect a certain amount of throw-back and thoroughly mix the material before it is forced out by the action of the previous screw.

B. M. VENABLES.

**Mixing apparatus.** C. R. and A. B. SMITH (B.P. 350,644, 10.4.30).—An apparatus for purposes such as mixing foundry sand comprises a stationary pan with blades rotating in the space between the bottom of the pan and fixed inclined blades. The material is thus given a surging motion and is discharged through a slot in the side of the pan (which does not extend to the bottom), aided, if desired, by a paddle device.

B. M. VENABLES.

**Emulsifying or homogenising apparatus.** Q. MOORE (B.P. 350,716, 5.6.30).—The fluids are forced through a series of superposed spaced plates which are provided with grooves on one side; small apertures extend from some of the grooves through the plates, but no two apertures are opposite each other.

B. M. VENABLES.

**Machines for mixing liquid, semi-liquid, or viscid materials.** A. SONSTHAGEN (B.P. 350,689, 17.5.30).—Within a cylindrical, horizontally disposed casing, which may be heated, are rotated helical members having inwardly projecting teeth or blades which intercalate with other radial blades fixed to a central shaft. Both the inlet and outlet for material are in the upper part of the casing, at opposite ends. B. M. VENABLES.

**Apparatus for the abstraction of heat from liquids.** R. J. WATT (B.P. 350,632, 4.4.30).—The exchanger is especially suitable for the cooling of  $H_2O$  or oil on an aeroplane and consists mainly of a casing in stream-line form to the interior of which is attached corrugated sheets forming passages for the liquid to be cooled.

B. M. VENABLES.

**Water-cooling towers.** L. G. MOUCHEL & PARTNERS, LTD., and A. T. J. GUERITTE (B.P. 350,081, 20.3.30).—A ferro-concrete tower is constructed of precast slabs having diagonal bars protruding from the corners; the bars are hooked or pinned to each other and the spaces filled with concrete cast *in situ*.

B. M. VENABLES.

**Atomisers.** C. FORTIER (B.P. 350,091, 24.3.30).—An atomiser of the type in which a spray of liquid is expelled by a stream of vapour of the same liquid from the same vessel is provided with a jet having means of closure, a mixing chamber, and two collecting pipes terminating at opposite ends both longitudinally and diametrically of the interior of the vessel, so that, whatever the position of the latter, one pipe will deliver vapour and the other liquid.

B. M. VENABLES.

**Apparatus for mixing liquids.** F. DUWE, Assr. to MASCHINENFABR. AUGSBURG-NÜRNBERG A.-G. (U.S.P. 1,786,009, 23.12.30. Appl., 28.5.29. Ger., 13.6.28).—Liquid in a tank is mixed by means of a propeller drawing upwards from the lower part of the tank and

distributing the liquid in a horizontal veil over the surface.

B. M. VENABLES.

**Apparatus for producing aqueous dispersions [of thermo-plastic substances].** A. L. CLAPP, Assr. to BENNETT, INC. (U.S.P. 1,787,339, 30.12.30. Appl., 1.2.27).—The materials are heated separately and charged together into a conveyor-mixer of the worm type. Cold diluting  $H_2O$  may be added in the later part.

B. M. VENABLES.

**Crystallisation [of sugar etc.].** J. R. and T. RAY (U.S.P. 1,785,530, 16.12.30. Appl., 18.1.28).—Four evaporators are operated in quadruple effect of the steam and the flow of solution is partly intermittent and partly continuous from No. 4 to No. 1, then to Nos. 2 and 3 together, No. 1 being operated at the highest temp. and No. 4 at the lowest.

B. M. VENABLES.

**Treatment of fluids with solids.** I. H. LEVIN, Assr. to GAS INDUSTRIES CO. (U.S.P. 1,785,273, 16.12.30. Appl., 1.6.25).—The apparatus is particularly suitable for the purification and dehydration of air by means of solid NaOH. It comprises a number of pressure-tight towers so connected that the gas may be passed through them in any cyclic order. Provision is made for the draining away of NaOH solution and for flushing out one tower at a time with  $H_2O$  or other solvent.

B. M. VENABLES.

**Carrying out gas reactions at high temperatures.** IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, and W. B. FLETCHER (B.P. 349,958, 27.1.30).—Gases which react endothermically are passed at high velocity through unpacked chambers which are long and deep, but very narrow, and alternate with long and deep heating flues, by which the temp. is maintained above  $1000^\circ$ . A method of construction of a furnace with regenerators is described.

B. M. VENABLES.

**Apparatus for chemical reactions.** T. GRISWOLD, JUN., Assr. to DOW CHEM. CO. (U.S.P. 1,785,080, 16.12.30. Appl., 27.1.27).—The apparatus is suitable for reactions between vapours and consumable solid material or between fluids in the presence of a solid catalyst, an example of the former being the manufacture of  $CS_2$ . A number of towers, preferably of ceramic material, are arranged in pairs in a heated setting; one tower of each pair is kept filled with the solid material, *e.g.*, charcoal, and is connected by a passage at the bottom to a second tower which contains a spiral shelf upon the upper end of which the other material, *e.g.*, S, is charged in the molten state through a swan neck, becoming vaporised as it trickles down the shelf and passing upwards through the C. The lower cross-passage also serves for removal of ash, and the heating flues are arranged so that the combustion gases may pass round either tube first and the other second or around both in parallel.

B. M. VENABLES.

**Treatment of flue gases, residual trade gases, and the like.** LONDON POWER CO., LTD., and S. L. PEARCE (B.P. 334,660, 16.7.29).—The gases are first passed through a chamber or conduit in which they are saturated by heated fluid and passed between baffle boards, then, after a change in direction, they are subjected in another chamber to douching with

substantial quantities of liquid, and finally they pass through scrubbers and again change their direction. Besides the usual dry grit catchers, the primary chamber may be adapted to collect grit, another set of scrubbers or moisture eliminators may be provided after the second change in direction, and last of all a quantity of hot air may be added. Catalytic oxidising agents may be provided in the primary chambers, or ozonised air added after pretreatment; the wash-water may be made alkaline.

B. M. VENABLES.

**Condenser for mixed vapours.** G. L. KOTHNY, Assr. to C. H. WHEELER MANUFACTURING CO. (U.S.P. 1,786,163, 23.12.30. Appl., 6.7.25).—The apparatus is suitable for the separate condensation within one shell of oil vapour and steam. The bundle of tubes is divided into sections through which the cooling  $H_2O$  passes in series, baffles in the vapour space being provided to ensure that the incoming vapours strike the warmest tubes first. The oil is condensed on these and floats on the surface of the  $H_2O$  which is condensed on the cooler tubes. The two liquids are withdrawn together in easily separable form.

B. M. VENABLES.

**Apparatus for purifying gases.** S. C. CUTLER (U.S.P. 1,786,150, 23.12.30. Appl., 22.10.26).—The apparatus comprises a tower or like vessel divided into two chambers each of which is provided with a number of atomising discs. In one chamber the gas is purified by contact with, *e.g.*, NaOH solution, in the other the caustic is regenerated by contact with air (*sic*).

B. M. VENABLES.

**Gas-treating apparatus.** W. L. SPALDING, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,785,792, 23.12.30. Appl., 5.10.25).—A gas, *e.g.*, ammonia vented from an autoclave, is bubbled into the lower part of a liquid circuit comprising a rising pipe including a cooling coil, a separating vessel from which permanent gases are vented, and a return pipe, the whole system being maintained under pressure to increase the solubility; provision is made for adding and withdrawing liquor.

B. M. VENABLES.

**Filtering and cleaning of [air] filters.** L. L. DOLLINGER, Assr. to STAYNEW FILTER CORP. (U.S.P. 1,784,278, 9.12.30. Appl., 1.4.25).—A number of air filters are divided into sections, each having a separate conduit leading to the exhaust fan, and hence the air during filtering passes through in parallel. By means of shutters which may be automatically operated by the increase of pressure across the filters, one section at a time is subjected to a high-velocity reverse current from the delivery side of the fan.

B. M. VENABLES.

**Bag filters.** W. SCHEIDT (B.P. 349,913, 27.2.30).—A framework for flat filter bags the sides of which are kept apart by chains, and also detachable means of connexion to the vacuum 'bus-pipe, are described.

B. M. VENABLES.

**Steam dryers and separators and like apparatus for separating liquid particles from steam, air, or other gases.** J. JONES, and A. MCCONWELL & CO., LTD. (B.P. 349,936, 1.3.30).—A separator of the deflexion type suitable for placing in the steam space of a boiler is described.

B. M. VENABLES.

**Separation of the constituents of gaseous mixtures.** C. C. VAN NUYS and J. L. SCHLITT, Assrs. to AIR REDUCTION CO., INC. (U.S.P. 1,784,120, 9.12.30. Appl., 23.10.26).—The original mixture in liquid form is subjected to primary rectification with the object of obtaining a liquid constituent of substantial purity. The effluent and other gaseous mixtures containing the constituents of the original are liquefied and passed through a secondary rectifier with the object of obtaining pure gaseous constituent. The impure liquid from the secondary is introduced into the primary at a point where the downflow liquid has a similar composition, and the pure liquid is subjected to heat exchange with the secondary rectifier to maintain the refrigeration.

B. M. VENABLES.

**Arrangements for delivery of liquefied gases.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 350,882, 29.4.30. Fr., 30.4.29).—The delivery pump and suction chamber for delivering liquefied gas are made of non-corrodible metal and, as a precaution against access of air, are swept by the vapour given off from the upper part of the storage vessel. A shock absorber connected to the delivery of the pump comprises a long-length small-bore tube closed at one end and exposed to the heat of the atm. so that its contents are always gaseous.

B. M. VENABLES.

**Defrosting of heat exchangers in apparatus for liquefaction, or separation by liquefaction, of gaseous mixtures.** L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROD. G. CLAUDE (B.P. 350,057, 14.3.30. Fr., 15.3.29).—Two exchangers are used, one being in operation as such, while the other is being defrosted by a reverse current of a reheated constituent of the gas.

B. M. VENABLES.

**Gas analysing apparatus.** G. A. ALDRICH (B.P. 350,584, 3.12.30).—A gas burette is provided with a transparent gauge tube extending through it, which indicates the level of the  $H_2O$  or other displacing liquid in the pump chamber, to which it is connected by a tail taken downwards and sideways so that any air drawn in will not enter the burette; the upper end of the gauge is open to the atm.

B. M. VENABLES.

**Hygrometers.** NAT. FED. OF IRON & STEEL MANUFACTURERS (CORPORATE ORGANISATION), LTD., and L. REEVE (B.P. 349,941, 4.3.30).—A sample of the air is brought to a const. temp., which is above the dew point and preferably a few degrees above the room temp., by means of a thermostatically controlled heater and allowed to impinge at a const. and sufficient speed (preferably over 5 miles/hr.) upon a single wet-bulb thermometer, which may be calibrated to give direct readings of the moisture content.

B. M. VENABLES.

**Viscosimeter.** A. G. M. MICHELL (U.S.P. 1,786,574, 30.12.30. Appl., 20.7.28).—A viscosimeter (as described in U.S.P. 1,398,878) comprises a ball and a cup fitting on part of the surface, the time being noted for the ball to fall away from the cup under its own wt. when they are stuck together by a film of the liquid. To adapt the apparatus to liquids of high viscosity a collar is provided as an extra wt. for the ball.

B. M. VENABLES.

**Pulverised-coal-burning furnaces.** TODD DRY DOCK, ENGINEERING & REPAIR CORP. (B.P. 351,621, 11.4.30. U.S., 22.4.29).

[Cooled] furnace walls. BABCOCK & WILCOX, LTD. From TOYO BABCOCK KABUSHIKI KAISHA (B.P. 351,822, 5.9.30).

**Reversing valves for regenerative furnaces.** H. CROWE (B.P. 351,573, 18.10.30).

**Heat-recuperating structures.** H. DE GAILLARD & Co. (B.P. 351,863, 17.10.30. Fr., 17.10.29).

**Process and apparatus for refrigeration.** VOGT INSTANT FREEZERS, INC., ASSECS. of C. W. VOGT (B.P. 352,125, 22.4.30. U.S., 13.5.29).

**Refrigerating or freezing plants.** DEUTS. WERKE KIEL A.-G., and P. SOMMER (B.P. 351,751, 7.7.30).

**Heat-insulating walls or partitions.** V. C. FROM and C. D. ROWLEY (B.P. 351,493, 26.3.30).

**Lubricants.**—See II.  $\text{NH}_3$  detector for refrigerators. **Refrigerant.**—See VII. **Coated Fe pipes.** **Boiler parts etc.**—See X.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Rational analysis of coal.** R. V. WHEELER (Gas World, 1931, 94, Coking Sect., 13—16).—Chemical and microscopical studies of coal have indicated that the principal ingredients are (1) free hydrocarbons; (2) resins; (3) structured plant entities resistant to decay, such as spore exines and cuticles; and (4) ulmins, the degradation and condensation products of the cellular tissues and proteins of the plants. Methods are given for the approx. determination of these substances and of the reactivity of the ulmins. Modifications of these methods are necessary when dull coals (durains) are to be examined. Typical results of the rational analyses of a number of British coals are given. C. B. MARSON.

**Microscopical and X-ray study of Pennsylvanian anthracite.** H. G. TURNER and H. V. ANDERSON (Ind. Eng. Chem., 1931, 23, 811—815).—Coal constituents other than fusain are classified as anthraxylon (woody constituents) and attritus. Vitrain is anthraxylon, clarain and durain are mixtures of it with attritus. Coal samples containing anthraxylon bands were studied microscopically and radiographically, the anthraxylon being much more transparent to X-rays than the mineral matter. X-Ray diffraction photographs of anthraxylon show a fibre pattern resembling that of cellulose or graphite. Attritus shows no fibre pattern, but produces Debye-Scherrer rings due to  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , mica, etc. The anthraxylon particle is calc. to contain about 48 C atoms. C. IRWIN.

**Catalytic hydrogenation of bituminous and anthracitic coal and their distillation products.** B. HLAVICA and E. TRČA (Chem. Obzor, 1930, 5, 121—124; Chem. Zentr., 1931, i, 1997).—Hydrogenation of the coals is preferable to that of the products of low-temp. coking, much greater yields of tar and benzene being obtained. A. A. ELDRIDGE.

**Tests with aluminium apparatus for determining the caking power of coal and other substances.**

D. J. W. KREULEN (Chem. Weekblad, 1931, 28, 434—440).—A record of the effects of variations in details of manipulation, preparation of the sample, and conditions of test. S. I. LEVY.

**Effect on certain measurable properties of coke of quenching with ammoniacal liquor.** C. B. MARSON and H. V. A. BRISCOE (Gas World, 1931, 94, Coking Sect., 55—57).—Two series of tests in which cokes made from the same Durham coking coal were quenched (a) with  $\text{H}_2\text{O}$  and (b) with dil. ammoniacal liquor showed that the shatter index of the coke was slightly lowered by liquor-quenching. Examination of the cokes by a laboratory combustibility test in which a measured and const. blast of air was passed through an ignited column of sized coke indicated that the temp. developed and the rate of coke consumption were identical for both cokes. Liquor-quenching did not affect the size or colour of the coke or its behaviour in a domestic grate, and it is concluded that the quenching of such a coke with the dil. liquor is not likely to affect adversely its properties for metallurgical or domestic use or for use in the production of water-gas. It is emphasised that the tests deal only with the effects of quenching with dil. ammoniacal liquor and give no information as to the effects of using effluent liquors. C. B. MARSON.

**Brown-coal gas.** W. ALLNER (Gas- u. Wasserfach, 1931, 74, 305—311).—The analytical characteristics and effects of distillation of typical brown coals and of a bituminous coal are compared. Passage of brown- or bituminous-coal tar (alone or mixed with steam) over incandescent coke produces a gas intermediate in properties between oil gas and coal gas; the character of the liquid and gaseous products is largely determined by the temp. and reaction time. The experimental plant built at Kassel by the Braunkohlengas G.m.b.H. to produce town gas from brown coal on this basis is described in detail. Raw or briquetted brown coal is charged into a continuous vertical retort and the gas produced withdrawn near the bottom of the retort so that the steam and tar resulting from distillation undergo reaction at the surface of the incandescent coke; supplementary steam may also be injected. By variation of the speed of throughput and amount of added steam the composition of the gas may be controlled and, after washing to reduce the  $\text{CO}_2$  content, a suitable town gas results. The composition of the gas and tar obtained in experiments with different brown coals is described. Appreciable amounts of benzol may be extracted from the gas. The character of the coke produced is dependent on the source of the coal, but it is generally suitable for briquetting with brown coal or for water-gas production, and the fine coke is suitable for boiler firing. The heat requirements and the economics of the process are discussed. H. E. BLAYDEN.

**Gas from brown coal.** A. THAU (Gas- u. Wasserfach, 1931, 74, 385—390).—Experiments at the Halle (Saale) gasworks showed that town gas comparable with that made from bituminous coal could be prepared by carbonising brown-coal briquettes in 7 retorts of a battery of 10 vertical retorts and cracking the resulting gas by passage through the remaining 3 retorts packed

with incandescent coke. The throughput was greater with brown coal than with ordinary coal and the coke size was dependent on that of the briquettes used. In the process designed by Seidenschneur, small brown coal is dried in a rotary dryer, briquetted in rotary presses, and the briquettes are transferred to a hopper above the retort and heated to 300° by waste gases, when they lose a large proportion of  $\text{H}_2\text{O}$ ,  $\text{CO}_2$ , and  $\text{H}_2\text{S}$ . They then fall into the retort and are carbonised. The off-take pipe is placed in the hottest zone of the retort so that distillation products undergo a cracking effect at the surface of the incandescent coke. The coke may be used in the gas producer. Methods of removing  $\text{CO}_2$  from brown-coal gas are briefly discussed. H. E. BLAYDEN.

**Effect of properties of petroleum wash oil in removal of light oil from coke-oven gas.** H. R. MATHIAS (Ind. Eng. Chem., 1931, 23, 804—807).—Plant trials with various petroleum wash oils indicate that sludge formation has no connexion with the olefine content of the fresh oil, but that this is minimised by keeping the sp. gr. and viscosity as low as possible. The pitch-like deposit sometimes produced in scrubbers is a mixture of coal tar and petroleum bitumens, and here again the olefine content of the oil is of little value as a guide. Oils of high viscosity were found in the laboratory to give rather higher percentage absorption, but the sludge question is the more important. The "oxidation sludge" test, in which 10 g. of oil are maintained at 162.8° for 5 hr. and the insol. residue is weighed, is of value, but tests for emulsification on steam-distillation, colour, boiling range (except initial b.p.), and olefines are of no use. C. IRWIN.

**Determination of hydrogen and methane [in coal gas etc.] in the Orsat apparatus by Jäger's method of combustion over copper oxide.** H. PAUSCHARDT (Gas- u. Wasserfach, 1931, 74, 613—616).—An Orsat apparatus is provided with a  $\text{SiO}_2$  tube packed with  $\text{CuO}$ , which can be maintained at 290° or 900° by means of a gas burner, and communicates on one side with the water-jacketed gas burette and on the other through a three-way tap with an absorption pipette charged with  $\text{NaOH}$  solution. Between the tap and the pipette is a short length of glass tube which is calibrated and contains a float valve. In analysing a coal gas the  $\text{CO}_2$ , heavy hydrocarbons,  $\text{O}_2$ , and  $\text{CO}$  are absorbed in the usual manner. The combustion tube is then heated to 290°, the three-way tap being turned to allow the displaced  $\text{N}_2$  to escape to air. When the temp. of the tube is const., connexion is made to the pipette and gas burette, and the residual gas is passed backwards and forwards until combustion is complete. The diminution in vol., giving the  $\text{H}_2$  content of the gas, is measured, the temp. of the combustion tube being maintained const. The tube is then heated to 900°, the gas being allowed to expand into the pipette to an observed calibration mark. Combustion is then completed, and the diminution in vol. again measured, with the liquid level in the pipette at the observed calibration mark, while the temp. of the tube is maintained at 900°. The apparatus permits the determination of  $\text{H}_2$  and  $\text{CH}_4$  with accuracy, whilst, by avoiding the necessity of bringing the  $\text{CuO}$  tube to room temp.

before taking each reading, the time required is much shortened.

A. B. MANNING.

**Disposal of ammonia liquor.** F. SCHUSTER (Gas- u. Wasserfach, 1931, 74, 318—319).—Disposal by injection into furnaces or by rejection after treatment is impracticable except in special cases [e.g., when by the use of  $\text{Fe}$  (or  $\text{Al}$ ) sulphate the liquid product may be used directly as a fertiliser]. The direct application of ammonia liquor for land fertilisation is briefly discussed. Distillation of the liquor without addition of  $\text{CaO}$  so as to recover volatile  $\text{NH}_3$  and the use of the waste liquor as a fertiliser, or rejection after suitable treatment (e.g., passage over active C), is considered a practical method. The  $\text{NH}_3$  recovered might be utilised by passing it through a column of incandescent coke partly to decompose it, and mixing the gases produced (i.e.,  $\text{N}_2$ ,  $\text{H}_2$ , and undecomposed  $\text{NH}_3$ ) with the crude coal gas.

H. E. BLAYDEN.

**Treatment of pyroligneous acid.** H. GUINOT (Chim. et Ind., 1931, 25, 1354—1360).—Crude pyroligneous acid freed from the bulk of its tar is treated with  $\text{AcOEt}$  in small proportions to remove the residual tar. It is then treated in a second apparatus with twice its vol. of  $\text{AcOEt}$ , which dissolves all the  $\text{AcOH}$ , together with some  $\text{H}_2\text{O}$ . The extract is distilled, when higher acids, which form const.-boiling mixtures with  $\text{H}_2\text{O}$ , come over first. Each process is worked on the countercurrent principle in a battery of 6—10 units in series. The rectification column yields at the bottom a mixture composed of  $\text{AcOH}$  2 pts. and  $\text{AcOEt}$  1 pt., the components of which are finally separated in a second column, from which 98%  $\text{AcOH}$  is obtained as vapour at two trays from the bottom, the liquid residue consisting of oil and tar. It may be redistilled. The extract from the tar separator is distilled in another column into which vapour of a special petrol fraction (b.p. 100°) is blown. This forms a mixture of min. b.p. with 30% of  $\text{AcOH}$  and the tar is thus freed from acid. The aq. residues, which contain  $\text{AcOEt}$ ,  $\text{MeOH}$ , and  $\text{COMe}_2$ , are distilled so as to remove the  $\text{AcOEt}$  as vapour and the other products are concentrated as usual. The yield of acid by this process is 95% and the steam consumption 10 kg. per kg. of acid. The loss of  $\text{AcOEt}$  is below 1% on the pyroligneous acid treated; the apparatus, which is constructed of bronze and  $\text{Cu}$ , requires only one attendant. C. IRWIN.

**Comparison of liquid fuels in regard to knocking; methods of measurement and reference scales.** C. BONNIER (Chim. et Ind., 1931, 25, 1339—1347).—The antiknock value of a fuel cannot be determined in the laboratory and is not measured by the temp. of spontaneous ignition. Test-bench measurement involves the use either of a motor of variable compression ratio or of one of high fixed compression ratio with controlled admission. All variables such as cooling-water temp., composition of mixture, etc. must be rigidly controlled and, even so, results vary with the wear of the engine. Reliable results can consequently be obtained only by comparison of the test result with that given under the same conditions by a standard fuel. This requires a mixture of two fuels of high and low antiknock values and the tendency is for the

general adoption of *isooctane* and *heptane*. It would be better if the scale could be prolonged, as  $C_6H_6$ -EtOH mixtures have a much higher antiknock value than has octane. C. IRWIN.

**Improved Engler-Heusler apparatus for the determination of sulphur in benzine, petrol, and benzene.** J. FORMÁNEK (Chem.-Ztg., 1931, 55, 531).—The burner consists of two concentric glass tubes through the inner of which a gentle current of air is passed to assist combustion of the oil, the vapour of which is drawn up the annular space between the tubes. These tubes are pushed through the central opening of a perforated cork at the bottom of a conical cylinder, the top of which is connected to the absorption apparatus by a pipe which passes to the bottom and terminates in a fine jet. The absorption apparatus comprises a long cylinder with lower stopcock and is filled with glass beads covered with neutral 3%  $H_2O_2$  solution. A. R. POWELL.

**Chemistry of the "doctor" sweetening process [for petroleum distillates].** A. LACHMAN (Ind. Eng. Chem., 1931, 23, 354–357).—On treating gasoline containing mercaptans with Na plumbite, Pb mercaptide is produced first, which is sol. in the petroleum phase. The addition of S then produces an org. disulphide and PbS. In the presence of air the PbS is reconverted into plumbite, but this takes place only when the  $Na_2S$  has been completely converted into sulphate or thiosulphate. PbS appears also to be able to act catalytically in bringing about the conversion of mercaptans into disulphides in the presence of S and NaOH. Experiments show that mercaptans can be oxidised to disulphides in the presence of NaOH and air without the addition of PbO or PbS. The knowledge of the reactions involved is useful in controlling the doctor process for sweetening gasoline. It is advantageous to remove  $H_2S$  before treating with doctor solution, and air for agitation should be finely divided. Efficient agitation is necessary. The calc. quantity of finely-divided S should be added at once and not in small amounts. T. A. SMITH.

**Determining the refining degree of lubricating and transformer oils by means of the "sulphuric acid-absorption diagram."** G. TOENNIES (Petroleum, 1931, 27, 527–534).—In determining the percentage of lubricating and transformer oils by the action of 90–100%  $H_2SO_4$  and plotting the values for the amounts of absorption and concentration of the acid, curves of characteristic and individual shape for each kind of oil are obtained. Comparison of these diagrams with those given for the oxidising qualities of the oils indicates that there is a relationship existing between them. E. DOCTOR.

**Lubricating oils from crude oil with or without paraffin.** H. ORDELT (Petroleum, 1931, 27, Motor-entbtr., 4, 3–9).—Temp.-viscosity diagrams for various kinds of paraffin and ceresin show a distinct relationship to the setting point, but no relation to the fraction of distillation. Oils containing paraffin are not always of inferior lubricating value. E. DOCTOR.

**Solubility of water in liquid hydrocarbons.** D. N. TARASENKOV and E. N. POLOSHINCEVA (J. Gen.

Chem. Russ., 1931, 1, 71–79).—The % solubility of  $H_2O$  at 20° is 0.053 in  $C_6H_6$ , 0.035 in PhMe, 0.023 in xylene, 0.010 in *cyclohexane*, 0.008 in Grosny light petroleum, 0.06 in kerosene, 0.004 in paraffin oil. The  $H_2O$  content of hydrocarbons may be estimated from a determination of the temp. at which turbidity appears. R. TRUSZKOWSKI.

**Effect of scrapers on heating etc. Tank insulation.**—See I. **Partial oxidation of  $CH_4$ .**—See III. **Bleaching clay.**—See VII. **Deterioration of structures. Asphalt fillers.**—See IX. **Agglomeration of Fe ore.**—See X. **C black.**—See XIII. **Soot for rubber compounding.**—See XIV.

#### PATENTS.

**Combustion systems for powdered fuel.** E. F. WEISS (B.P. 349,495, 20.2.30. U.S., 25.2.29).—An electric control system is provided for automatically starting and stopping the prime movers (steam turbines, electric motors, etc.) which operate the fuel and the combustion air supplies, in such a sequence as to prevent the accumulation of fuel or explosive mixture in the furnace. A. B. MANNING.

**Combustion of fuel for production of high temperatures.** H. WADE. From DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 349,585, 28.11.29).—The rate of flow of the combustible mixture (gas, atomised oils, or coal dust etc. and air) entering the combustion chamber is regulated, at a relatively low pressure, to at least 6–7 m./sec., the time during which the gases remain therein is maintained below 0.02 sec. (preferably about 0.01 sec.), and the combustion space is so designed that the ratio of surface area to vol. is at least 20 sq. dm./litre. Catalytic surfaces formed of substances of strong electronic emission, e.g., alkaline-earth oxides, may be introduced into the combustion chamber. Furnaces embodying these features are described. A. B. MANNING.

**Combustion of fuel.** H. A. GILL. From STRATTON ENG. CORP. (B.P. 349,915, 1.3.30).—Crushed fuel (up to  $\frac{1}{4}$ -in. size) is burned in a chamber while held in suspension therein in a blast of air projected upwardly from a nozzle underneath the chamber. The nozzle can be moved laterally. The velocity of the air blast is insufficient to support the ash, which falls past the nozzle on to a grate which can be given a reciprocating motion in order to maintain the ash in a loose condition. By lowering the velocity of the air blast and permitting the accumulation of fuel in the lower part of the chamber the fire can be banked. A. B. MANNING.

**Coke ovens.** N.V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & Co. G.M.B.H. (B.P. 349,604, 27.2.30. Ger., 27.2.29).—A coke oven with pairs of regenerator chambers for preheating gas and air, alternating with other pairs for storing the waste heat, is provided with horizontal or vertical channels in the walls separating the pairs of regenerators. These channels communicate with the outside air and prevent leakage of air or gas from the preheating regenerators to the waste-heat regenerators. A. B. MANNING.

**Coking retort oven.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,784,414, 9.12.30. Appl., 2.1.23. Renewed

1.4.30. Cf. U.S.P. 1,374,546; B., 1921, 378 A).—The vertical combustion flues of each heating wall of the oven are connected in two or more sets by horizontal flues, which communicate with the corresponding flues of an adjacent heating wall. Below the coking chambers are tunnel structures extending longitudinally of the oven, and divided into individually regulable and reversible regenerators, which are connected with the flues of the heating walls in such a manner that the air and the heating gas, or the air only, when a rich gas is used, can be preheated therein. A. B. MANNING.

**Carbonisation of coal.** N.V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & Co. G.M.B.H. (B.P. 350,262, 25.6.30. Ger., 25.6.29).—Swelling coals are carbonised without difficulty in ordinary retorts or coke ovens by maintaining their bulk density as low as possible. This is effected by charging the finely-ground coal, the  $H_2O$  content of which has been adjusted to lie between 6% and 10%, into the carbonising chamber as loosely as possible by means of a suitable mechanical device. A. B. MANNING.

**Operation of carbonising chambers.** N.V. SILICA EN OVENBOUW MIJ., and W. HIBY (B.P. 349,459, 22.2.30).—Water or steam is introduced into the upper part of a carbonising chamber, *e.g.*, a coke oven, towards the end of the carbonising period, and flows down through the charge, the water-gas produced being withdrawn through apertures in the bottom or in the walls adjacent to the bottom of the chamber. The water or steam may be introduced through steel tubes sunk into the charge from the top of the chamber. A. B. MANNING.

**Carbonisation of bituminous materials.** H. L. DOHERTY, Assee. of H. O. LOEBELL (B.P. 349,937, 1.3.30. U.S., 18.3.29).—The fuel is carbonised at 450–850° in a thin, moving layer which is subjected to a gradually increasing pressure during a part or the whole of the process. The layer of fuel is held in contact with the outer surface of an internally heated rotating drum by means of a tensioned endless conveyor constructed of heat-resisting material, the whole being enclosed in a suitable retort. The outer wall of the retort may also be heated if desired. Means are provided for feeding the fuel on to the conveyor and for discharging the coke from the retort after it leaves the rotating drum. The outer surface of the drum and the inner surface of the conveyor may be so designed as to form moulds for containing the coal, which is thereby briquetted as it is carbonised. A. B. MANNING.

**Bone-char drying apparatus.** J. HAMILL and J. F. TADDIKEN (U.S.P. 1,784,626, 9.12.30. Appl., 25.6.25).—A pre-dryer for a char-reviving apparatus consists of a chamber traversed by heating flues so spaced as to divide the chamber into a series of narrow channels down which the char is passed. Fins extending from the heating flues form horizontal channels in the char through which air can be passed for the purpose of removing the evaporated moisture and gaseous impurities. The dryer is superposed on the reactivating kiln. A. B. MANNING.

**Manufacture of combustible gas.** H. O. LOEBELL, Assr. to H. L. DOHERTY (U.S.P. 1,785,519, 16.12.30. Appl., 29.6.21. Cf. B.P. 132,488; B., 1920, 684 A).—

A mixture of coal and coke is passed continuously down a shaft in which the coal is distilled to form coal gas, and the coke produced, together with that admitted with the coal, is gasified to form blue water-gas. A comparatively long zone of high temp. is maintained in the mid-portion of the fuel column by periodically blasting air there-through, the blast gases being burned in regenerators used for preheating the air and for generating and superheating steam. During the gas-making operation,  $H_2O$  is supplied to the bottom of the fuel column to quench the coke and ash and thereby produce steam, which passes up through the high-temp. zone where it joins the steam from the regenerators to form gas. The water-gas so formed passes up through the column of fuel, its sensible heat being utilised in carbonising the coal, and the mixed gas is withdrawn from the upper part of the shaft. A. B. MANNING.

**Manufacture of carburetted water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of J. A. PERRY (B.P. 350,866, 20.10.30. U.S., 20.11.29).—The fuel bed of a water-gas plant is upwardly air-blasted and the blow gases are burned in the carburettor and superheater. During the subsequent up-run with steam the water-gas produced is partly carburetted in the generator by spraying the fuel bed with an oil which leaves a relatively large quantity of coke when cracked, carburetting being completed with a low-coke oil supplied in the ordinary way to the carburettor. The up-run may be followed by a down-run with steam passed backwards through the set as a whole. A. B. MANNING.

**Water-gas process.** T. W. STONE, Assr. to WESTERN GAS CONSTRUCTION Co. (U.S.P. 1,784,765, 9.12.30. Appl., 22.11.23).—In a water-gas plant the carburettor and superheater are each divided into two compartments, and the process is carried out in the following cycle: the fuel bed is air-blasted and the blow gases are burned in parallel streams in both compartments of the carburettor and superheater; steam is then passed successively through a compartment of the superheater, a compartment of the carburettor, and upwardly through the fuel bed, the water-gas produced being passed through the other compartments of the carburettor and superheater in succession, and carburetted therein; the steam flow is then reversed, *i.e.*, passed through the compartments of the carburettor and superheater in which the water-gas was carburetted in the preceding step, and thence down through the fuel bed, the water-gas produced being carburetted in the other compartments of the carburettor and superheater. A. B. MANNING.

**Removal of carbon disulphide from gas.** W. J. KLAIBER, Assr. to KOPPERS Co. (U.S.P. 1,785,415, 16.12.30. Appl., 22.4.27).—The gas is scrubbed with an oil which will absorb both  $C_{10}H_8$  and  $CS_2$ , and the latter is extracted from the circulating oil by treating it with a solution of an alkaline sulphide. A. B. MANNING.

**Obtaining potassium and/or sodium ferrocyanide in the purification of coal gas.** R. BRANDT (B.P. 349,692, 24.3.30).—Coal gas freed from tar and  $NH_3$  is washed on the countercurrent principle with an alkaline suspension of Fe compounds, containing more than 25% of alkali in excess of that required for the formation of ferrocyanides, the excess alkali, which is



converted into carbonates and/or bicarbonates, being used again in making up fresh quantities of washing liquor.

A. B. MANNING.

**Refining of crude oils and the like.** S. T. HENDERSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 350,494, 12.3.30).—Volatile hydrocarbon oils, adapted for use as motor fuels, *e.g.*, crude benzol, are refined by treatment in the liquid phase with nascent H, *e.g.*, by refluxing with Zn dust and dil.  $\text{H}_2\text{SO}_4$ , followed by treatment in known manner with  $\text{H}_2\text{SO}_4$  or other refining agent.

A. B. MANNING.

**Destructive hydrogenation of carbonaceous materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,892, 6.3.30. Cf. B.P. 340,470; B., 1931, 330).—Residues obtained in the industrial working up of crude heavy hydrocarbons with alkalis are subjected to destructive hydrogenation in the presence or absence of catalysts.

A. B. MANNING.

**Manufacture of hydrocarbons rich in hydrogen from tars or like carbonaceous materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,588, 23.1.30).—The hydrogenation of tars etc. under pressure is carried out, without appreciable cracking of the initial hydrocarbons and without deterioration of the catalyst, by diluting the tars etc. with 20–100% of tetralin or other suitable polynuclear hydrogenated aromatic hydrocarbon as diluent.

A. B. MANNING.

**Manufacture of valuable products from carbonaceous materials wholly or mainly consisting of hydrocarbons of aromatic nature.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 349,470, 26.2.30).—The initial materials, *e.g.*, Edeleanu extracts, are treated with  $\text{H}_2$  under pressure and at raised temp. in the presence of catalysts consisting of Mo compounds, preferably the oxide, adsorbed on activated carbon (cf. B.P. 331,199; B., 1930, 940).  $\text{C}_{10}\text{H}_8$  and its homologues are readily hydrogenated under these conditions without cracking.

A. B. MANNING.

**Conversion of hydrocarbons having a high b.p. into hydrocarbons having a lower b.p. by destructive hydrogenation.** GAS LIGHT & COKE CO., and R. H. GRIFFITH (B.P. 349,991, 25.1., 8.3., and 29.3.30).—The process is carried out in the presence of a Mo catalyst activated by the addition of a promoter, *e.g.*, Si, B, Li, P, Ca, or compound thereof, the proportion of promoter to catalyst being chosen to correspond with the peak of the catalyst-activity curve. The curves with Si or B as promoter may exhibit two marked peaks. The catalyst and promoter are brought into intimate contact, *e.g.*, by mixing a solution or colloidal suspension of the latter into a paste with the finely-ground Mo compound, pressing the paste through a die, and drying the threads so formed.

A. B. MANNING.

**Dehydrogenation, polymerisation, and condensation of hydrocarbons with the aid of a catalyst.** GAS LIGHT & COKE CO., and R. H. GRIFFITH (B.P. 349,444, 25.1.30).—The catalyst consists of a metal, *e.g.*, Ti, W, or Cr, or compound thereof, activated by the addition of a suitable promoter, *e.g.*, Al, Zn, B, Mo, etc., or a compound thereof, the ratio of promoter to catalyst

being selected to correspond with the marked peak on the catalyst-activity curve.

A. B. MANNING.

**Production of low-boiling oils from higher-boiling oils.** STANDARD OIL DEVELOPMENT CO., ASSEES, of W. C. ASBURY (B.P. 348,889, 20.5.30. U.S., 4.6.29).—Hydrogenation is carried out in a two-stage process. Heavy oil is first hydrogenated in the presence of a catalyst at 100–200 atm. and 400–510° in the vapour phase. The products are fractionated and the portion of the condensate which is too heavy to use as motor fuel is further hydrogenated in the vapour phase at 455–565° and 25 atm. The product of this hydrogenation is useful for blending purposes.

T. A. SMITH.

**Forming hydrocarbon compounds [during reduction of metal oxides].** W. H. SMITH (U.S.P. 1,775,700, 16.9.30. Appl., 28.10.26).—A fixed gas high in  $\text{CH}_4$  is produced during the reduction of Fe ore, or similar metallic oxides, by adding to the ore a hydrocarbonaceous reducing agent and passing the mixture through a retort in which it is fed through a preheating zone in the presence of a continuously moving catalyst which catalyses the breaking up of the hydrocarbon. The liberated C is utilised in an ore-reducing zone at a temp. below the m.p. of the ore to promote the formation of spongy Fe. The  $\text{H}_2$  and some of the C are subjected to a relatively low temp. and a slight pressure in the presence of a catalyst which catalyses the formation of  $\text{CH}_4$ , which is removed at a point above the reducing zone.

H. S. GARLICK.

**Production of hydrogen, carbon monoxide-hydrogen mixtures, or nitrogen-hydrogen mixtures [from hydrocarbon gases].** M. D. BONE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,471, 26.2.30).—Hydrocarbon gases are burned with an amount of  $\text{O}_2$  or air insufficient for complete combustion, but sufficient to avoid the formation of C, and the resulting gases are passed over or through a mass of C at a temp. high enough to convert the steam in the gases into water-gas. The temp. of the C is maintained at the necessary level by utilising the heat generated in the primary combustion. Steam may be added to the initial gases before or after their combustion.

A. B. MANNING.

**Improvement of hydrocarbons to be used as fuel for internal-combustion engines.** L. MELLERSH-JACKSON. From H/H OIL CO. A./S. (B.P. 350,440, 7.3.30).—To the hydrocarbon fuel is added a small proportion of a product obtained by treating an aromatic hydrocarbon, *e.g.*,  $\text{C}_6\text{H}_6$  or PhMe, successively with  $\text{HNO}_3$  (*d* 1.41) and  $\text{H}_2\text{SO}_4$  (*d* 1.84). The mixture is then subjected to the action of  $\text{O}_2$  or  $\text{O}_3$ , preferably under pressure (1.5–2.5 atm.).

A. B. MANNING.

**[Oil] dehydrator having horizontal revolving electrodes.** J. T. WORTHINGTON, ASSR. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 1,783,595, 2.12.30. Appl., 11.11.26).—Within a horizontal shell are disposed a stationary, foraminous, cylindrical, live electrode and within that a rotating, foraminous, earthed electrode supported on a hollow shaft through which the material, *e.g.*, oil-water emulsion, is supplied to spraying apertures. The shell also acts as an earthed electrode,

and the water, after agglomeration by the electricity, settles to the bottom, the oil being drawn off in a pure state through a dome.  
B. M. VENABLES.

**Cracking oil.** W. BRINK and G. L. HESS, Assrs. to GEN. OIL PRODUCTS Co. (U.S.P. 1,783,697, 2.12.30. Appl., 9.11.27).—Oil is charged into a still near the bottom, whence it flows through a cycling drum and a series of pipes in parallel, inclined at an angle of 45°, and placed in a furnace. These pipes are continued outside the furnace and carry the oil into the still near the top. The vapours pass off through separating drums to a condenser and the oil flows down the still and is recirculated. Provision is made for the removal of the carbonaceous residues.  
D. K. MOORE.

**[Cracking] treatment of oils.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,783,229, 2.12.30. Appl., 11.2.22. Renewed 14.9.28).—Raw oil and reflux from a condenser enter the bottom of the annular space of a still consisting of a cylindrical vessel inside a larger one, and flow through pipes from the top of this space into the lower part of the inner vessel. The latter is connected to the condenser and is fitted with a run-off pipe.  
D. K. MOORE.

**Cracking of petroleum oil.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,783,228, 2.12.30. Appl., 8.10.20. Renewed 19.11.29).—Oil is partly cracked in the first of a series of stills each of which is connected to a condenser. The uncracked condensate, being more difficult to crack, flows into the second still maintained at a higher temp. than the first. The uncracked condensate from the second condenser flows into the third still maintained at a yet higher temp. The uncondensed vapour from the third condenser pass into the second and those from the latter into the first, whilst those from the first are condensed in another condenser. The formation of unsaturated compounds is reduced since no cracked product is submitted to a temp. above that necessary for its formation.  
D. K. MOORE.

**Conversion of petroleum oil.** G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,783,231, 2.12.30. Appl., 1.12.22. Renewed 5.2.29).—Oil is heated to a suitable temp. in a furnace and then passed into a chamber from which the vapour passes through dephlegmators to a condenser. The uncondensed vapour is passed into a tower containing absorbent material, *e.g.*, charcoal, where the light hydrocarbons are absorbed. They are subsequently recovered by passing hot flue gas into the tower.  
D. K. MOORE.

**Alkaline treatment of petroleum vapours.** R. C. WHEELER and P. W. PRUTZMAN (U.S.P. 1,784,262, 9.12.30. Appl., 24.6.27).—Petroleum vapour, usually mixed with steam, is washed with NaOH solution of such concentration that it boils at the same temp. as the incoming vapour, in a bubbler type of scrubber. The Na salts of the phenolic and naphthenic acids form a layer on the unacted-on NaOH at the bottom of the scrubber and are separated; the NaOH is recirculated.  
D. K. MOORE.

**Separation of fatty or wax-like bodies from their solutions.** W. W. TRIGGS. From DEUTS. GASOLIN

A.-G. (B.P. 350,388, 6.3.30).—Wax-like substances which tend to crystallise badly or not at all, *e.g.*, paraffin wax, are separated from their solutions by adding to the latter while hot a substance which readily crystallises and is sol. in the same solvent, *e.g.*,  $C_{10}H_8$ , and then precipitating the two solids together by cooling the solution. The  $C_{10}H_8$  (etc.) is separated from the wax by steam-distillation.  
A. B. MANNING.

**Treatment of aqueous bituminous dispersions [for road construction].** H. D. ELKINGTON. From FLINTKOTE Co. (B.P. 350,040, 11.3.30).—A substance which will react with the  $CO_2$  of the air to form a coagulating agent, but itself is not a coagulating agent, *e.g.*,  $Na_3AlO_3$  or Na zincate, is incorporated with the emulsion.  
A. B. MANNING.

**Liquids used for lubricating and hydraulic purposes.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 348,642, 10.2.30).—Liquids for transferring energy are produced by mixing lubricants with ethers (excluding purely aromatic ethers) which meet below 0° and boil above 50°. A mixture of castor oil with its own wt. of ethylene glycol monoethyl ether is suitable. The change of viscosity with temp. is less with the mixture than with pure oils.  
T. A. SMITH.

**Production of lubricants.** STANDARD OIL DEVELOPMENT Co., Assecs. of R. P. RUSSELL and G. H. B. DAVIS (B.P. 348,834, 11.4.30. U.S., 17.5.29).—Heavy oil is freed from asphalt and similar impurities by vacuum distillation. The distillate is then hydrogenated with or without pretreatment with  $H_2SO_4$ . The hydrogenation is carried out at pressures above 100 atm. and at 370–425°. The product is distilled. It gives lower Conradson C test and the viscosity shows less change with temp. than does untreated vacuum-distilled oil.  
T. A. SMITH.

**Motor fuels.** H. WADE. From STANDARD OIL Co. (B.P. 350,438, 5.3.30).—Cracked hydrocarbon motor fuels which tend to deteriorate in antiknock value and to develop gum on storage are stabilised by the addition of less than 0.05% of a substituted aminophenol, *e.g.*, methyl- or benzyl-aminophenol.  
A. B. MANNING.

**Washing apparatus for coal and the like.** A. WITZAL, A. G. RABEL, and H. F. STEVENS (B.P. 352,152, 9.5.30).—See U.S.P. 1,760,293; B., 1931, 8.

**Pulverised-fuel furnaces [for locomotives].** STUG KOHLENSTAUBFEUERUNG PATENTVERWERTUNG G.M.B.H. (B.P. 352,340, 4.10.30. Ger., 9.10.29).

**Predrying of fuel during its delivery to furnace grates.** VESUVIO FEUERUNGSBAU GES.M.B.H. (B.P. 352,358, 4.11.30).

**[Machine for] briquetting of wood waste and other pulverulent materials.** C. J. GOUSPEYRE (B.P. 352,289, 7.8.30).

**Acetylene generators.** C. S. MILNE (B.P. 351,646, 30.4.30).

**Gas burners.** W. R. PAIGE (B.P. 352,121, 17.4.30).

**Heating material to high temps. Separation of dry materials. Temp. control in pulverising mills. Condenser for mixed vapours. Gas-treating apparatus. Heat exchangers. Gas analysing**

apparatus.—See I. Softening of filaments.—See VI. Adsorbent for  $C_6H_6$  vapours etc.—See VII. Coated Fe pipes. Soldering fluxes.—See X. Oleaginous compositions. Detergents.—See XII. Insecticides.—See XVI.

### III.—ORGANIC INTERMEDIATES.

Reaction between chlorine gas and acetylene gas, and catalytic preparation of tetrachloroethane. K. FUKAGAWA (Proc. World Eng. Congr., 1929, 31, 387—395).— $Cl_2$  and  $C_2H_2$  combine to give tetrachloroethane without explosion in presence of Al or Fe filings or, better, an Fe—Al—Si alloy. The reaction is of the 1st order in relation to  $C_2H_2$ . C. HOLLINS.

Cuprene (carbene), the commercially most important product of pyrogenic acetylene condensation. W. HERZOG (Chem.-Ztg., 1931, 55, 461—462, 478—479).—A review of the chemistry, technical prep. methods, and commercial applications of cuprene. C. HOLLINS.

Partial oxidation of methane in the presence of oxides of nitrogen. D. F. SMITH and R. T. MILNER (Ind. Eng. Chem., 1931, 23, 357—360).—The effect of adding small quantities of  $HNO_3$  vapour,  $N_2O_4$ , and NO in the oxidation of  $CH_4$  shows that, under the various conditions tried, the production of  $CH_2O$  would not be commercially feasible. Mixtures of  $CH_4$ ,  $O_2$ , and oxides of N were passed through quartz or pyrex glass tubes at temp. of 500—700° and the amounts of  $CH_2O$ , CO, and  $CO_2$  produced were determined. Tabulated results show that a max. of 3.7% of the  $CH_4$  was converted into  $CH_2O$ , the greater part of the changed  $CH_4$  being converted into CO. A possible mechanism for the reaction is suggested. T. A. SMITH.

Nitration of chlorobenzene. Y. UYEDA (Proc. World Eng. Congr., 1929, 31, 93—101).—In the prep. of 1-chloro-2:4-dinitrobenzene 95—97% yields are obtained by nitrating initially below 10°, rising to 50—60°, to give mainly *p*-chloronitrobenzene, and then with more nitrating acid at 110—120°. Excess  $HNO_3$ , good agitation, and a pure starting material are required. C. HOLLINS.

Constitution of Ciba naphthol RP. H. LEDERER (Textilber., 1931, 12, 461—462).—Ciba naphthol RP (S.C.I.) is a new naphthol for use (as naphthol AS) in producing fast insol. azo colours on cotton. It is 95% sol. in  $COMe_2$  (the residue, 5%, consists of  $Na_2SO_4$ ) and yields yellow needles, m.p. 163°, from  $C_6H_6$ ; it is phenyl  $\alpha$ -4-hydroxynaphthyl ketone and can be readily ethylated with NaOH and  $Et_2SO_4$  to form 4-ethoxynaphthophenone, m.p. 73°. A. J. HALL.

By-product fermentation gas.—See XVIII.

#### PATENTS.

Manufacture of dichloroethylene [from  $\alpha\alpha\beta$ -trichloroethane]. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 348,346, 17.4.30. Ger., 20.4.29).— $\alpha\alpha\beta$ -Trichloroethane is passed rapidly over a heated surface, e.g., pumice, impregnated with catalyst ( $CuCl_2$ ,  $BaCl_2$ , Fe salts) if desired, at 300—500°. *s*-Dichloroethylene usually predominate in the product. C. HOLLINS.

Manufacture of alkylene cyanohydrins. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,134, 31.1. and 8.5.30).—An alkylene oxide is treated at 10—30° with alkali or alkaline-earth cyanides dispersed in a hydroxylic solvent. The cyanides may be replaced in part by HCN, or HCN and alkylene oxide may be used alternately; in these cases hydroxides may replace the cyanides. An aq. solution of ethylene oxide, stirred into aq.  $Ca(CN)_2$ , gives 92% of ethylene cyanohydrin, b.p. 120—122°/20 mm.,  $CaO$  being precipitated. Propylene (b.p. 94°/8 mm.) and  $\alpha$ -butylene (b.p. 113—115°/20 mm.) cyanohydrins,  $\beta$ - $\gamma$ -dihydroxybutyronitrile (from glycide), and  $\gamma$ -chloro- $\beta$ -hydroxybutyronitrile, b.p. 124°/10 mm. (from epichlorohydrin), are similarly prepared. C. HOLLINS.

Separation of anhydrous organic [aliphatic] acids from their aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 348,282, 14.3.30. Fr., 19.3.29. Addn. to B.P. 296,974; B., 1929, 349).—In applying the process of the prior patent to acids other than  $AcOH$ , a solvent which has b.p. higher than that of the acid and gives no azeotropic mixture with the anhyd. acid, and an entraining liquid which forms with  $H_2O$  an azeotropic mixture of lower b.p. than the solvent- $H_2O$  azeotrope, are used. For  $EtCO_2H$  the solvent may be amyl propionate, the entrainer  $Et$  propionate, and the accessory liquid (if any) a petroleum of b.p. 138—140°. For formic acid amyl formate may be the solvent, propyl formate the entrainer, and the accessory liquid (if any) *n*-heptane. C. HOLLINS.

Manufacture of amino-aldehydes. C. MANNICH (B.P. 348,382, 14.5.30).—An aliphatic or hydroaromatic aldehyde is condensed with  $CH_2O$  and a primary or secondary aliphatic or cyclic amine hydrochloride in boiling  $EtOH$ ; the aldehyde may be generated *in situ*, e.g., from an acetal.  $\beta$ -Dimethylamino- $\alpha$ -dimethylpropaldehyde, b.p. 145° or 82°/88 mm. (hydrochloride, m.p. 152—153°), and the corresponding methylamino- (reduced to alcohol, m.p. 54°, b.p. 85°/12 mm.), diethylamino- (b.p. 175—177°), and piperidino- (b.p. 95°/12 mm.; hydrochloride, m.p. 164°) compounds are obtained from isobutaldehyde;  $\beta$ -dimethylamino- $\alpha$ -isopropylpropaldehyde, b.p. 66—68°/12 mm. (reduced to alcohol, b.p. 85—86°/15 mm.), from isovaleraldehyde; 1-(diethylaminomethyl)- and 1-(*N*-piperidinomethyl)-hexahydrobenzaldehydes, b.p. 120—122°/12 mm. and 141—142°/12 mm. (hydrochloride, m.p. 165°), respectively, from hexahydrobenzaldehyde. C. HOLLINS.

Regeneration of non-aqueous solvents, such as those employed in the dry-cleaning industry. W. Y. AGNEW (B.P. 350,904, 10.3.30).—The liquid is treated with < 0.05% of, e.g., conc.  $H_2SO_4$  or  $H_3PO_4$  or HCl gas, flocculated impurities are removed, and the liquid is neutralised, e.g., by filtration through  $CaO$  or a mixture of fuller's earth and NaOH or by treatment with anhyd.  $NH_3$ . Residual solvent is recovered from the flocculated material by steam-distillation. L. A. COLES.

Manufacture of alkali salts of organic acids [from alcohols and alkali]. IMPERIAL CHEM. INDUSTRIES, LTD., Assees. of E. E. REID (B.P. 348,255, 27.2.30. U.S., 28.2.29).—An alcohol is treated with aq. caustic

alkali at 300°/above 100 atm., preferably for less than 4 hr. Examples are NaOAc from EtOH, EtCO<sub>2</sub>Na from Pr<sup>n</sup>OH, Pr<sup>n</sup>CO<sub>2</sub>Na from BuOH, and K octoate from octyl alcohol. C. HOLLINS.

**Manufacture of 1:3[ $\alpha\gamma$ ]-butylene glycol.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,248, 22.2.30).—Crude aldol is reduced with H<sub>2</sub>, or gases containing at least 80% H<sub>2</sub>, in presence of a catalyst (Cu on SiO<sub>2</sub> gel) initially at 30–80° and subsequently at higher temp., the liquor being maintained acid ( $p_H$  4–8) throughout. The product is substantially free from BuOH. C. HOLLINS.

**Manufacture of alkoxybenzophenone-di- and -tricarboxylic acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 348,037, 5.2.30).—An *o*-benzoylbenzoic acid carrying in the Bz group an alkoxy and one or two Me groups is oxidised with alkaline permanganate, preferably in excess. The following benzophenones are described: 4-methoxy-2:5'-dicarboxylic acid, m.p. 234–235° [from the Me ether, m.p. 187°, of 2-(6'-hydroxy-*m*-toluoyl)benzoic acid, m.p. 229–230°]; 2-methoxy-2':4-dicarboxylic acid, m.p. 249–250° [from the Me ether, m.p. 115°, of 2-(3'-hydroxy-*p*-toluoyl)benzoic acid, m.p. 211–213°]; 2-methoxy-2':5-dicarboxylic acid, m.p. 238–239° [from the Me ether, m.p. 156–158°, of 2-(4'-hydroxy-*o*-toluoyl)benzoic acid, m.p. 196–197°]; 5-chloro-2-methoxy-2':4-dicarboxylic acid, m.p. 202–204° [from the Me ether, m.p. 172–173°, of 2-(6'-chloro-3-hydroxy-*p*-toluoyl)benzoic acid, m.p. 205–207°]; 2-methoxy-2':3:5-tricarboxylic acid, m.p. 140–142° [from the Me ether, m.p. 123–126°, of 2-(2'-hydroxy-3':5'-dimethylbenzoyl)benzoic acid, m.p. 173–174°]; 3'-chloro-2-methoxy-2':5-dicarboxylic acid, m.p. 294–296° [from the Me ether, m.p. 186–187°, of 6-chloro-2-(4'-hydroxy-*m*-toluoyl)benzoic acid, m.p. 257–258°]; 3':5-dichloro-2-methoxy-2':4-dicarboxylic acid, m.p. 219–221° [from the Me ether, m.p. 198–200°, of 6-chloro-2-(2'-chloro-5'-hydroxy-*p*-toluoyl)benzoic acid, m.p. 232°]. C. HOLLINS.

**Manufacture of *N*-methyl compounds of the pyridine series.** I. G. FARBENIND. A.-G. (B.P. 348,345, 17.4.30. Ger., 19.4.29).—Pyridines are treated with MeCl below 200°, preferably under pressure and in presence of CuCl<sub>2</sub> or FeCl<sub>3</sub> and/or a diluent. Methochlorides of pyridine,  $\alpha$ - and  $\gamma$ -picolines, 6-chloro- $\alpha$ -picoline, and 2-methoxypyridine are described.

C. HOLLINS.

**Manufacture of derivatives of the thiazolanthrone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 348,241, 20.2.30).—Thiazolanthrones are oxidised with acid oxidants (H<sub>2</sub>O<sub>2</sub>, Cl<sub>2</sub>, or CrO<sub>3</sub> in AcOH), whereby the S atom is converted into a sulphone group. Sulphones from thiazolanthrone, its 3-methyl, 3-carboxy-, and 6-chloro-derivatives, and 1:5-dithiazolanthrone are described. C. HOLLINS.

**Manufacture of agents for use as wetting, cleansing, and dispersing agents.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,040, 26.10.29).—An aliphatic carboxylic acid below C<sub>10</sub> (or its halide or glyceride), containing at least 1 double linking or OH group, is esterified with a nitrogen-free alcohol

above C<sub>5</sub> (or its sulphuric ester), and sulphonated before, during, or after esterification. Examples are: 90% lactic acid, dehydrated at 180° in a current of gas, esterified with octodecyl alcohol, cyclohexanol, or benzyl alcohol, distilled at 1 mm., and sulphonated in Et<sub>2</sub>O with ClSO<sub>3</sub>H; hexahydrosalicylic acid, esterified with octodecyl alcohol at 180° in a current of N<sub>2</sub>, and sulphonated with 100% H<sub>2</sub>SO<sub>4</sub>. [Stat. ref.] C. HOLLINS.

**Apparatus for chemical reactions.**—See I. CS<sub>2</sub> from gas. **Condensation of hydrocarbons.**—See II. **Ca benzoate.**—See VII. **Soldering fluxes.**—See X.

#### IV.—DYESTUFFS.

**Azo dyes of the dinaphthyl series.** H. TAKAOKA and H. YAMAUCHI (Proc. World Eng. Congr., 1929, 31, 103–104).—Di- $\beta$ -naphthol [2:2'-dihydroxy-1:1'-dinaphthyl], obtained by heating an aq. alcoholic solution of  $\beta$ -naphthol and FeCl<sub>3</sub>, is nitrated and the resulting 6:6'-dinitro-compound is reduced with hyposulphite to diamine, which is tetrazotised and coupled with *J*-acid (red), *H*-acid (blue-violet), or naphthionic acid (orange). The Me and Et ethers of di- $\beta$ -naphthol, similarly treated, give dyes faster to soaping, but of poor tinctorial power. C. HOLLINS.

**Fastness of dyestuffs in ultra-violet light.** IV. G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 210–220).—Quant. measurements of the fastness of dyes to ultra-violet light (cf. B., 1931, 195) may be made by means of Pulfrich's step-photometer which, although not so accurate as the photoelectric-cell spectrophotometer, gives sufficiently exact results. Dyed filter paper is examined before and after exposure to the rays, the particular one of the three spectral filters, L1 (field of transparency 600–800  $\mu$ ), L2 (510–570  $\mu$ ), and L3 (430–485  $\mu$ ), corresponding most nearly with the predominant colour being inserted. Black colours are examined without filter. A number of dyes have been investigated in this way. T. H. POPE.

#### PATENTS.

**Manufacture of vat dyes [of the benzanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,100, 16.12.29 and 21.5.30).— $\alpha$ -Amino-derivatives of the diphthaloylcarbazole from 1:1'-dianthraquinonylamine are condensed with 2- or 3-halogenobenzanthrones and the product is treated with alkaline or acid condensing agents. Examples are: carbazole from 4:4', 4:5', or 5:5'-diamino-1:1'-dianthraquinonylamine with 3-bromo- or 2-chloro-benzanthrone, fused with alcoholic KOH, or heated with pyridine and AlCl<sub>3</sub>; carbazole from 5-amino-4'-benzamido-1:1'-dianthraquinonylamine with 2:10-dichlorobenzanthrone (1 mol.), fused with alcoholic KOH and hydrolysed with H<sub>2</sub>SO<sub>4</sub>. The products are brown to violet-brown vat dyes. C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,236, 25.11.29. Addn to B.P. 306,874; B., 1929, 35).—The process of the prior patent is applied to products obtainable from halogenobenzanthrones and aminoaldehydoanthraquinones. Alkaline fusion of products from 9- or 11-chlorobenzanthrone

or dibrominated 4:4'-dibenzanthronyl and 1-amino-2-aldehydoanthraquinone gives grey to black vat dyes.

C. HOLLINS.

**Manufacture of vat dyes [dibenzanthrones] containing halogen.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,234, 24.10.29. Addn. to B.P. 340,262; B., 1931, 338).—Chlorobromo-dibenzanthrones and -isodibenzanthrones are prepared by chlorination or bromination of bromo- or chloro-compounds or by successive chlorination and bromination, in an org. medium ( $\text{PhNO}_2$ ,  $\text{C}_6\text{H}_3\text{Cl}_3$ ,  $\text{C}_6\text{H}_4\text{Cl}_2$ ,  $\text{Ac}_2\text{O}$ , quinoline). The products are no longer sensitive to temps. used in dyeing.

C. HOLLINS.

**Production of dyes [of the dibenzanthrone series].** C. SHAW, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 347,233, 17.10.29).—Nitrodibenzanthrone or nitroisobenzanthrone is converted into a grey to black vat dye by heating at 250–260° in molten phthalic anhydride, the latter being subsequently removed by extraction with hot  $\text{H}_2\text{O}$ .

C. HOLLINS.

**Manufacture of vat dyes of the N-dihydro-1:2:2':1'-anthraquinoneazine [indanthrone] series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,170, 9.1.30).—The condensation products from indanthrone and  $\text{CH}_2\text{O}$  are converted into greener vat dyes by heating, e.g., at 200–280°, alone or with inert org. or inert neutral, acid, or weakly alkaline inorg. media, e.g., in benzophenone, 27%  $\text{H}_2\text{SO}_4$ , or  $\text{H}_3\text{PO}_4$ .

C. HOLLINS.

**Production and use of benzanthrone derivatives.** S. THORNLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 348,159 and 348,160, 31.10.29).—(A) A dibenzanthronyl is treated with  $\text{NH}_2\text{OH}$  etc. and  $\text{FeSO}_4$ , preferably after sulphonation, and the product is fused with alkali to give grey vat dyes. Suitable starting materials are 2:2'- and 3:3'-dibenzanthronyls and their chlorination products. (B) The products are alkylated to improve their Cl fastness.

C. HOLLINS.

**Manufacture of [stilbeneazo] dyes.** SOC. CHEM. IND. IN BASLE (B.P. 348,283, 14.3.30. Switz., 16.3.29).—4:4'-Dinitrostilbene-2:2'-disulphonic or 4:4'-dinitrodibenzyl-2:2'-disulphonic acid is condensed under pressure with an amine, e.g., an aminoazo dye, and, if desired, oxidised and/or converted into metal compound. 4:4'-Dinitrostilbene-2:2'-disulphonic acid is condensed, e.g., with metanilic acid  $\rightarrow \text{NH}_2\text{Ph}$  (orange on cotton); with metanilic acid  $\rightarrow \text{NH}_2\text{Ph}$  and metanilic acid  $\rightarrow$  cresidine (red-orange); with 4-chloro-o-aminophenol  $\rightarrow$  J-acid (red-brown); or with p-aminoacetanilide  $\rightarrow$  salicylic acid, hydrolysed (orange, red-brown on coppering).

C. HOLLINS.

**Production of [azo] colouring matters.** J. F. TURSKEI (B.P. 347,609, 28.1.30).—Aminoarylamides of hydroxynaphthoic, acetoacetic, and other hydroxy- or amino-acids capable of self-coupling, are treated in substance or on the fibre with  $\text{HNO}_2$  and then with alkali or other medium to bring about self-coupling, which may be inter- or intra-mol. 2:3-Hydroxynaphthoic m-aminoanilide gives a red pigment for dyeing. The prep. of m-aminoanilides of 2:3-hydroxynaphthoic, salicylic, m-aminobenzoic, and 1-phenyl-5-pyrazolone-3-carboxylic acids, of the 4-amino-o-toluidide

of 2:3-hydroxynaphthoic acid, and of monosalicyloylbenzidine, is described.

C. HOLLINS.

**Manufacture of vat dyes of the perylene series.** F. BENSA (B.P. 347,099, 16.12.29. Austr., 9.1.29).—Perylene-3:9-dicarboxylic chloride is condensed with aminoanthraquinones or aminoperylenequinones, e.g.,  $\beta$ -aminoanthraquinone (yellow-orange), 1:5-diaminoanthraquinone (gold-brown), 1-chloro-4-aminoanthraquinone (gold-orange), diaminoperylene-3:10-quinone (deep brown).

C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Porosity: a primary property of textiles. V. Porosity of fabrics and penetration problems.** J. SCHOFIELD (J. Soc. Dyers and Col., 1931, 47, 166–168).—The mesh porosity (due to interlacing of weft and warp yarns) of a woven fabric calc. as before (B., 1931, 152) differs substantially from the porosity calc. from weaving settings and the area of a mesh cell thereby obtained. Penetration of yarns by liquors in wet processing is much more dependent on capillary porosity (the capillary channels between adjacent individual fibres) than on mesh porosity; such penetration is very much assisted by alternate immersions and squeezings.

A. J. HALL.

**Preparation of a standard cotton cellulose.** K. KANAMARU (J. Cellulose Inst., Tokyo, 1931, 7, 149–157).—Samples of American, Egyptian, and Indian cotton were cleaned by hand-picking, extracted with EtOH and then with Et<sub>2</sub>O, and scoured with NaOH solutions under various conditions, and the purification process was followed by examination of tensile strength and elongation, Cu number, methylene-blue val., and viscosity of 1% sol. in cuprammonium. It is concluded that there are 3 types of non-cellulosic impurities in the raw cotton which cannot be easily removed and differ in the way in which they are progressively dissolved, viz., the material causing decrease of viscosity, that causing high Cu number, and that having a high methylene-blue absorption.

B. P. RIDGE.

**Cellulose ethers.** A. CAILLE (Chim. et Ind., 1931, 25, 276–285).—The differences between the amounts of water absorbed in non-saturated atm. by sulphocellulose and regenerated cellulose (B., 1928, 600) are maintained when the atm. is saturated. The degrees of swelling, as measured by Hofmeister's method, of sulphocellulose and sulphonitrocellulose are greater than those of regenerated cellulose and nitrocellulose, respectively. The  $\text{H}_2\text{SO}_4$  contents in nitrocellulose and cellulose acetate precipitated by  $\text{H}_2\text{O}$  from the residues and filtrates after ultrafiltration of their  $\text{COMe}_2$  solutions seem to show that the smaller micelles contain most  $\text{H}_2\text{SO}_4$ . Cellulose acetate in a half-saturated atm. at 15° takes up 6% of moisture. The moisture taken up at saturations up to 0.95 are given also. The methods of Barthélemy and Schwalbe for the determination of acetate in cellulose acetate are criticised and the mechanism of the washing of nitrocellulose and cellulose acetate is discussed. Fractional precipitation by  $\text{H}_2\text{O}$  of  $\text{COMe}_2$  solutions of nitrated paper containing 1.11% of combined  $\text{H}_2\text{SO}_4$  indicates that, contrary to Bréguet and others, the last fractions are richer in combined

$\text{H}_2\text{SO}_4$  than the first. Under identical conditions, the fractional precipitation of  $\text{COMe}_2$  solutions containing 1.13 and 0.17%, respectively, gives widely different results. Thus the addition of 50% of  $\text{COMe}_2$  ppts. 4.8 and 14.0 g., respectively.

A. RENFREW.

**Swelling phenomena of beech wood.** E. MÖRATH (Kolloidchem. Beih., 1931, 33, 131—178).—Experiments on the swelling of beech wood in different structural directions and its dependence on the  $\text{H}_2\text{O}$  content of the wood are described, and the results obtained are compared with the swelling of other woods. A saturation point is reached between 25 and 45%  $\text{H}_2\text{O}$ . Measurements of the diminution of resistance to pressure in a direction parallel to the fibre show that with increasing  $\text{H}_2\text{O}$  content the greatest change takes place at 25—35%  $\text{H}_2\text{O}$ . A rapid electrical method for determining  $\text{H}_2\text{O}$  in wood is described. Hygroscopicity isotherms for untreated wood at 20° and for samples heated for 2 hr. at different temp. show that an essential change in the hygroscopicity occurs in wood heated at 190°. The experiments on the variation of the  $\text{H}_2\text{O}$  content of wood with the R.H. of the atm. indicate that saturation is reached at 30%  $\text{H}_2\text{O}$ .

E. S. HEDGES.

**Bleaching of pulp.**—See VI. **X-Rays in research.** See XI. **Air conditioning.**—See XXIII.

## PATENTS.

**Production of viscose.** F. G. C. KLEIN (B.P. 350,515, 14.2.30. Ger., 14.2.29).—Cellulose is continuously treated according to the usual processes for conversion into viscose while being forwarded from one stage to the next by worm conveyors.

F. R. ENNOS.

**Preparation of artificial [viscose] silk filaments.** LUSTRAFIL, LTD., and S. W. BARKER (B.P. 347,396, 6.3.30).—Dull-lustre viscose silk is made by spinning in the usual manner solutions of viscose to which up to 0.5% of rice starch has been added in the form of a cold aq. suspension.

A. J. HALL.

**Production of [curled or crimped] artificial silk from viscose.** ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 350,863, 14.10.30. Holl., 12.11.29).—Several freshly-spun filament bundles, which are produced from viscoses of either different composition or degree of ripening, or by the use of spinning baths of different composition, temp., or immersion length so that they differ in shrinkage capability, are united to form a twisted thread.

F. R. ENNOS.

**Improvement of artificial fibres made from viscose.** HEBERLEIN & Co., A.-G. (B.P. 350,902, 10.3.30. Ger., 8.3.29. Addn. to B.P. 264,529; B., 1928, 295).—The fibres, directly after their formation or when in a finished condition, are subjected to swelling in presence of a Cu compound with alkali solution of mercerising concentration (not above 18%) at room temp. or below 0°.

F. R. ENNOS.

**Manufacture of artificial filaments, threads, ribbons, films, etc. [from cellulose derivatives].** H. DREYFUS (B.P. 346,793, 17.10.29).—Dull-lustre cellulose ester or ether threads etc. are manufactured by spinning by dry or wet processes solutions of the cellulose deriva-

tive containing natural or artificial resins, preferably those of m.p. above 250°.

A. J. HALL.

**Apparatus for manufacture of artificial filaments by the dry-spinning method.** CELLULOSE ACETATE SILK CO., LTD., and P. C. CHAUMETON (B.P. 351,091, 25.3.30).—A cell is divided longitudinally into a heating chamber *A*, which may occupy a portion only of the length of the cell, and a spinning chamber *B*. The gaseous atm. passes upwards through *A* and downwards through *B* in the same direction as the filaments to a smaller chamber, and thence, through a main common to all the cells, to the solvent-recovery plant.

F. R. ENNOS.

**Manufacture of sulphate cellulose and soda cellulose.** A. F. ZENNSTRÖM (B.P. 350,491, 6.3.30. Finland, 13.5.29).—Resinous wood shavings are treated for the recovery of the by-products in such a manner that volatilisation of the turpentine by steam and extraction of the resin by alkali are carried out in different zones of the same boiler; the hot lye saturated with resin soap is sprayed into an equivalent quantity of acid vapour or mist, and is afterwards treated with turpentine vapour or mist, the mixture being condensed rapidly and the solution of resin in turpentine decanted from the liquor and lignin. The leached shavings are washed and cooked by one of the above processes to yield cellulose.

F. R. ENNOS.

**Manufacture of nitrocellulose.** II. C. HEIDE. From E. TSCHUDIN (B.P. 351,133, 8.4.30).—Cellulose in dense form, prepared as described in B.P. 322,998 and 323,019 (B., 1930, 184) or by treatment with small quantities of  $\text{CS}_2$  and  $\text{NaOH}$  and subsequent hardening with acid, is nitrated with two acid mixtures, the proportions of  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{H}_2\text{O}$  in the first being 60:20:20 and in the second 25:66:9. The product contains up to 13.4% N.

F. R. ENNOS.

**Changing the solubility of cellulose acetate.** KODAK, LTD., Assees. of C. J. MALM (B.P. 350,417, 9.12.29. U.S., 12.12.28).—The  $\text{COMe}_2$ -insol. ester is hydrolysed to the sol. form by treatment, for not less than 4 days below 40°, with excess (7 times its wt.) of  $\text{AcOH}$  and  $\text{H}_2\text{O}$  containing not more than 0.6% of  $\text{H}_2\text{SO}_4$  or a corresponding amount of another acid catalyst.

F. R. ENNOS.

**Preparation of butyric cellulose esters.** J. LEFRANÇ, and SOC. DES BREVETS ÉTRANGERS LEFRANÇ & CIE. (B.P. 350,906, 11.3.30).—Cellulosic material is rendered permeable by treatment with glacial  $\text{AcOH}$  under vac. in presence of  $\text{BuOH}$  or  $\text{COMe}_2$  as diluent, and the  $\text{AcOH}$  retained by the material is removed by lixiviation with the diluting agent prior to esterification.

F. R. ENNOS.

**Coating of articles prepared from cellulose hydrate material.** F. G. C. KLEIN (B.P. 313,410, 10.6.29. Ger., 9.6.28).—Articles of  $\text{H}_2\text{O}$ -swollen hydrated cellulose are treated with a substance such as (a)  $\text{COMe}_2$ , to replace the  $\text{H}_2\text{O}$  in the pores and to keep them open, and to serve as solvent for the coating material to be subsequently applied ( $\text{COMe}_2$  solution of cellulose esters or ethers); (b) acetin, which is sol. in the coating solvent ( $\text{COMe}_2$ ); or (c)  $\text{COMe}_2$ , which is

removed by treatment with another solvent ( $C_6H_6$ ), the latter being the coating solvent. Coating may also be effected on the pretreated article by pressing thereon films of the coating material. F. R. ENNOS.

**[Manufacture of] stencil sheets.** S. HORII (B.P. 350,699, 22.5.30).—Fibrous material is coated with polysaccharide esters, sperm oil, and a tempering agent (fatty or hydrocarbon oils) dissolved in a suitable solvent mixture. F. R. ENNOS.

**Production of variegated textile yarns [by twisting].** J. SYKES & SONS, LTD., and E. SYKES (B.P. 352,212, 19.6.30).

**Non-aq. solvents.**—See III.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Bleaching of pulp from maritime pine-wood.** J. L. LUSSAUD (Bull. Inst. Pin, 1931, 17—21, 37).—Soda pulps have been prepared from maritime pine, employing 24—36% NaOH on the wt. of wood, digestion being for 6 hr. at 7 kg. per sq. cm. The effects of time and concentration of bleaching solution on these pulps are tabulated. T. T. POTTS.

**Dyeing of [Bemberg] cuprammonium silk and of cotton fabrics containing it.** ANON. (Rev. Gén. Mat. Col., 1931, 35, 201—204).—Bemberg silk differs from cotton and viscose silk in having the much stronger affinity for direct dyes. Curves are given showing the absorption by viscose, mercerised cotton, and Bemberg silk for a number of Diazol dyes over the temp. range 20—100°; in each case Bemberg silk has the greatest affinity at 20°, and whereas the affinities of viscose and mercerised cotton for these dyes increases rapidly from 80° to 100°, the affinity of Bemberg silk rapidly decreases. In order to secure level and well penetrated dyeings it is preferred to dye Bemberg silk at the boil; the dye bath should contain 3% of Turkey-red oil, 0.5—1.0% of soda ash, and 5—20% of Glauber's salt, and in dyeing deep shades the bath should be gradually cooled to about 60° after the first hr. Cotton goods containing Bemberg silk should be dyed at 90—100° so that equal depths of shade may be obtained on both fibres; a list of the most satisfactory dyes for this purpose is given. No instance has been found in which the fastness to light of a direct dye on Bemberg silk is less than that on cotton. Bemberg silk may be distinguished from viscose silk by immersion for 10 min. at 40° in a 0.05% solution of Wool Black N4B containing a small amount of  $Na_2SO_4$ ; Bemberg silk becomes dyed a deep grey whilst viscose silk is only slightly tinted. A. J. HALL.

**Coloured reserves under aniline black [by printing].** P. SEYDER (Bull. Soc. Ind. Mulhouse, 1931, 97, 141—143).—Clear, deep, and fast-to-rubbing pattern effects are obtained by printing fabric with a paste containing a Rapid Fast dye, e.g., Rapid Fast Scarlet LH, and finely-divided  $CaCO_3$ , steaming, then padding with an aniline-black liquor, and again steaming; the result is much better than when the coloured effects are over-printed on the fabric already padded with aniline-black as in the Prudhomme style. Multicolour effects may be obtained by simultaneously printing with vat dyes. A. J. HALL.

**Action of boiling caustic soda (kier-boiling) on insoluble azo colours on the fibre.** II. F. M. ROWE and F. H. JOWETT (J. Soc. Dyers and Col., 1931, 47, 163—166).—Previous investigations (B., 1931, 484) were extended to the 2- and 3-nitrobenzeneazo- $\beta$ -naphthols, and Para Red (the 4-derivative) was again investigated but in greater detail. In the partial destruction of Para Red (mainly by reduction to 4-aminobenzeneazo- $\beta$ -naphthol) a small quantity of *p*-nitroaniline is formed. The proportion of 4-aminobenzeneazo- $\beta$ -naphthol formed increases as the original depth of the dyeing is decreased. Para Red is decomposed to a much less extent by boiling with a 1%  $Na_2CO_3$  solution than with 0.36% NaOH, and no 4-aminobenzeneazo- $\beta$ -naphthol is formed. Similarly 3-nitrobenzeneazo- $\beta$ -naphthol is destroyed (to about the same degree as Para Red) with formation of equal quantities of 3-aminobenzeneazo- $\beta$ -naphthol and azoxybenzenedisazobis- $\beta$ -naphthol, but the proportion of pigment destroyed is dependent on the amount of cellulose-NaOH present and not on the depth of shade. Under similar conditions 2-nitrobenzeneazo- $\beta$ -naphthol is almost completely destroyed with formation of phenylazoimino- $\beta$ -naphthol oxide and a small quantity of *o*-nitroaniline. A. J. HALL.

**Use of nitrites in the nitrate discharge of indigo.** L. PAULUS (Bull. Soc. Ind. Mulhouse, 1931, 97, 216).—Priority for the discovery of the catalytic effect of nitrites as described by Vosnessensky (B., 1928, 478) is given to Freiberger (B., 1913, 696). A. J. HALL.

**Iodine mercerisation test.** W. F. A. ERMEN (J. Soc. Dyers and Col., 1931, 47, 161—163).—A modification of the Lange iodine test consists in immersing similar pieces of mercerised and non-mercerised cotton fabrics for a few minutes in a 6% solution of I in  $H_2O$ , saturated with KI, then washing the pieces with cold  $H_2O$  until the non-mercerised piece is colourless, and plunging them into a dil. boiling solution of Indigosol Black IB; after further washing and treatment in a boiling soap solution the non-mercerised piece remains almost white, whilst the mercerised piece is permanently coloured blue or black, the depth of shade being proportional to the degree of mercerisation. The test allows differentiation between cottons boiled with soda ash and with NaOH (*d* 1.01). Indigosol O4B may be used instead of Indigosol Black IB, but red and orange indigosol dyes are less satisfactory. A solution of  $AgNO_3$  may be used instead of Indigosol Black IB, whereby the blue I coloration of the mercerised fabric is replaced by a pale cream-coloured deposit of AgI, but this is only satisfactory for thin fabrics. In testing unknown samples of fabric, pre-removal of starch is essential. A. J. HALL.

**Method for determining the degree of mercerisation of cotton.** K. SCHWERTASSEK (Textilber., 1931, 12, 457—458).—0.3 g. of the dry cotton material, previously freed from starch by treatment with a starch-liquefying enzyme, is kneaded for a few min. with 1.2 c.c. of an I + KI solution (5 g. of I and 40 g. of KI in 50 c.c. of  $H_2O$ ), and the mixture transferred to 100 c.c. of a cold saturated solution of  $Na_2SO_4$  (*d* 1.18). After 1 hr. (with shaking), the I removed from the cotton by the  $Na_2SO_4$  solution is determined by titration with 0.02N- $Na_2S_2O_3$ ; from the result, taken in conjunction with a



similar blank experiment without cotton, the I absorbed by the cotton is calc. and is termed the "absorption number." Small differences in the degree of mercerisation produced by NaOH liquors ( $d > 1.09$ ) are easily detectable. The absorption numbers of raw and bleached cottons after mercerisation with liquors of  $d$  1.26, 1.16, and 1.09 were 81.6, 69.1; 66.8, 53.4; and 41.1, 38.2, respectively. A curve is given showing the absorption numbers obtained with liquors having  $d$  1.00—1.26.

A. J. HALL.

## PATENTS.

**Treatment of [iron] vessels to render them suitable for bleaching textile goods.** H. O. KAUFFMANN, and ELEKTROCHEM. WERKE MÜNCHEN A.-G. (B.P. 350,726, 18.6.30).—Fe vessels to be used in the bleaching of textiles with  $H_2O_2$  solutions are protected by applying to their inside surface a thin coat of a slurry containing free CaO, Portland cement, and preferably also MgO, allowing this partly to dry for 12—24 hr., bringing in contact a hot solution of Na silicate and  $Na_2CO_3$ , and allowing to dry completely.

A. J. HALL.

**Desulphurisation and bleaching of viscose artificial silk.** W. H. FRANCKE (B.P. 349,367, 22.7.30. Ger., 22.7.29).—The raw silk is treated at 60—70° with a bath or baths containing a solution of  $H_2O_2$  and a dil. water-glass solution.

A. J. HALL.

**Wet treatment of fabrics, particularly the cleaning and bleaching thereof.** J. MAURER (B.P. 346,528, 25.2.30. Ger., 25.2.29).—The relative motion between fabrics and a detergent liquor which is necessary in cleaning and bleaching processes is produced by imparting to the liquor oscillations exceeding 2 per sec. by means of a vibrating diaphragm (preferably operated electrically) immersed in the liquor in which the fabrics are submerged.

A. J. HALL.

**Production of insoluble azo dyes [ice colours] on cellulose esters or ethers.** I. G. FARBENIND. A.-G. (B.P. 348,269, 6.3.30. Ger., 6.3.29).—The fibre is padded at 15—50° with a weakly alkaline 2:3-hydroxynaphthoic arylamide bath to which has been added a  $H_2O$ -miscible org. solvent or swelling agent (pyridine, EtOH,  $COMe_2$ , etc.) in which the arylamide or its alkali salt is sol., the quantity added being enough only to swell the cellulose ester or ether without dissolving it. The colour is developed with a diazo solution in the usual manner.

C. HOLLINS.

**Coloration of textile materials [with sulphide and vat dyes].** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 347,682, 28.1.30).—Injury to acetate silk and other textiles by alkaline vats is prevented by maintaining the bath below 10°. Effects are thus obtained on acetate silk-cotton mixtures etc.

C. HOLLINS.

**Printing of materials containing cellulose ethers or esters.** IMPERIAL CHEM. INDUSTRIES, LTD., A. SHEPHERDSON, and L. SMITH (B.P. 351,056, 21.3.30).—Increased depth of shade is obtained in printing cellulose ethers and esters with  $H_2O$ -insol. azo and nitro-substituted diphenylamine dyes by the addition of up to 5% of a hydroxyethylamine (e.g., "triethanolamine") to the printing paste.

A. J. HALL.

**Manufacture of agents for protection of textiles and the like from mildew and other fungoid growth.**

IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, and A. STEWART (B.P. 350,642, 9.4.30).—The penetrative power and efficiency of the arylamides of salicylic acid (cf. B.P. 323,579; B., 1930, 238) as antiseptics in textile materials is increased by dispersing, before use, such substances with the aid of a wetting-out agent, e.g., the Na salt of the sulphonated condensation product of  $CH_2O$  and  $C_{10}H_8$ , whereby substantially all the particles of the arylamide are  $< 2 \mu$  in linear dimensions.

A. J. HALL.

**[Protective] treatment of animal fibre.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 347,292, 27.1.30. Ger., 6.2.29).—Wool, hair, bristles, etc. are purified and rendered resistant to attack by bacteria and fungi by treatment with a solution of  $H_2O_2$  followed by drying at 10—80° subsequent to a washing treatment sufficient to remove only a part of the  $H_2O_2$  from the fibres.

A. J. HALL.

**Treatment of [textile] materials to retard fading.** E. W. GEISLER, Assr. to R. H. COMEY BROOKLYN Co., INC. (U.S.P. 1,783,114, 25.11.30. Appl., 16.11.26).—White or coloured straw, straw braid, and other vegetable fibrous material, particularly that used in the manufacture of hats, is impregnated with a 5% solution of  $Na_2S_2O_3$  and then dried, whereby its fastness to light is much increased.

A. J. HALL.

**["Moiré"] treatment of [cellulose acetate] fabrics.** A. MILHOMME (B.P. 346,561, 3.4.30. U.S., 13.4.29).—In the production of moiré scratch patterns in such fabrics, the fabric is moistened before scratching the pattern thereon and is thus protected from damage.

A. J. HALL.

**Treatment of natural and artificial cellulosic fibres with alkali.** CHEM. FABR. VORM. SANDOZ (B.P. 350,018, 7.3.30. Addn. to B.P. 279,784; B., 1928, 228).—The wetting-out power in alkalis of the mixtures of phenols with hydrogenated aromatic compounds described previously is much increased by the addition of aliphatic, aliphatic-aromatic, or ketone alcohols, such as  $Pr^sOH$ ,  $Bu^sOH$ ,  $Bu^tOH$ , amyl, benzyl, or diacetone alcohol.

A. J. HALL.

**Treatment of textile materials.** BRIT. CELANESE, LTD. (B.P. 346,511, 15.2.30. U.S., 25.2.29).—Materials consisting of cellulose or cellulose derivatives are coloured, delustred, and made less liable to slip, ladder, or split by impregnation with a dispersion containing rubber latex and one or more insol. substances such as  $ZnO$ ,  $Pb_3O_4$ ,  $Al(OH)_3$ ,  $ZnS$ , and graphite, and preferably also a swelling agent (for cellulose acetate silk) such as a thiocyanate or  $COMe_2$ , followed by drying. Fast colouring of the cellulose materials may be obtained simultaneously by adding suitable insol. pigments to the dispersion used for impregnation.

A. J. HALL.

**Manufacture of rubberised materials comprising organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 347,422, 25.3.30. U.S., 30.3.29).—A firmly adherent film of rubber is applied to cellulose acetate etc. fabric by coating one or both sides with a solution of rubber containing a vulcanising agent, e.g., S, and an accelerator, and afterwards vulcanising at a temp. of 100° or above, but below that at which the fabric is damaged by heat. Vulcanising may be effected

satisfactorily with  $\text{SnCl}_2$ . Rubberised cellulose acetate fabrics have a decreased liability to crease.

A. J. HALL.

**Waterproofing of materials.** W. A. BRIDGEMAN, Assr. to WILBUR WHITE CHEM. CO. (U.S.P. 1,785,434, 16.12.30. Appl., 17.4.28).—The cloth is impregnated at about  $40^\circ$  with an emulsion of a solid fat (e.g., tallow, 6%), in a 0.5–1.5% soap solution (adjusted to  $p_H$  7.6) containing 0.5% of  $\text{PhOH}$ , and is then treated with a 3% aq. solution of  $\text{Al}_2(\text{SO}_4)_3$ . E. LEWKOWITSCH.

**[Softening] treatment of filaments or yarns composed wholly or partly of organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 351,084, 25.3.30. U.S., 25.3.29).—The pliability and softness of cellulose acetate etc. yarn is improved so that it can be satisfactorily used in knitting fabrics containing 60 or more courses per in., by impregnation with a mixture of lubricating and softening agents dispersed in a suitable solvent. Suitable lubricants include vegetable and animal oils (e.g., castor, olive, and neatsfoot oils) and petroleum oils; softening agents include  $\text{EtOH}$ ,  $\text{BuOH}$ ,  $\text{PhMe}$ , or xylene. Usually, equal parts of lubricant, softener, and solvent are used.

A. J. HALL.

**Dyeing machines. Dye beams.** J. T. LANCASTER. (B.P. 351,593—4, 4.4.30).

**Regeneration of solvents. Wetting etc. agents.**—See III. **Benzanthrone derivatives.**—See IV. **Cleaning compositions. Wetting-out agents.**—See XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Production of thermo-phosphates from Khibinsk apatite.** S. I. VOLFKOVICH and S. S. PERELMAN (Udobr. Urozhai, 1930, 2, 570—580).—Flotation afforded a product containing 40.5%  $\text{P}_2\text{O}_5$ ; this was heated for 30 min. at  $1200^\circ$  with 45% of  $\text{Na}_2\text{CO}_3$ . Optimal conditions for other samples (27.12, 19.47%  $\text{P}_2\text{O}_5$ ) were also determined. Addition of  $\text{SiO}_2$  (10%) or  $\text{CaCO}_3$  slightly increased the amount of decomp. The fertiliser value of thermo-phosphate is equal to that of Thomas slag, but slightly inferior to that of superphosphate.

CHEMICAL ABSTRACTS.

**Activated bleaching clays.** O. BURGHARDT (Ind. Eng. Chem., 1931, 23, 800—802).—A typical German clay, "Isartone," suitable for activation with acid contains 52.9%  $\text{SiO}_2$ , 16.3%  $\text{Al}_2\text{O}_3$ , and loses 11.4% on ignition. If the combined  $\text{H}_2\text{O}$  is removed, the bleaching power of the treated product is lost, but other clays of similar composition may yet have no bleaching value. The clay is reduced to slime and boiled for 2–3 hr. with 23.30% of  $\text{HCl}$  (on the wt. of dry clay). The proportion of acid required varies with each clay. The acid sludge is filter-pressed and washed to neutrality. Pine wood is used for the apparatus required for these processes. The treated clay is dried in a rotary kiln and milled. The finished product is tested for bleaching power with linseed or soya-bean oil.

C. IRWIN.

**Preparation of alums by action of oxides of nitrogen on dichromates.** S. I. ORLOVA, N. N. PETIN, and A. L. SCHNEERSON (J. gen. Chem. Russ., 1931, 1,

31—38).—Chrome alums are prepared from  $\text{NO}_2$  or  $\text{NOHSO}_4$  and  $\text{K}_2\text{Cr}_2\text{O}_7$  and  $\text{H}_2\text{SO}_4$ .  $\text{HNO}_3$  is recovered as a by-product.

R. TRUSZKOWSKI.

**[Preparation of] chromium acetate [for printing].** C. SUNDER (Bull. Soc. Ind. Mulhouse, 1931, 97, 205—215).—An investigation and comparison from the viewpoints of cost and technical difficulties of methods for preparing solutions of Cr acetate (suitable for mordanting) by reduction of a dichromate as alternatives to the method of precipitation of  $\text{Cr}_2(\text{OH})_6$  and its dissolution in  $\text{AcOH}$ . About 50 kg. of glucose, 75 kg. of starch, 75 litres of  $\text{MeOH}$ , and 75 kg. of glycerin are required for reduction of the same wt. of  $\text{Na}_2\text{Cr}_2\text{O}_7$ . In the reduction of the dichromate with  $\text{MeOH}$  or  $\text{EtOH}$  a corresponding aldehyde is produced, and if this is recovered the cost of the process is much lower than that of Metzger and co-workers (B., 1931, 348) and of Richard (B., 1924, 507). A very convenient and comparatively cheap method for preparing a violet solution of Cr acetate consists in heating together  $\text{K}_2\text{Cr}_2\text{O}_7$ , 2H<sub>2</sub>O 300 pts.,  $\text{Na}_2\text{S}_2\text{O}_3$  190 pts.,  $\text{H}_2\text{SO}_4$  ( $d$  1.84) 27 pts., and glacial  $\text{AcOH}$  180 pts.

A. J. HALL.

**Analysis of cassiterite.** S. TAMARU and N. ANDŌ (Z. anal. Chem., 1931, 84, 89—98).—By heating cassiterite for 1 hr. at  $900^\circ$  in a reducing atm. with  $\text{CaCO}_3$  and C in the mol. ratio 1  $\text{SnO}_2$  : 5—7  $\text{CaO}$  : 0.2 C all the Sn is converted into  $\text{Ca}_2\text{SnO}_4$ , which is completely sol. in  $\text{HCl}$ . The cause of the incomplete attack of  $\text{KOH}$  on  $\text{SnO}_2$  at  $500^\circ$  is shown to be the formation of an irreparable film of K stannate on the cassiterite particles.

A. R. POWELL.

**Preparation of stannous sulphate.** F. C. MATHERS and H. S. ROTHROCK (Ind. Eng. Chem., 1931, 23, 831—832).— $\text{SnSO}_4$  may be prepared for use in electroplating by treating 15 pts. of Sn foil or finely-divided electro-deposited Sn in 40 pts. of 75%  $\text{H}_2\text{SO}_4$  with a final temp. of  $140^\circ$ . Sn—Pb alloys remain undissolved. Feathered Sn is converted rapidly if alternately exposed to 50%  $\text{H}_2\text{SO}_4$  and to air by tumbling, the stannic salt film being mechanically detached and the  $\text{H}_2$  polarised.

C. A. KING.

**Removal of oxygen from ferric and ferrous oxides by means of solid carbon in a vacuum.** W. BAUKLOH and R. DURRER (Arch. Eisenhüttenw., 1930—1, 4, 455—460; Stahl u. Eisen, 1931, 51, 644—655).—An intimate mixture of finely-powdered  $\text{Fe}_2\text{O}_3$  and graphite commences to evolve  $\text{CO}_2$  in vac. at  $300^\circ$  with the formation of  $\text{Fe}_3\text{O}_4$ , which is further reduced to  $\text{FeO}$  between  $400^\circ$  and  $600^\circ$ .  $\text{FeO}$  is reduced by solid C only above  $700^\circ$  at a rate which shows that the reaction proceeds from the surface inwards at the same rate at which C diffuses through Fe.

A. R. POWELL.

**Alkalimetric titration of magnesium and determination of magnesium chloride in carnallite.** A. RAUCH (Z. anal. Chem., 1931, 84, 336—347).—The method is based on the precipitation of  $\text{Mg}(\text{OH})_2$  from a neutral  $\text{MgCl}_2$  solution in 70%  $\text{EtOH}$  by addition of an excess of 0.5N- $\text{NaOH}$  free from  $\text{Na}_2\text{CO}_3$  and titration of the excess  $\text{NaOH}$  with 0.5N- $\text{HCl}$ , using thymolphthalein as indicator.  $\text{MgCl}_2$  is extracted from carnallite by grinding the salt with 96%  $\text{EtOH}$  and

shaking the suspension vigorously for 10 min.; after filtration of an aliquot part Mg is determined as above.

A. R. POWELL.

**Anhydrous liquid sulphur dioxide.** L. B. HITCHCOCK and A. K. SCRIBNER (Ind. Eng. Chem., 1931, 23, 743–749).—The use of  $\text{SO}_2$  as an operating liquid in refrigerating plant is dependent on a low moisture content ( $< 25$  p.p.m.). S is burned in a rotary burner to give an 8–15%  $\text{SO}_2$  content, which is absorbed in a series of five towers by sea-water. The absorber liquor is then stripped by means of exhaust steam from a turbo-generator, the gaseous mixture of  $\text{SO}_2$  and  $\text{H}_2\text{O}$  leaving at  $35^\circ$  under vac. The gases are compressed in stages, fractionated, and finally compressed to 120 lb. A liquid containing 10–15 p.p.m. of  $\text{H}_2\text{O}$  is obtained.

C. A. KING.

**Huillard furnaces.**—See I. Determining  $\text{H}_2$  in coal gas.—See II. Acid-resistant linings.—See IX. Solid  $\text{CO}_2$ .—See XVIII.

#### PATENTS.

**Contact sulphuric acid process.** SELDEN Co., Assocs. of A. O. JAEGER (B.P. 348,669, 16.12.29. U.S., 22.1.29).—The catalyst comprises at least one catalytically active, non-base-exchanging silicate containing  $\text{V}^{IV}$  prepared in solutions within the range between neutrality and slight acidity to litmus. [Stat. ref.]

A. R. POWELL.

**Production of concentrated gaseous hydrochloric acid.** CHEM. FABR. KALK GES.M.B.H., and II. OEHME (B.P. 351,264, 3.7.30. Ger., 22.4.30).—Gases containing HCl are brought in contact with, e.g., anhyd.  $\text{CuSO}_4$  to form  $\text{CuSO}_4 \cdot 2\text{HCl}$ , and the HCl is expelled by heat.

L. A. COLES.

**Ammonia-gas detector [for refrigerating plant].** L. A. HAVENS, JUN. (U.S.P. 1,780,483, 4.11.30. Appl., 3.10.28).—The apparatus is covered with a hood (in which escaping  $\text{NH}_3$  collects) provided at its upper end with a vertically swinging  $\text{H}_2\text{O}$  container closed at the bottom with a foraminous screen which actuates an alarm and/or closes the supply of  $\text{NH}_3$  to the system. When  $\text{NH}_3$  escapes from the plant the surface tension of the  $\text{H}_2\text{O}$  in the container is lowered, owing to absorption of  $\text{NH}_3$ , and the liquid then passes through the screen and the consequent loss in wt. of the receptacle brings the alarm device into action.

A. R. POWELL.

**Crystallisation process for recovery of borax [from Searles Lake brine].** W. E. BURKE, ASSR. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,787,356, 30.12.30. Appl., 23.5.27).—The brine is concentrated at  $110^\circ$  until it is saturated with KCl and  $\text{Na}_2\text{B}_4\text{O}_7$ , then cooled rapidly to cause the KCl to crystallise, leaving a super-saturated solution of  $\text{Na}_2\text{B}_4\text{O}_7$ , which is mixed with some of the original brine, saturated with  $\text{Na}_2\text{B}_4\text{O}_7$  but not with KCl, cooled further, and circulated upwardly through an agitated sludge of fine borax crystals. As these crystals grow they are removed from the bottom of the crystalliser and the mother-liquor is returned to the evaporators.

A. R. POWELL.

**Recovery of cyanides [from waste solutions from gold extraction].** R. R. BRYAN, ASSR. to MERRILL Co. (U.S.P. 1,787,033, 30.12.30. Appl., 2.6.26).—The solution is treated with  $\text{SO}_2$  and the mixture of  $\text{CO}_2$ ,  $\text{SO}_2$ , and

HCN evolved is passed through further quantities of the original solution to remove  $\text{SO}_2$  and  $\text{CO}_2$  and neutralise the alkalinity of the solution. The HCN is eventually absorbed in  $\text{Ca}(\text{OH})_2$  or NaOH.

A. R. POWELL.

**Production of ammonium or potassium carnallite.** KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 351,845, 26.9.30. Ger., 28.11.29).—Mother-liquors from the crystallisation of  $\text{NH}_4$  (or K) carnallite are stirred with  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{NH}_4\text{Cl}$  or KCl in correct proportions to form the desired double salt.

A. R. POWELL.

**Refrigerating composition.** O. HANNACH, ASSR. to W. KASH (U.S.P. 1,786,842, 30.12.30. Appl., 2.6.26. Ger., 21.11.25).—A mixture of 2 pts. of  $\text{NH}_4\text{Cl}$  and 3 pts. of  $\text{Na}_2\text{CO}_3$  is claimed; on dissolving this mixture in 6 pts. of  $\text{H}_2\text{O}$  a fall in temp. of  $31^\circ$  occurs.

A. R. POWELL.

**Production of potassium nitrate.** CHEMIEVERFAHREN-GES.M.B.H. (B.P. 351,271, 11.7.30. Ger., 14.8.29).—The liquor from the process, which contains  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{SO}_4$  in solution and  $\text{CaCO}_3$  in suspension, is treated with  $\text{NH}_3$  in the presence of KCl equiv. to the  $(\text{NH}_4)_2\text{SO}_4$ ; the ppt. of  $\text{K}_2\text{SO}_4$  and the  $\text{CaCO}_3$  are removed together and treated with  $\text{HNO}_3$  to yield a ppt. of  $\text{CaSO}_4 + \text{K}_2\text{SO}_4$  and a solution from which  $\text{KNO}_3$  is recovered by evaporation or cooling. The mother-liquor from the  $\text{K}_2\text{SO}_4$  and  $\text{CaCO}_3$  is cooled to crystallise and remove part of the  $\text{NH}_4\text{Cl}$ , and the liquor is then treated with  $\text{CO}_2$  and stirred with the  $\text{CaSO}_4 + \text{K}_2\text{SO}_4$  ppt. to yield liquor for re-use.

L. A. COLES.

**Solubilising of crude calcium phosphates.** J. MARGOLES (B.P. 351,026, 17.1.30).—The phosphates are treated with  $\text{HNO}_3$ , which may be mixed with another mineral acid except  $\text{H}_2\text{SO}_4$ , in the presence of an oxide or oxidising agent, e.g.,  $\text{NaNO}_3$ .

L. A. COLES.

**Treatment of borax ores. Production of calcium borate and sodium nitrate.** H. BLUMENBERG, JUN. (B.P. 352,137, 28.4.30, and Addn. B.P. 352,138, 29.4.30).—(A) The ore is leached with  $\text{H}_2\text{O}$  and the conc. borax solution so obtained is treated with  $\text{CaCl}_2$  solution to ppt.  $\text{CaB}_4\text{O}_7 \cdot 6\text{H}_2\text{O}$ . The ppt. is collected and calcined at  $300$ – $350^\circ$  to remove the  $\text{H}_2\text{O}$ . (B)  $\text{Ca}(\text{NO}_3)_2$  is used as the precipitant and the filtrate from the  $\text{CaB}_4\text{O}_7$  is conc. to recover  $\text{NaNO}_3$ .

A. R. POWELL.

**Manufacture of pure alumina by electrothermal reduction of the impurities in aluminiferous raw materials.** H. SIEGENS (U.S.P. 1,787,124, 30.12.30. Appl., 22.12.27. Ger., 1.6.26).—Clay or bauxite mixed with a carbonaceous reducing agent is charged into the electric furnace in such a way that the proportion of C in the charge is below that required to reduce all the Fe, Si, and Ti compounds at the beginning of the process, but is gradually increased with successive charges until the fused mass contains a slight excess of C. The operation is carried out in a reducing atm. and at the end of the process a charge of highly ferruginous clay (free from Ti and Si) and sufficient C to reduce the  $\text{Fe}_2\text{O}_3$  is added to provide a shower of fine Fe globules which collect suspended particles of reduced Si and Ti compounds. The  $\text{Al}_2\text{O}_3$  is tapped when it is still light grey in colour, and is then of sufficient purity for the manufacture of Al.

A. R. POWELL.

**Production of chromic acid.** M. J. UDY, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,784,950, 16.12.30. Appl., 16.12.27).—Sol. chromates are treated with a suspension of  $\text{PbSO}_4$  in  $\text{H}_2\text{O}$  and the resulting  $\text{PbCrO}_4$  is collected, washed, and decomposed with hot  $\text{H}_2\text{SO}_4$  ( $d$  1.505). The ppt. of  $\text{PbSO}_4$  is separated and returned to the first stage of the process, whilst the solution is evaporated for the recovery of  $\text{H}_2\text{CrO}_4$ . A. R. POWELL.

**Production of acid titanium sulphate.** F. VON BICHOWSKY, Assr. to TITANIA CORP. (U.S.P. 1,783,684, 2.12.30. Appl., 20.11.28).—Powdered Ti nitride or cyanonitride is stirred into 2:1  $\text{H}_2\text{SO}_4$  at  $80^\circ$  and the mixture oxidised by gradual addition of  $\text{HNO}_3$ . After filtration the liquid is evaporated in vac. until the temp. reaches  $130^\circ$ ; on cooling, a glassy solid sol. in  $\text{H}_2\text{O}$  and abs. MeOH is obtained corresponding with the formula  $\text{H}_2\text{SO}_4 \cdot \text{Ti}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$ . With  $\text{K}_2\text{SO}_4$  a white cryst. ppt. of  $4\text{K}_2\text{SO}_4 \cdot 5\text{TiOSO}_4 \cdot 2\text{TiO}_2$  is obtained. The use of  $\text{NaNO}_3$  in place of  $\text{HNO}_3$  affords needles of  $\text{NaHSO}_4 \cdot \text{Ti}(\text{SO}_4)_2 \cdot 9\text{H}_2\text{O}$ . A. R. POWELL.

**Manufacture of titanium dioxide.** METAL & THERMIT CORP., Assees. of S. J. LUBOWSKY (B.P. 351,841, 25.9.30. Ger., 28.9.29).—Finely-ground rutile is intimately mixed with half its wt. of  $\text{MgO}$  in a ball mill, the mixture is briquetted, and the briquettes are heated at  $1400$ – $1500^\circ$  until a hard semi-vitreous product is obtained. This is ground through a 150-mesh sieve and heated with conc.  $\text{H}_2\text{SO}_4$  (3 pts.) at  $90^\circ$ ; the slurry is diluted with 6 pts. of  $\text{H}_2\text{O}$  and the solution cooled to  $0^\circ$ , whereby  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  separates. The mother-liquor is further diluted and heated at  $160^\circ$  under pressure to hydrolyse the  $\text{Ti}(\text{SO}_4)_2$ , the  $\text{H}_2\text{SO}_4$  being reconc. for use again. A. R. POWELL.

**Production of calcium benzoate.** T. GOLDSCHMIDT A.-G. (B.P. 351,548, 27.3.30. Ger., 3.4.29).—Commercial phthalic anhydride is ground with  $\text{H}_2\text{O}$  and  $\text{Ca}(\text{OH})_2$  in the mol. ratio 1:1.5 and the mixture is fed continuously by means of a worm into a jacketed tubular reaction chamber provided with stirrers and heated at  $440^\circ$  in a bath of molten S. The Ca benzoate is extracted from the reaction product with hot  $\text{H}_2\text{O}$  and the residue is converted into Ca phthalate for use again.

A. R. POWELL.

**Desilicification of liquids with regeneration of the desilicifying agent.** I. G. FARBERIND. A.-G. (B.P. 351,021, 19.3.30. Ger., 21.3.29).— $\text{H}_2\text{O}$  is treated with, e.g.,  $\text{Fe}(\text{OH})_3$  or  $\text{Zn}(\text{OH})_2$  at  $p_H$  7 or above, and the hydroxides are desilicified by treatment with a dil. mineral acid having  $p_H$  6 or below. L. A. COLES.

**Increasing the base-exchanging properties [of glauconite].** O. LIEBKNECHT, Assr. to PERMUTIT Co. (U.S.P. 1,787,008, 30.12.30. Appl., 2.2.27. Ger., 4.2.26).—Glauconite is heated at  $100$ – $250^\circ$ , digested with a 10–30% solution of  $\text{Na}_2\text{SiO}_3$  at  $200^\circ$  under pressure, washed, dried, and again heated at  $100$ – $350^\circ$ .

A. R. POWELL.

**Manufacture of a [silicate] mineral adsorbent [for benzene vapours etc.].** J. T. TRAVERS, C. H. LEWIS, and O. M. URBAIN, Assrs. to OHIO SANITARY ENG. CORP. (U.S.P. 1,783,396, 2.12.30. Appl., 20.10.28).—Kaolinite is mixed with 75% of its wt. of sawdust, 1.5 times its wt. of  $\text{Na}_2\text{SiO}_3$  solution ( $d$  1.38), and 2.4

times its wt. of  $\text{H}_2\text{SO}_4$  ( $d$  1.25) and the mixture set aside until gelation occurs. The stiff gelatinous product is then dried slowly at gradually rising temp. and finally maintained at  $90^\circ$  until no further moisture is expelled. The mass is then heated at  $650^\circ$  in a reducing atm. until volatile matter is no longer evolved, whereby it is converted into a hard porous mass of high adsorptive capacity.

A. R. POWELL.

**Manufacture of dry carbon dioxide products.** DRY ICE CORP. OF AMERICA, Assees. of C. L. JONES (B.P. 346,367, 7.1.30. U.S., 21.2.29).—Liquid  $\text{CO}_2$  at  $25^\circ$  and under 1000 lb./sq. in. pressure is allowed to expand through a nozzle at the upper part of a vertical cylindrical chamber so that the liquid is cooled to a temp. below  $0^\circ$ , but above the triple-point temp. and pressure of  $\text{CO}_2$ . Surrounding the expansion nozzle is a long basket of fine-mesh gauze which serves to collect the small ice crystals formed by the freezing of the  $\text{H}_2\text{O}$  contained in the liquid. Means are provided for removing this strainer from the chamber and for melting the ice crystals and separately draining off the  $\text{H}_2\text{O}$  without removing the strainer.

A. R. POWELL.

**Device for detection of oxygen in hydrogen [in hydrogen-cooled dynamo-electric machines].** G. W. PENNEY, Assr. to WESTINGHOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,778,834, 21.10.30. Appl., 30.4.26).—Use is made of the fact that the pressure developed by a fan running at a given speed in a gas is directly proportional to the density of the gas; hence any increase in the pressure of a fan running in  $\text{H}_2$  indicates the leakage of air into the gas container. This increase of pressure serves to operate a relay or signalling device adapted to give visual or oral indication of such leakage in the case of turbo-generators cooled with  $\text{H}_2$ .

A. R. POWELL.

**Hydrogen generator [for the steam-iron process].** C. MIEDEBRODT (U.S.P. 1,777,444, 7.10.30. Appl., 27.2.29. Ger., 22.2.28).—The apparatus comprises an ore chamber above which are a series of superimposed cells arranged radially round the central shaft. The reduction gas as well as the steam used in the subsequent generation of  $\text{H}_2$  pass upwards through the central shaft, then through the cells in series so as to preheat it before it passes through the bed of ore or reduced Fe.

A. R. POWELL.

**Gas-treating apparatus.**—See I. Ferrocyanides from coal gas.  $\text{H}_2$ , CO, etc. from hydrocarbons.—See II. Treatment of sulphide Fe ores. Treatment of lead-bearing material. Cd-containing material to Cd. Mg from its compounds. Reduction of metallic compounds.—See X. Fertilisers.—See XVI. Colloidal AgI.—See XX.

## VIII.—GLASS; CERAMICS.

**Permeability to gas of refractory materials, particularly at high temperatures.** H. IMMKE and W. MIEHR (Sprechsaal, 1931, 64, 85–87, 107–109; Chem. Zentr., 1931, i, 2102).—The permeability is proportional to the gas pressure and is smaller at higher temp. Two methods of determination are described, and the application of the results to gases other than air is indicated.

A. A. ELDRIDGE.

**Manufacture and performance of slip-cast tank blocks.** P. HALLER (J. Soc. Glass Tech., 1931, 15, 83—98 r).—The prep. of a satisfactory slip demands a nice adjustment of the ratio of Na silicate, which increases fluidity, to  $\text{Na}_2\text{CO}_3$ , which increases the viscosity and stabilises the slip. There is no fixed optimum ratio. Only 0.5 in. of plaster is required as a lining to the moulds used in casting. The harder, denser, and less porous is the grog, the better it is suited for slip casting, calcined Indian sillimanite being almost ideal in these respects. Good results have been obtained with a grading: >5-mesh I.M.M. 1.2, <5 but >10-mesh 41.5, <10 but >20-mesh 24.4, <20 but >40-mesh 10.3, <40 but >80-mesh 7.7, <80-mesh 14.9%. Cast blocks are more tender as regards warping and yielding under pressure in the kiln. The porosity of cast blocks is not very much less (24—28%) than that of hand-made plastic ones (27—30) from the same clay composition. Addition of 4% of felspar may lower the porosity of the cast blocks to 18%. The cast blocks have considerably greater mechanical strength than ordinary ones, and a much more regular texture. In service, porosity was found to be of less importance than the formation of a mullitic layer on the face of the block in contact with the molten glass, and the addition of the felspar promoted this. Better service was given by the slip-cast blocks than by hand-made ones from plastic clay of the same composition.

M. PARKIN.

**Standards for testing probable behaviour of tank blocks.** W. MIEHR (J. Soc. Glass Tech., 1931, 15, 30—40 r).—The Bowmaker test (B., 1929, 815) alone was insufficient to determine probable durability of tank blocks, but was found of value in conjunction with a knowledge of (a) the solubility of the mullite-free portion, (b) the porosity, and (c) the chemical composition. The HF-solubility figures obtained by the author and his co-workers for German blocks varied from 4 to 20%, whilst those quoted by Bowmaker ranged from 18.9 to 59.8%. More definite relationships must be established before standard methods can be specified.

M. PARKIN.

**Clay mixtures for glass-melting pots. I. Resistance to thermal shock. II. Shrinkage at high temperatures.** J. H. PARTRIDGE and G. F. ADAMS (J. Soc. Glass Tech., 1931, 15, 59—76 r).—Spalling tests of specimens prepared from clay mixtures containing 20—30%  $\text{Al}_2\text{O}_3$  and having varying gradings of grog, indicated that silicious clays had less tendency to crack than aluminous ones under the same treatment. Too much fine grog increases the cracking tendency, and for any particular clay there is an optimal amount of the grog of given grading that it can carry. Prolonged firing of test pieces at 1320° showed the silicious mixtures to remain fairly const. in vol., whilst the aluminous ones decreased 12—20% over a considerable period of time (400—500 hr.). Practical results were also in favour of silicious pots, 19 of which gave one quarter of the service given by 156 aluminous ones. Proportion of grog affected strength in drying, a reduction from 50% to 40% in one case raising tensile strength from roughly 11 to 14 kg./sq. cm.

M. PARKIN.

**Experimental firing of a Scottish fireclay material.** A. T. GREEN and A. E. J. VICKERS (Trans. Ceram. Soc., 1930, 29, 169—181).—An account of the burning of a batch of 600 bricks to a time schedule in an experimental furnace fired by town's gas is given, with discussion of the results. The time schedule was devised from the following considerations. (a) Water-smoking, 0—250°: Green and Theobald (B., 1925, 501) had expressed the opinion that 17 hr. would be adequate, but to test the abs. min. 11 hr. were decided on for these experiments and the result was still satisfactory. (b) Range 400—800°, during which combined  $\text{H}_2\text{O}$  and carbonaceous matter (the latter was low) are eliminated and the kaolinite mol. is broken down: preliminary experiments with blocks 2 in. thick showed a large dark core after 4 hr. which disappeared after 8 hr.; 12 hr. was aimed at for the full-size bricks against the 30 or so commonly allowed by manufacturers. (c) Vitrification 850—1450°: it was decided to raise the temp. at the rate of 40° per hr. up to 1285°, then 15° per hr. to 1450°, and to give a final soak for 3½ hr. The total time of burning was 55 hr. The amount of  $\text{H}_2\text{O}$  vapour and of  $\text{SO}_2$  in the furnace gases was determined from time to time: the former shows three peaks; the latter was steady till the later stages, then it rose rapidly. From an examination of the bricks for porosity etc., the authors consider that a determination of the sealed pores and the crushing strength would be useful to control vitrification. Actually the temp. was raised from 450° to 850° in only 9 hr., and although the bricks were still good the authors consider that the time could not safely be further reduced.

B. M. VENABLES.

**Influence of water content of clays on their properties in drying and firing.** V. P. ZUBCHANINOV and D. I. SMIRNOV (Trans. Ceram. Res. Inst. Moscow, 1930, No. 24, 30—62).—During drying, three periods are distinguished: (a) shrinkage and loss of  $\text{H}_2\text{O}$  proceed with const. velocity, (b) the decrease in  $\text{H}_2\text{O}$  (pore  $\text{H}_2\text{O}$ ) proceeds with changing velocity while the vol. of the clay body remains const., (c) the hygroscopic  $\text{H}_2\text{O}$  is removed by heating at 110°. The quantity of shrinkage  $\text{H}_2\text{O}$ , but not that of the pore or hygroscopic  $\text{H}_2\text{O}$ , depends on the initial  $\text{H}_2\text{O}$  content. The relation between the amount of shrinkage and that of pore  $\text{H}_2\text{O}$  is characteristic for each clay. The relative content of pore  $\text{H}_2\text{O}$  in kaolins is greater than that in clays.

CHEMICAL ABSTRACTS.

**Production of tridymite bricks.** H. SALMANG and B. WENTZ (Ber. deut. Keram. Ges., 1931, 12, 1—29; Chem. Zentr., 1931, i, 2102—2103).

Acid-resistant ceramic linings.—See IX.

## PATENTS.

**Tempering of glass.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 341,218, 29.11.29. Fr., 2.9.29. Addn. to B.P. 340,108; B., 1931, 350).—Apparatus suitable for carrying out the process of air-quenching described in the prior patent is described.

M. PARKIN.

**Manufacture of unsplinterable glass.** A. V. JOHNSON (B.P. 351,257, 28.6.30).—The sheets, after

adhesion under high pressure, are subjected with their edges protected by rubber strips of U-section to light pressure, *e.g.*, by compressed air in an autoclave.

L. A. COLES.

[Clay-free] refractory mortars [for use with silica bricks]. E. LUX, Assee. of H. KOPPERS A.-G. (B.P. 351,067, 22.3.30. Ger., 25.3.29).—A mixture of fine quartzite, an org. agglutinant (tragantane), and an insol. Ca salt, *e.g.*,  $\text{CaCO}_3$ , is mixed to a paste with  $\text{H}_2\text{O}$ .

L. A. COLES.

Manufacture of artificial grinding or abrading wheels. E. A. SNOWDEN (B.P. 351,182, 10.5.30).—The wheels are built up of a centre core, which may be reinforced, and an annular outer layer, both of which contain limestone particles bonded with Portland or high-aluminous cement, the outer layer containing abrasive material (*e.g.*,  $\text{Al}_2\text{O}_3$ ,  $\text{SiC}$ ) in addition.

L. A. COLES.

Manufacture of [sand-faced] ceramic articles. J. F. JORDAN (B.P. 351,122, 3.4.30).—The moulded clay is coated with a mixture of sand containing not more than 5% Fe (sea sand) with a pigment, *e.g.*,  $\text{Cr}_2\text{O}_3$ , and is then fired.

L. A. COLES.

Purification of natural and artificial matter. [Removal of iron from ceramic materials.] C. R. B. YOUNG (U.S.P. 1,786,383, 23.12.30. Appl., 30.8.26. Ger., 11.9.25).—Coarsely-ground quartz or felspar is freed from Fe, Mn, etc. by boiling it with a solution containing 18%  $\text{H}_2\text{SO}_4$ , 2% NaCl, and 1%  $\text{H}_2\text{C}_2\text{O}_4$ .

A. R. POWELL.

Glass-bending furnaces. J. ZINGG (B.P. 351,862, 17.10.30. Ger., 13.12.29).

Tempering of glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNEY, & CIREY (B.P. 352,359, 5.11.30. Fr., 6.1.30. Addn. to B.P. 340,108).

Rolling of glass. Y. BRANCART (B.P. 352,346, 16.10.30. Belg., 18.10.29).

Power press for pressing or shaping articles from clay dust or other material. W. F., E. J., and S. H. KENT (W. KENT) (B.P. 352,236, 28.6.30).

Sealing Cu to glass.—See X. Glass for discharge tubes.—See XI.

## IX.—BUILDING MATERIALS.

Deterioration of structures in sea-water. ELEVENTH (INTERIM) REPORT OF THE COMMITTEE OF THE INST. CIVIL ENG. (Dept. Sci. Ind. Res., 1930, pp. 24).—A further inspection of painted steel plates which have now been exposed to aerial corrosion for more than 5 years showed that galvanised plates and plates with 2 or 3 coats of  $\text{Fe}_2\text{O}_3$  paint are still very good. Dehydrated coal tar with or without red lead in addition is also good, but coal-tar varnish, bitumen, and asphaltum paint are all very bad. Tarred plates completely immersed in sea-water were found to be more corroded than others at half-tide. Horizontal-retort tar was better than that from vertical retorts. The former was improved by the addition of slaked lime, the latter by paraffin wax. Tar is best applied at 200° to plates only slightly warmed. A summary of tests of roofing paint (graphite,

iron oxide, and "navy-grey") is given. Tests on wood blocks immersed in sea-water at various tropical stations showed that those treated with creosote were free from *Teredo* after 5 years' exposure. No advantage was thus obtained by adding chlorodihydrophenarsazine ("DM") to creosote, but this compound used in alcoholic solution was superior to other arsenical compounds. Fuel oil fails as a protective probably owing to poor penetration. A 2% solution of "DM" in fuel oil protected completely as far as it penetrated. Pintsch gas tar, manufactured from mineral colza, appears to penetrate and preserve effectively. Descriptions are given of repairs to concrete structures in sea-water. Iron and steel bars of varying composition have now been exposed to sea-water for 8 years. Cr- and Ni-steels are generally in the best condition. Reinforced concrete piles which have been exposed to (1) sea-water, (2) artificial sea-water of 3 times normal concentration, show little deterioration after 7 months. C. IRWIN.

Acid-resistant ceramic linings. L. KÖGEL (Chem. Fabr., 1931, 4, 285—287).—Linings for chemical plant built up of acid-resistant blocks and cement are porous to moisture. Nevertheless the corrosion of a lined Fe container filled with HCl soon stops, perhaps owing to the acid in the pores being held in place by capillary action and then neutralised. If the outer container is not liquid-tight the lining does not remedy this. Cements used consist of quartz, heavy spar, etc. made into a paste with Na silicate. The mineral grains must be completely covered with the silicate to give uniform setting. Hardening is complete when  $\text{H}_2\text{O}$  is entirely removed and all alkali is neutralised with a subsequent acid treatment. This process depends on the porosity of the blocks and is facilitated by warming to 50—60° for a few days, and  $\text{H}_2\text{O}$  must be excluded until the acid has been applied. The Höchst cements made by the I. G. Farbenind. have the special characteristic that setting proceeds even in absence of air. C. IRWIN.

Emulsifying action of asphalt fillers. A. R. EBBERTS (Roads, 1931, 9, 124—125).—Contradictions which have arisen in the correlation of grading and voids of the aggregate with the compressibility and resistance to  $\text{H}_2\text{O}$  action of paving mixtures are explained by the emulsifying action of the fillers.  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , and gypsum give asphalt-in- $\text{H}_2\text{O}$  emulsions, whilst limestone,  $\text{MgCO}_3$ , hydrated  $\text{CaO}$ ,  $\text{Mg(OH)}_2$ , and Portland cement give  $\text{H}_2\text{O}$ -in-asphalt emulsions. According to its composition slag may be in either class. Of the alkaline-earth compounds tried, only  $\text{CaSO}_4$  gave an oil-in- $\text{H}_2\text{O}$  emulsion, but emulsification was incomplete and not strictly typical. Depending on the proportion, a mixture of  $\text{SiO}_2$  and limestone may give either type of emulsion. Limestone is the best aggregate for asphaltic concrete, and there is usually enough limestone surface to overcome the action of added filler. When trouble is experienced in getting the asphalt to stick to the aggregate, the addition of  $\text{CaO}$  to the mix will overcome the difficulty. Emulsions of either type will adhere to limestone chips or pea gravel, due to their adsorptive surface. The predominance of calcareous aggregates in various proprietary emulsions accounts for the success of such products. Actual

rubbing tests under water on compressed pavement specimens showed that a limestone filler decreased the tendency of the asphalt to be dispersed and lost by drainage. When silicious aggregates are the cheapest and most easily obtained it is advised that a small amount of calcareous material be added to render them neutral.

J. A. SUGDEN.

**Tank insulation.**—See I.

#### PATENTS.

**Floor and wall coverings [of cement and leather].** J. SEJVEL (B.P. 351,521, 24.3.30).

**Grinding of materials. Mixer [for concrete].**—See I. **Bituminous emulsions.**—See II. **Vessels for bleaching.**—See VI. **Refractory mortars.**—See VIII. **Slag. Metal-clad shingles.**—See X.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Agglomeration of Siegerland [iron] ore fines by coking with coal.** W. LUYKEN and E. BIERBRAUER (Arch. Eisenhüttenw., 1930—1, 4, 505—511; Stahl u. Eisen, 1931, 51, 739—740).—Satisfactory agglomerates were obtained by coking a mixture of the fine ore (through a 5-mm. screen) with a good bituminous coal; less than 10% of the coked material passed a 40-mm. screen. With 43% of ore added to the coal 63% of the  $\text{NH}_3$  and 16% of the  $\text{C}_6\text{H}_6$  normally produced during coking were lost, whilst the calorific val. of the gas was reduced from 4800 to 4167 kg.-cal./cu. m. On the other hand, the ore coke was highly reactive and did not crush or pack in the blast furnace.

A. R. POWELL.

**Influence of certain physical and chemical factors on the working of blast furnaces.** A. PETERS (Chim. et Ind., 1931, 25, 1327—1338).—To obtain the greatest fuel economy it is desirable to increase indirect reduction of  $\text{Fe}_2\text{O}_3$  (by CO) and decrease direct reduction by solid coke. Indirect reduction removes 50—70% of the O in the  $\text{Fe}_2\text{O}_3$  in usual practice, according to the type of ore. This could, in theory, be improved by reducing the size of the pieces of ore charged, but this is not likely to be economical in practice in most cases. It may be improved by reducing the zone of combustion to a minimum, thus increasing the time of contact between the gas and the charge. Also by increasing the blast pressure in the tuyères the favourable effect of increased pressure more than compensates for the consequent reduction of time of contact. Reduction in the size of the coke is desirable as tending both to promote even gas distribution through the furnace and to reduce the oxidising zone before the tuyères. The latter purpose is also served by the use of a coke of low reactivity at below 900°. It is desirable that the coke should be weighed and not merely measured.

C. IRWIN.

**Control of the excess air in open-hearth furnaces.** A. HERBERHOLZ (Arch. Eisenhüttenw., 1930—1, 4, 461—468; Stahl u. Eisen, 1931, 51, 670—671).—Control is effected by systematically passing a definite vol. of flue gas through an automatic  $\text{O}_2$  recorder in which the decrease in vol. after catalytic combustion

of the  $\text{O}_2$  with pure  $\text{H}_2$  is recorded and the results are used for the automatic regulation of the air supply to the furnace. Tests on numerous furnaces with various types of charges have shown that min. fuel consumption with max. output is obtained when the flue gas contains 2.5—4.5%  $\text{O}_2$ , preferably about 3.8%  $\text{O}_2$ . Under these conditions the furnace has a melting capacity of 11 tons/hr. with a heat consumption of 900 kg.-cal./kg.

A. R. POWELL.

**Ternary system iron-sulphur-carbon.** R. VOGEL and G. RITZAU (Arch. Eisenhüttenw., 1930—1, 4, 549—556; Stahl u. Eisen, 1931, 51, 793—794).—In this ternary system, besides the immiscibility gap in the liquid state, there are a secondary region of immiscibility in which when part of a homogeneous liquid phase has solidified drops of a second liquid phase ( $\text{FeS}$ ) separate, and a tertiary region in which a second liquid phase separates in droplets which disappear again at lower temp. Only three cryst. constituents occur in the solid alloys, viz., binary solid solution of C in Fe,  $\text{Fe}_3\text{C}$ , and  $\text{FeS}$ . The temp. of the four-phase equilibrium liquid 1  $\rightleftharpoons$  liquid 2 +  $\gamma$  +  $\text{Fe}_3\text{C}$  is 1190° and the compositions of the liquids are (1) 95.2% Fe, 4% C, and 0.8% S, and (2) 70.25% Fe, 29.5% S, and 0.25% C. The ternary eutectic, m.p. 975°, contains 0.15% C and 31% S, and consists of  $\text{FeS}$  and a binary solid solution of C in Fe. The decomp. of the solid solution of C in Fe into ferrite and cementite and the temp. of the pearlite point of the Fe-C system are not appreciably affected by the presence of C. The droplet shape of troilite in meteoric Fe is attributed to the presence of C in the Fe.

A. R. POWELL.

**System iron-nitrogen.** W. KÖSTER (Arch. Eisenhüttenw., 1930—1, 4, 537—539; Stahl u. Eisen, 1931, 51, 740).—Micrographical investigation of the nitrified layer on Fe heated for 8 hr. at 650° in  $\text{NH}_3$  confirms the observation of Lehrer (A., 1930, 1121) that the system Fe-N contains two eutectoids (cf. also A., 1930, 996). The first eutectoid, braunite, is converted into martensite and austenite by quenching from 600°, whereas the second remains unchanged. Fe-N alloys with up to 3% N resemble closely in structure and behaviour on heat-treatment the Fe-C alloys.

A. R. POWELL.

**Ternary system iron-carbon-vanadium.** R. VOGEL and E. MARTIN (Arch. Eisenhüttenw., 1930—1, 4, 487—495; Stahl u. Eisen, 1931, 51, 715).—The  $\gamma$ -field of the Fe-C system extends only to 1.8% V in the ternary system and V raises the temp. of the magnetic transformation which can be detected in alloys containing up to 25% V. Only one V carbide,  $\text{V}_4\text{C}_3$ , but no double carbide, exists in the partial system Fe-V- $\text{V}_4\text{C}_3$ - $\text{Fe}_3\text{C}$  and a field of ternary  $\gamma$ -solid solution extends from the system Fe-C to the boundary of the system V- $\text{V}_4\text{C}_3$ . The saturated ternary  $\gamma$ -phase (3.4% C and 6.5% V) forms a ternary eutectic with  $\text{Fe}_3\text{C}$  and  $\text{V}_4\text{C}_3$  containing 81.8% Fe, 6.2% C, and 12% V, m.p. 1095°. In steels with a low V content ternary  $\gamma$  decomposes on cooling with the separation of ferrite and pearlite composed of  $\text{Fe}_3\text{C}$  and ternary  $\alpha$  with a variable content of V and C. In V-rich steels  $\gamma$  decomposes according to the composition into ferrite and (a) a ternary pearlite consisting of cementite,  $\text{V}_4\text{C}_3$ , and ferrite with 0.2% C and 0.8% V,



this phase being formed at 700°, or (b) a binary pearlite consisting of  $V_4C_3$  and ferrite with a high V and low C content. V even in small quantities retards the separation of cementite from pearlite, but the separation of  $V_4C_3$  from the ternary phases is scarcely affected even by very rapid cooling. The increase in the hardness of steel produced by addition of V is ascribed to the greater hardness of the binary ferrite containing V in the pearlite eutectoid, to the more finely-dispersed condition of decomposed pearlite, and to the separation of finely-divided, very hard  $V_4C_3$ . A. R. POWELL.

**System iron-carbon-vanadium.** H. HUGARDY (Arch. Eisenhüttenw., 1930—1, 4, 497—503; Stahl u. Eisen, 1931, 51, 592—593).—In the Fe-V system the  $\gamma$ -field extends to 1.1% V, but the addition of C to the alloys extends the  $\gamma$ -range in such a way that the max. amount of V in the  $\gamma$ -phase is given by the equation  $V = C/0.175 + 1.1$ ; this implies that the additional V is all present as  $V_4C_3$  and that the system is pseudo-binary.  $V_4C_3$  does not form a double carbide with  $Fe_3C$ , both being precipitated independently when sufficient C is present, and forms no solid solutions with  $\alpha$ - or  $\delta$ -Fe, but a limited series of solid solutions with  $\gamma$ -Fe. The A1 point of the Fe-C system occurs at 10° above the normal temp. in the presence of V, independent of the amount present, provided that the alloy contains cementite or pearlite. The hardness of quenched ternary alloys ceases to increase above a definite V content dependent on the C present, and these alloys have a lustrous, coarsely cryst. fracture which cannot be changed by heat treatment; their microstructure shows the presence of  $V_4C_3$  and ferrite only.

A. R. POWELL.

**Hardened and tempered steels.** I. Alkaline sodium picrate etching and its application to the study of the mechanism of tempering hardened steels. H. HANEMANN and A. SCHRADER. II. Mechanism of the formation of the martensite structure. H. HANEMANN, K. HERRMANN, V. HOFMANN, and A. SCHRADER. III. Carbon content of the  $\eta$ -phase. H. HANEMANN (Arch. Eisenhüttenw., 1930—1, 4, 475—477, 479—484, 485—486; Stahl u. Eisen, 1931, 51, 645, 646—647, 647—648).—I. Cementite is etched deep black by alkaline picrate solution whether present in the microstructure in a coarse form or as pearlite, sorbite, or quenched troostite. Ferrite, austenite, and the  $\eta$ -phase are not coloured by the reagent at 20°, but above 70° the last-named commences to decompose and it is then darkened by the picrate. The  $\zeta$ -phase is slowly darkened by the reagent, but can be distinguished from cementite by etching at 40° for 40 min., when the cementite becomes deep black, but  $\zeta$  is scarcely attacked. To distinguish between  $\zeta$  and  $\delta$  the steel is etched at 50—60°, when  $\zeta$  is blackened in 50 min., but  $\delta$  is darkened only above 70°. Thus suitable selection of temp. and time of etching with alkaline picrate serves to distinguish between all the constituents associated with the martensite transformations.

II. Röntgenographic and microscopical investigation of hardened steels indicates that the  $\eta$ -phase has a tetragonal lattice with the C atoms in the basal plane of the elementary cell, the structure of which slowly changes on ageing at the ordinary temp. or tempering

at 70° without any visible change in the microstructure. Tempering at 100° converts the  $\eta$ -phase into a pseudomorph of  $\alpha$ -Fe having a similar lattice structure but a different crystal form ( $\delta$ -phase). Cementite is formed only above the second transformation temp. of martensite. The transition state,  $\zeta$ , between the two transformations of martensite has a distorted  $\alpha$ -Fe lattice containing adsorbed C atoms.

III. Quenching and tempering tests on austenitic and martensitic steels indicate that the  $\eta$ -martensite needles contain about 1.1% C. A. R. POWELL.

Some diagrams registered with a self-recording dilatometer during quenching [of steel], and the mechanism of nodular troostite formation in carbon steels. S. SATÔ (Sci. Rep. Tôhoku, 1931, 20, 260—267).—A new type of recording differential dilatometer has been used in quenching experiments on a steel of eutectoid composition. The efficiency of various quenching media and of “facing” upon the surface of the steel has been estimated from the form of the dilatometric curves and from the microstructure of the steel. Particular consideration is given to the mechanism of formation of nodular troostite in quenched steel.

E. H. BUCKNALL.

**Fibrous structure of steel and its mechanical property.** K. NAGASAWA (Sci. Rep. Tôhoku, 1931, 20, 299—312).—The fibrous macrostructure may be developed by etching in 40—60% HCl. This structure is shown to be related to the original dendritic form. The mechanical properties are superior along the direction of the fibres to those measured at right angles. Annealing at temp. below 1300° has little effect in removing fibre. Cracks formed by quenching tend to lie along the fibres. E. H. BUCKNALL.

**X-Ray analysis of the cementite obtained by tempering quenched steels.** S. SEKITO (Sci. Rep. Tôhoku, 1931, 20, 313—322).—The presence of cementite in steel is indicated in X-ray spectra. By fastening a strip of Au to the steel specimen a comparison standard is obtained, and the % cementite may be measured by the relative intensity of the cementite line (113  $K\alpha$ ) to the Au line (220  $K\beta$ ). In steels of varying C content, the % cementite is proportional to the C content, given similar heat treatment. Formation of cementite in a quenched steel commences on tempering at 300° and development of the cementite lattice is complete at 550—600° with short annealings and at 350—400° with longer treatment. E. H. BUCKNALL.

**Hot-shortness of steel [produced] by [contact with] metals.** H. SCHOTTKY, K. SCHICHTL, and R. STOLLE (Arch. Eisenhüttenw., 1930—1, 4, 541—547; Stahl u. Eisen, 1931, 51, 769—770).—Metals insol. in Fe, e.g., Pb, Bi, Cd, and Ag, and metals completely sol., e.g., Ni, do not produce hot-shortness of steel when brought into contact with it at 1200°. On the other hand, hot-shortness is rapidly produced by contact with molten metals or alloys of metals which are completely miscible with molten Fe, but have only a limited range of solid solution in Fe, e.g., Sn, Zn, Sb, Al. Thus 35:65 Sn-Cu bronze produces hot-shortness at 740°, i.e., 10° above the m.p., but Sn, Sb, and Al produce hot-shortness only at 1000°. Ce and Si produce hot-short-

ness of high-C steels at 1200°, owing to the formation of ternary eutectics.

A. R. POWELL.

**Properties of "degassed" and "gassy" cast steel.** W. OERTEL and A. SCHEPERS (*Stahl u. Eisen*, 1931, **51**, 710—715).—The properties and structure of steel containing 0.07% C, 0.3% Mn, 0.02—0.03% P, and 0.024—0.035% S cast without addition of Si or Al to the ladle have been compared with those of the same steel after treatment with Si and Al to remove gas. Segregation of S, P, and C towards the centre of the ingot was much more marked in the untreated steel, and this produced more rapid grain growth and recrystallisation after rolling and annealing. The tensile properties of the untreated steel approach those of the treated steel only at the bottom of the ingot; at the top they are much poorer owing to the presence of numerous small blowholes. The effect of the degassing treatment on the magnetic ageing of the steel is illustrated diagrammatically.

A. R. POWELL.

**Decarburisation of carbon steels in fused salt baths.** A. SEUTHE and E. H. SCHULZ (*Mitt. Forsch.-Inst. Ver. Stahlwerke*, 1931, **2**, 61—76; *Stahl u. Eisen*, 1931, 791—793).—The decarburising action of the ordinary salt bath (NaCl, KCl, or BaCl<sub>2</sub>) used in annealing steel is due entirely to dissolved O<sub>2</sub> derived from the air. Part of this O<sub>2</sub> is liberated when the bath solidifies as well as when the temp. is reduced by the introduction of cold steel. The formation of Fe<sub>2</sub>O<sub>3</sub> on the steel surface is accompanied by contamination of the bath with particles of this Fe<sub>2</sub>O<sub>3</sub> which flake off, and hence with prolonged use the decarburising action of the bath tends to increase. The presence of Na<sub>2</sub>SO<sub>4</sub> or of alkali or alkaline-earth oxides or carbonates in the bath also accelerates decarburisation, but NaCl, KCl, or BaCl<sub>2</sub> in the absence of O<sub>2</sub> or oxides is entirely inert.

A. R. POWELL.

**Corrosion prevention of ferrous metals.** V. V. KENDALL and F. N. SPELLER (*Ind. Eng. Chem.*, 1931, **23**, 735—742).—In reviewing the principal developments in recent corrosion research, mention is made of the resistance of 0.25% Cu steel to atm. corrosion, the de-aeration of boiler-water supplies, the addition of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> or other film-forming inhibitors to water-circulating systems, and improved paints and lacquers including synthetic resins.

C. A. KING.

**Analysis of copper alloys and white metals.** W. BERG (*Chem.-Ztg.*, 1931, **55**, 530—531).—Slight modifications are made in the well-known procedure for the analysis of brass, bronze, and white metals.

A. R. POWELL.

**Age-hardening mechanism in aluminium-magnesium silicide alloys and in duralumin.** S. KOKUBO (*Sci. Rep. Tôhoku*, 1931, **20**, 268—298).—As a result of measurements of the density, electrical resistance, and hardness of the alloys after various thermal treatments, together with dilatometric and thermal study of the quenched materials, the author concludes that the precipitation theory of age-hardening is untenable. The main grounds for this conclusion are that precipitation would be accompanied by (a) an evolution of heat and (b) a decrease in resistivity, whereas age-hardening proceeds with absorption of heat and increase in resistivity. Annealing at higher temp. (450°) which

does not result in hardening results in the changes (a) and (b), which are ascribed to precipitation. It is therefore suggested that the hardening resulting from ageing of quenched Al-Mg<sub>2</sub>Si alloys is brought about by lattice distortion arising out of local concentration of the stronger atoms in the lattice. Mg<sub>2</sub>Si is not formed at this temp. Experiments with duralumin lead to the conclusion that the hardening in this case is due to similar causes, with the difference that Cu atoms also become conc. in some parts of the crystals, as is shown by their reaction to form CuAl<sub>2</sub> when the temp. is raised sufficiently.

E. H. BUCKNALL.

**Refining of aluminium and its alloys by treatment with chlorine and nitrogen.** W. KOCH (*Metallwirt.*, 1931, **10**, 69—72, 85—88; *Chem. Zentr.*, 1931, i, 2109).—By passing Cl<sub>2</sub> through the molten mass two periods are differentiated: (1) almost complete absorption of Cl<sub>2</sub>, (2) escape of Cl<sub>2</sub> and volatile metallic chlorides. Various alloys were chlorinated and then degassed with N<sub>2</sub>; the properties of the products are described. A finer structure is produced by separation of the foreign metals in a highly disperse form. Selective volatilisation of the impurities is impossible.

A. A. ELDRIDGE.

**Corrosion of tin plate by fruit juice.** A. PELLERIN and E. LASAUSSE (*Chim. et Ind.*, 1931, **25**, 1348—1353).—Experiments with dil. solutions of acid sealed into tin-plate containers under conditions comparable with those of fruit packing show that the presence of air greatly promotes corrosion. NaCl acts as a partial protective agent in 2% concentration. Cold citric acid attacks the tin, whilst with AcOH chiefly Fe is dissolved. Na<sub>2</sub>HPO<sub>4</sub>.12H<sub>2</sub>O prevents corrosion at *p*<sub>H</sub> 7.7. The bursting of containers is most readily produced by AcOH. Double tinning or varnishing is useless as an additional protection against acid corrosion, but reduce corrosion by fatty materials or by neutral substances, e.g., peas. In all cases corrosion was measured by Sn and Fe found in solution after periods up to 2 years.

C. IRWIN.

**Italian production of cadmium.** L. CAMBI and V. TOJA (*Giorn. Chim. Ind. Appl.*, 1931, **13**, 281—286).—Calamines (0.051—0.056% Cd) are utilised at the Montepioni electrolytic Zn works. The cadmiferous Zn deposits are roasted in an Oxland furnace and the Cd oxides are recovered by volatilisation (yield 94—95% of the Cd content). The Cd is precipitated by cathodic Zn plates from H<sub>2</sub>SO<sub>4</sub> solution (1—2.5 g. per litre). This ppt. oxidised in air for 48 hr. is dissolved in H<sub>2</sub>SO<sub>4</sub> containing 0.2—0.3% HNO<sub>3</sub>; the solution (74—83 g. Cd and 18—24 g. Zn per litre) is electrolysed in cells provided with pulsating cathodes (90 vibrations per min.) and the electrolyte is stirred every 30 min. The current is 50 amp. per sq. m. and 3.5 volts. The Cd deposits are fused first under heavy oil and then under NaOH. The annual output of the plant is 12 tons. The purity is 99.92% Cd.

O. F. LUBATTI.

**Dip-sampling cyanide gold bullion at the Sons of Gwalia Gold Mine, Gwalia, W. Australia.** C. O. A. THOMAS (*Proc. Austral. Inst. Min. Met.*, 1931, 17—22).—An average sample of the bullion contains 82.5% Au, 6.7% Ag, 7.5% Cu, 2.6% Pb, 0.5% Zn,

and 0.2% Fe. When cast into 920-oz. ingots the outside bottom of the bar contains less Au and the outside top more Au than the average. Accurate samples were obtained by taking a dip from the molten metal by means of a hollow graphite rod and casting beneath castor oil in an Fe mould. A. R. POWELL.

**Structures in sea-water.**—See IX. **Purification of blast-furnace gas.** **Induction furnaces for steel.** **Magnetic induction of steels.** **Flue gas purification.**—See XI.

## PATENTS.

**Case-hardening furnace with charging device of worm type.** H. LINDHORST (B.P. 350,812, 21.8.30. Addn. to B.P. 330,188; B., 1930, 823).—The feed hopper of the furnace previously claimed is in the form of an upright truncated cone and feeds the hardening powder to the worm charging device through an opening in the bottom, which slopes towards the centre. The powder is preheated by means of a coil in the hopper through which the hot furnace gases are passed and a double-bladed rotating scraper is provided at the bottom of the hopper to move the powder towards the opening and to prevent clogging. A. R. POWELL.

**Metal-treating furnaces [for case-hardening].** W. FAIRWEATHER. From SINGER MANUFG. CO. (B.P. 346,924, 3.3.30).—The furnace comprises a tubular horizontal retort inside a heating chamber and provided with a conveyor on which the articles buried in a thin layer of carburising powder are passed through the retort, the mouth of which is so restricted that this layer forms a practically gastight seal. A. R. POWELL.

**Heat treatment of metal articles.** H. ROSENBERG (B.P. 349,902, 23.12.29).—A mechanically operated, totally enclosed furnace arrangement is claimed by means of which articles can be carburised in a cyanide bath and transferred directly to a quenching bath without coming in contact with the air, without appreciable fall in temp. during transfer, and without escape of dangerous fumes from the plant. A. R. POWELL.

**Desulphurisation of cast iron.** L. W. SPRING, Assr. to CRANE CO. (U.S.P. 1,785,503, 16.12.30. Appl., 22.6.29).—The Fe from the cupola is passed through a trough into the top of a desulphurising chamber containing an alkaline flux and the treated metal is withdrawn from a trough leaving the bottom of the chamber and running parallel to and below the first trough and discharging into a ladle below the entrance into the first trough. A. R. POWELL.

**Reduction of pyritic ores [for recovery of sulphur].** L. MELLERSH-JACKSON. From PATENTAKTIEBOLAGET GRONDAL-RAMÉN (B.P. 350,625, 31.3.30).—Pyritic ores are smelted with fluxes and such quantities of solid carbonaceous reducing agents that a complete or partial pyritic smelting takes place in the lower part of the furnace and the  $\text{SO}_2$  and, if present,  $\text{As}_2\text{O}_3$  formed in this zone are reduced to S and As, respectively, in the upper zones of the furnace, whilst any Cu collects as matte in the hearth. A. R. POWELL.

**Treatment of ores and materials containing iron in combination with sulphur.** **Treatment**

**of [sulphide] iron ores.** COMSTOCK & WESCOTT, INC., and E. W. WESCOTT (B.P. 348,664—5, 18.11.29. [B] Addn. to B.P. 306,107; B., 1930, 106).—(A) The ore, e.g., pyrites, is heated in a dil.  $\text{Cl}_2$  atm. to convert the Fe into  $\text{FeCl}_2$  and liberate the S as vapour, which is removed and condensed. The  $\text{FeCl}_2$  is then treated with preheated air and  $\text{Cl}_2$  to convert it into  $\text{FeCl}_3$  which is volatilised and condensed and the  $\text{Cl}_2$ - $\text{N}_2$  gas mixture is used again in the first stage of the process. The  $\text{FeCl}_3$  is revaporised and burned with air to form  $\text{Fe}_2\text{O}_3$  and  $\text{Cl}_2$ , which is passed back to the process. (B) The  $\text{Cl}_2$  gases from the combustion of the  $\text{FeCl}_3$  together with fresh  $\text{Cl}_2$  are passed partly into the chamber wherein the  $\text{FeS}_2$  is converted into  $\text{FeCl}_2$  and partly into the chamber wherein the  $\text{FeCl}_2$  is converted into  $\text{FeCl}_3$ . A. R. POWELL.

**Obtaining a highly porous, cellular, light material from blast-furnace slag and similar fused materials.** C. H. SCHOL (B.P. 350,526, 7.3.30. Ger., 23.1.30).—The molten slag as it comes from the blast furnace runs down a short trough on to a water-cooled inclined plane from which it falls into the granulating trough where it meets a stream of preheated  $\text{H}_2\text{O}$ , which causes the slag stream to break up into a porous cellular mass. The granulating trough has only a short straight portion terminating in a long downwardly curved portion to allow the material to fall more readily as the  $\text{H}_2\text{O}$  is progressively evaporated. A. R. POWELL.

**Manufacture of low-carbon chromium-steel.** F. O. KICHLINE, Assr. to BETHLEHEM STEEL CO. (U.S.P. 1,785,199, 16.12.30. Appl., 20.4.26).—Granulated pig Fe containing Cr and C is melted in an electric furnace with an excess of chromite over that required to oxidise the C and with sufficient  $\text{CaO}$  to form a highly basic slag; this slag is removed and the resulting Fe-Cr alloy low in C is refined by the usual methods. A. R. POWELL.

**Manufacture of steel having good machining properties.** F. BORGGRAFE (B.P. 350,888 and 350,919, 10.1.30).—(A) The process claimed in B.P. 318,177 (B., 1931, 207) is carried out in a normal large Bessemer converter. (B) The additions of Cu and S or FeS claimed in B.P. 340,529 (B., 1931, 301) are made in the refining furnace or in the ladle. A. R. POWELL.

**Articles made of steel alloys and requiring high permanent strength, such as containers, pipes, boiler parts, etc.** F. KRUPP A.-G. (B.P. 348,668, 14.12.29. Ger., 24.12.28).—The alloy comprises Fe with about 0.15% C and either 1–2.5% W, 0.5–2% Cr, and 0.4% Mo or 0.8% Mo and 1% Cr; in addition it may contain 0.2–6% Si, 0.2–5% V, and 0.2–5% Ti. After fashioning into pipes or pressure vessels the articles are annealed, quenched, and tempered, and will then withstand pressures of up to 300 atm. at 250–600°. A. R. POWELL.

**Rolling and annealing of sheet metal [steel].** A. PATERSON (U.S.P. 1,785,786, 23.12.30. Appl., 26.3.28. Renewed 14.5.30).—After the usual hot-rolling process the sheet is allowed to cool within the "blue-short" zone (316–427°) and the rolling finished. The final annealing is effected by heating for a short

time in the "growth range," *e.g.*, just above the crit. point. A. R. POWELL.

**[Ferronickel] magnetic material [for transformer cores].** W. F. BRANDSMA, P. R. DIJKSTERHUIS, G. J. SZOO, and G. B. JONAS, Assrs. to RADIO CORP. OF AMERICA (U.S.P. 1,785,246, 16.12.30. Appl., 1.8.28. Holl., 19.7.27).—Transformer core plates consisting of 50 : 50 Fe-Ni alloy with 0.095% C are heated in air at 900° until the C is reduced to 0.04%, and the material is partly oxidised so that it gives a substantially straight hysteresis curve. A. R. POWELL.

**Rolling of alloy steel.** H. M. GIVENS, F. B. FOLEY, and J. L. COX (U.S.P. 1,786,297, 23.12.30. Appl., 20.11.29).—Martensitic alloy steel is heated to 1260° and passed through the rolls until the temp. falls to about 1090°; the steel is then allowed to cool below its crit. temp., *e.g.*, to 815°, reheated to 1040°, and the rolling finished as usual, but at not below 760°. A. R. POWELL.

**Salt-bath furnace for heat-treatment of steel.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 349,830, 19.7.30. Ger., 24.8.29).—The bath comprises a metal crucible which acts as one electrode and a second electrode in the form of a hollow cylinder projecting into the crucible and forming an annular space between the walls of the crucible and the cylinder. The heating devices for melting the salt and the materials to be heat-treated are introduced into the bath through the middle of the cylinder. A. R. POWELL.

**Electrically heated salt-bath furnace for heat-treatment of steel.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 349,846, 7.8.30. Ger., 9.8.29).—The furnace is heated by means of three U- or W-shaped electrodes connected separately to the three poles of a three-phase current supply. The electrodes are so constructed that they can be used as resistance heaters for melting the salt, then as conductors for passing the current through the fused salt to maintain the temp. of the bath. A. R. POWELL.

**Annealing.** J. R. CAIN, Assr. to RICHARDSON CO. (U.S.P. 1,784,221, 9.12.30. Appl., 2.8.27).—The annealing vessel is half-filled with molten Pb on which floats a layer of hydrocarbon oil. The greater part of the Pb bath is disposed inside a Ni-Cr wire-wound electric furnace which maintains it at the correct annealing temp., whilst the upper part of the vessel is maintained relatively cool so that the sheet metal after annealing is withdrawn at a temp. below that at which its surface becomes discoloured by oxidation. A. R. POWELL.

**Heat-treatment of cold-shaped manganese-steel articles.** H. WADE. From TAYLOR-WHARTON IRON & STEEL CO. (B.P. 351,691, 23.5.30).—Super-hardness of steel containing 10–15% Mn and <5% Ni, induced by cold-work, is eliminated by heating the partly formed article for 5–15 min. at 700–710°, air-cooling, completing the cold-work, and again annealing for 5–15 min. at 650°. A. R. POWELL.

**Working manganese-iron or -steel alloys.** H. WADE. From TAYLOR-WHARTON IRON & STEEL CO. (B.P. 335,076, 4.10.29).—Shaped articles made of steel containing 10–15% Mn are subjected to surface

abrasion, with or without shock, to increase the surface hardness locally in the parts to be subjected to wear. A. R. POWELL.

**Rust-proofing of iron, steel, and other metals.** W. H. COLE (B.P. 352,202, 10.6.30).—The articles are heated at 500–600° in a mixture of 3000 pts. of emery, 2000 pts. of Zn dust, 1000 pts. of Al powder, 200 pts. of Cr<sub>2</sub>O<sub>3</sub>, 30 pts. of CuO, 5 pts. of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, 5 pts. of C<sub>10</sub>H<sub>8</sub>, and 5 pts. of NH<sub>4</sub>Cl. A. R. POWELL.

**Manufacture and use of compositions for coating metals [iron].** PARKER RUST PROOF CO. (B.P. 346,401, 10.1.30. U.S., 10.1.29).—The coating bath comprises an acid phosphate solution containing MnH<sub>2</sub>P<sub>2</sub>O<sub>8</sub> and CuO in the ratio 40 : 1. A. R. POWELL.

**Proofing of iron and steel against rust.** W. H. COLE (B.P. 350,420, 10.12.29 and 20.5.30. Addn. to B.P. 289,906; B., 1928, 488).—The metal is boiled in a solution obtained by dissolving 50 g. of Fe, 60 g. of Zn, 10 g. of Al, and 10 g. of Cr in 1 litre of H<sub>3</sub>PO<sub>4</sub> (*d* 1.09) and diluting to *d* 1.02. A. R. POWELL.

**Coating metal articles [with an iron-chromium-silicon alloy].** F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,784,570, 9.12.30. Appl., 29.10.28).—The articles are heated in a reducing atm. with a powdered mixture of an alloy of 40% Fe, 40% Cr, and 20% Si with Al<sub>2</sub>O<sub>3</sub> and Na<sub>2</sub>SiF<sub>6</sub>, whereby a corrosion-resistant film of the alloy is formed on the surface of the articles. A. R. POWELL.

**Coating of metallic articles [iron pipes] with refractory materials.** A. E. WHITE. From DOHERTY RES. CO. (B.P. 350,003, 1.3.30).—The pipe is coated internally with a refractory mixture containing ZrSiO<sub>4</sub> and bonded with FeCl<sub>3</sub>, and the coated pipe is heated at 1370° until the FeCl<sub>3</sub> is completely decomposed and the Fe oxides formed by its action on the Fe of the pipe have diffused some distance into the refractory lining so as to form a firm joint between the pipe and the lining. Such pipes are useful for the transfer of corrosive liquids or hot gases. A. R. POWELL.

**Production of ferromolybdenum and ferro-tungsten.** P. SCHWARZKOPF (U.S.P. 1,786,393, 23.12.30. Appl., 29.4.29. Ger., 22.5.26).—MoO<sub>3</sub> or WO<sub>3</sub> vapour or the finely-divided product obtained by condensation therefrom (cf. U.S.P. 1,629,004; B., 1927, 528) is added directly to the bath of molten Fe. A. R. POWELL.

**Solvent inhibitor for the acid treatment [pickling] of metals.** W. S. CALCOTT and I. E. LEE, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,785,513, 16.12.30. Appl., 17.4.29).—Thiophenol, benzyl or isopropyl mercaptan, or similar substances are claimed as inhibitors in H<sub>2</sub>SO<sub>4</sub> pickling baths. A. R. POWELL.

**Facing of cutting, drilling, and boring tools, and welding rod [therefor].** H. J. C. FORRESTER. From STROODY CO. (B.P. 350,607—8, 22.3.30).—(A) The mild steel shank is first faced with a layer of hard steel, then with a layer of mild steel in which are embedded particles of WC. (B) The second operation is performed with an O<sub>2</sub>–C<sub>2</sub>H<sub>2</sub> flame and a welding rod comprising a mild steel tube packed with granular WC. A. R. POWELL.

**Refining [the grain structure] of non-ferrous metals or alloys.** D. R. TULLIS and P. OAKLEY (B.P. 351,177, 8.5.30).—Molten Al, Cu, Ni, or Pb, or their alloys, are treated with  $\text{Cl}_2$  and the vapour of  $\text{VOCl}_2$ .

A. R. POWELL.

**[Copper-tin-zinc] alloy.** H. KANZ (B.P. 350,889, 13.1.30. Switz., 18.1.29).—An alloy for the manufacture of acoustic instruments comprises Cu with 2—16 (6)% Sn, 10—46 (34)% Zn, 0.1—7.5% Al, Fe, Mn, or Ni (0.5% Ni), and 0.1—5% Pb, Sb, or Si (0.2% Si). The proportion of the constituents is so arranged that the structure of the alloy consists of  $\alpha$  + eutectoid.

A. R. POWELL.

**Copper alloys [containing sulphur].** A.-G. VORM. O. BRANDENBERGER METALLGIESSEREI U. HÜTTENPRODUKTE, ASSEES. OF W. STRAUS (B.P. 350,775, 16.7.30. Ger., 17.7.29).—The addition of up to 4% S to high-Cu bronze bearing metals is claimed.

A. R. POWELL.

**Copper-silicon-zinc alloy.** HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 350,750, 1.7.30. Ger., 27.5.30).—Alloys with 80—90% Cu, 2—5% Si, and the remainder Zn are claimed. Part of the Zn or Cu may be replaced by 1—3% of one or more of the metals Al, Mn, Co, Ni, Cr, Zr, W, Mo, Ti, and Sn. Alloys with 85% Cu, 5% Si, and 10% Zn, and with 81% Cu, 4.5% Si, 14% Zn, and 0.5% Sn are particularly suitable for the production of dense sand castings and for bearing metals.

A. R. POWELL.

**Sealing [copper] wire and other metal parts to glass.** WESTINGHOUSE LAMP CO., ASSEES. OF D. F. WAY (B.P. 348,885, 19.5.30. U.S., 17.5.29).—The wire is dipped into a solution containing  $\text{Na}_2\text{B}_4\text{O}_7$  and  $\text{NaNH}_4\text{HPO}_4$  and the wet wire is dried and heated until the salts form a thin glaze over its surface. The wire can then readily be sealed into glass.

A. R. POWELL.

**Treatment of nickel-copper-bearing material.** INTERNAT. NICKEL CO., INC., ASSEES. OF R. C. STANLEY (B.P. 351,150, 23.4.30. Appl., 17.8.29).—Bessemerised Cu-Ni matte is smelted with  $\text{Na}_2\text{SO}_4$  and C, the top layer of Cu-Na sulphides is removed, and the bottom layer is again smelted with  $\text{Na}_2\text{SO}_4$  and C. The combined top layers are treated in a converter to obtain blister Cu and the final bottom layer is ground, leached to remove  $\text{Na}_2\text{S}$ , and roasted to  $\text{NiO}$ , which is treated by the carbonyl process for the recovery of pure Ni.

A. R. POWELL.

**Separation [of antimony from] and purification of metals [lead] and alloys.** A. HANAK (U.S.P. 1,786,908, 30.12.30. Appl., 8.8.28).—Pb or Pb-Sn alloys are freed from Sb by melting at  $450^\circ$  with sufficient Na to form  $\text{Na}_2\text{Sb}$ , cooling to just above the m.p., stirring with molten NaOH, and skimming off the flux which contains all the Sb. On dissolving the flux in hot  $\text{H}_2\text{O}$ , the Sb separates as a powder containing over 90% Sb and the solution may be evaporated to recover NaOH for use again.

A. R. POWELL.

**Removal of lead coatings from metallic articles.** K. B. BOWMAN, ASSR. TO REPUBLIC STEEL CORP. (U.S.P. 1,785,245, 16.12.30. Appl., 8.10.29).—The article is immersed in molten  $\text{NaNO}_3$  to oxidise the Pb to  $\text{PbO}$  and the  $\text{PbO}$  is removed by pickling the article in HCl.

A. R. POWELL.

**Treatment of lead-bearing material [waste battery paste etc.].** A. STEWART, ASSR. TO C. L. CONSTANT CO. (U.S.P. 1,783,986, 9.12.30. Appl., 22.3.27).—The dry paste (1 ton) is heated with a solution of 600—700 lb. of NaCl in 100 gals. of  $\text{H}_2\text{O}$  and with 1000 lb. of  $\text{H}_2\text{SO}_4$  ( $d$  1.84) until copious fumes of  $\text{H}_2\text{SO}_4$  are evolved; most of the As and Sb are thereby evolved with the  $\text{Cl}_2$  and HCl vapours. The resulting  $\text{PbSO}_4$  paste is washed with a boiling 20%  $\text{NaHSO}_4$  solution to remove the remaining Sb and As, then with  $\text{H}_2\text{O}$  to remove acid, and dried.

A. R. POWELL.

**Reduction of metallic compounds.** G. N. KIRSEBOM (B.P. 351,653, 2.5.30. Swed., 14.5.29).—Oxides of Pb, Cd, Sn, Sb, or Bi are reduced to metal by fusion with NaOH and As. The process is particularly applicable to the treatment of flue dusts and the slags produced in the Harris process. If the latter contain  $\text{NaNO}_3$  this is first reduced by the As to  $\text{NH}_3$ , then with further quantities of As a Pb-Sb alloy is produced, and finally when the slag becomes very viscous Sn separates in prills which may be recovered by leaching out the sol.  $\text{Na}_3\text{AsO}_4$ .

A. R. POWELL.

**Production of aluminium alloys.** RUSELITE CORP. (B.P. 350,357, 30.9.30. U.S., 25.4.30).—The alloys comprise Al with 3—10% Cu, 0.75—5% Cr, 0.1—5% Mo, and 0.05—1% Ti, with a min. of 85% Al. The Al is first melted under a slag containing Ca, B, and F, and the Cu, Cr, and Mo are added in that order.

A. R. POWELL.

**Production of wires from aluminium alloys.** H. BOHNER (B.P. 349,978, 5.3.30).—The alloy is drawn down to wire slightly larger than that required, annealed at  $500$ — $520^\circ$ , quenched, tempered at  $200$ — $250^\circ$ , and drawn down to the desired size. This treatment gives a wire of high strength and electrical conductivity.

A. R. POWELL.

**Protection of aluminium and its alloys and/or other metals against corrosion.** H. SUTTON and H. C. COCKS (B.P. 351,396, 22.2. and 16.4.30).—The articles are plated with Cd in a bath containing 62 g. of  $\text{K}_2\text{Cd}(\text{CN})_4$  per litre or 52 g. of  $\text{CdSO}_4$  and 64 g. of NaCN per litre, using Cd anodes. Addition of 15 c.c. of aq.  $\text{NH}_3$  ( $d$  0.88) per litre assists anodic corrosion and prevents the formation of anode slimes.

A. R. POWELL.

**Production of aluminium and [silicon]-aluminium alloys.** W. NEUMANN (B.P. 351,524, 24.3.30).—Bauxite or clay low in Fe is reduced electrothermally to produce a Si-Al alloy which is allowed to cool slowly in a liquation furnace. The crystals of Si which collect at the surface are removed, drained, and pressed, and the liquid eutectic of Al with 12% Si is liquated away from the heavy residue of  $\text{Fe}_3\text{Al}$  and  $\text{TiAl}_4$ . Al is recovered from the eutectic by distillation in vac. and the residue is returned to the liquation furnace. The crude Si produced is returned to the electric furnace when bauxite with a low Si content is being reduced, to assist in the collection of the Al.

A. R. POWELL.

**Manufacture of [copper]-plated [lead] shot.** E. PUGSLEY, ASSR. TO WINCHESTER REPEATING ARMS CO. (U.S.P. 1,785,493, 16.12.30. Appl., 9.1.29).—Pb shot is prepared in the usual way except that the molten drop-

lets are allowed to fall into a solution of  $\text{CuSO}_4$ , *e.g.*, brass pickling liquor, containing metallic Fe.

A. R. POWELL.

**Treatment of zinciferous materials containing lead and/or cadmium.** L. E. TETER, Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,779,862, 28.10.30. Appl., 22.9.28).—The roasted ore is mixed with fuel and 2.5–5% of NaCl and the mixture is sintered in the Dwight-Lloyd apparatus in layers about 4 in. thick. The bottom 1 in. of the sintered material is collected separately, as it contains the greater part of the Pb and Cd which were present in the ore.

A. R. POWELL.

**Treatment of zinc ore [for recovery of zinc].** W. W. TRIGGS. From ST. JOSEPH LEAD CO. (B.P. 347,239, 17.12.29).—A granular charge of sintered Zn ore and carbonaceous material is fed continuously through an electrically heated shaft furnace built up of a series of independently supported sections, between which are provided outlets for the evolved gases, and provided with a number of spaced electrodes at the top of the furnace and a similar number at the bottom, by means of which a current is caused to pass through the charge sufficient to raise it to a temp. at which the Zn distils without fusion or slagging of the residue, but below that at which sulphides distil from the ore.

A. R. POWELL.

**Coating zinc and cadmium.** A. PACZ (U.S.P. 1,784,106, 9.12.30. Appl., 28.5.27).—Zn and Cd are coloured blue to black by immersion in a dil. solution of  $(\text{NH}_4)_2\text{MoO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$ .

A. R. POWELL.

**Preparation of metals [*e.g.*, nickel] for use as catalysts.** A. A. THORNTON. From SOC. D'ETUDES ET D'EXPLOIT. DES MATIÈRES ORGANIQUES (B.P. 350,451, 10.3.30).—Coiled Ni wires are heated at  $150^\circ$  in  $\text{Cl}_2$  until they become coated with a layer of  $\text{NiCl}_2$ , which is subsequently reduced to a porous Ni sponge by heating at  $150^\circ$  in  $\text{NH}_3$ .

A. R. POWELL.

**Production of protective metal coating [chromium-plate].** C. G. FINK and C. H. ELDRIDGE, Assrs. to CHEM. TREATMENT CO., INC. (U.S.P. 1,786,398, 23.12.30. Appl., 12.8.25. Renewed 6.5.30).—A gas-free Cr-plate is obtained by periodically raising the article being plated into a hood above the plating bath and therein subjecting it to reduced pressure.

A. R. POWELL.

**Chromium-plating small articles in quantity.** E. KRAUSE, and LANGBEIN-PFANHAUSER-WERKE A.-G. (B.P. 351,168, 5.5.30).—The apparatus used comprises a rotating metal drum containing an eccentric anode made in the shape of a ribbed sector of a cylindrical surface, the ribs consisting of flat parallel plates. The drum can be set in four positions by an adjusting device; in the first position the articles are Cr-plated, in the second the plated articles are allowed to drain, in the third they are rinsed, and in the fourth the drum is emptied and recharged. Provision is made for exhausting through the hollow central shaft the gases evolved during the plating operation.

A. R. POWELL.

**Production of metallic magnesium from its compounds.** OESTERR. AMERIKANISCHE MAGNESIT A. G. (B.P. 351,295, 30.7.30. Austr., 3.8.29).—A mixture of  $\text{MgCO}_3$  and C is allowed to fall through a luminous electric arc through which a current of  $\text{H}_2$  is passed.

The issuing gases are cooled below  $1200^\circ$  and passed through an electrostatic apparatus in which liquid Mg collects on the electrodes and falls continuously therefrom into a container. The issuing gases are cooled, freed from dust in another electrostatic apparatus, mixed with steam, and heated to convert CO into  $\text{CO}_2$ , which is absorbed and the  $\text{H}_2$  used again in the process.

A. R. POWELL.

**Producing metallic cadmium from cadmium-containing material.** H. H. MONROE, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,785,139, 16.12.30. Appl., 18.5.28).—Cd-rich residues obtained as by-products in the purification of  $\text{ZnSO}_4$  solutions, in the smelting of Zn ores, or in the distillation of crude Zn are melted with NaOH and a powdered carbonaceous fuel at  $420^\circ$ , whereby the Pb, As, S, and Zn dissolve in the caustic slag and Cd of 98–99% purity can be tapped from below the flux.

A. R. POWELL.

**Cleaning of liquid mercury and the like.** B. L. NEWKIRK, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,786,312, 23.12.30. Appl., 14.12.27).—Hg containing particles of Fe or Fe oxides, derived, *e.g.*, from a Hg turbine, is passed through a chamber or chambers packed with steel balls about  $\frac{1}{8}$  in. in diam., whereby there is formed on the balls a fine skin of Hg which retains all the suspended impurities.

A. R. POWELL.

**Separation of gold, silver, and lead from (A, B) crude antimonial metal, or (C, D) oxidised antimonial ores, flue dusts, or by-products.** (A–D) S. G. BLAYLOCK, (A–C) J. J. FINGLAND, (A–D) F. E. LEE, and (D) P. F. MCINTYRE, Assrs. to CONSOLIDATED MINING & SMELTING CO. OF CANADA, LTD. (U.S.P. 1,778,017–1,778,020, 14.10.30. Appl., 3.5.29. Can., 21.5.28).—(A) Crude Sb containing Au and Ag is melted with at least 100 pts. of Pb for every 2.25 pts. of Ag present and at least a further 100 pts. of Pb for every 4 pts. of Au present and the alloy is cast into large ingots, which are hung in a furnace and heated in a reducing atm. at  $550$ – $630^\circ$  to allow the Pb–Ag and Pb–Au alloys to liquate out and drip into a collecting chamber. (B) After addition of Pb as in (A) the alloy is subjected to fractional crystallisation as in the Pattinson process of desilverising Pb. (C) The material is smelted with the necessary Pb to collect the Au and Ag and with fluxes and reducing agents to obtain a Pb–Sb alloy, which is cast into a large, heat-insulated mould and allowed to cool slowly. The Sb solidifies round the sides and top and the Pb–Au–Ag towards the centre of the mass. (D) The material is smelted with Pb and fluxes as in (C), but with only enough C to reduce 10–20% of the total metal content to obtain an antimonial slag and a rich Sb alloy containing the precious metals.

A. R. POWELL.

**Gold alloy for jewelry.** CARTIER (B.P. 351,811, 21.8.30. Fr., 14.6.30).—The alloy contains 75% Au, 0.1–2.5 (1) % Cr, and the remainder Cu and Ag in the ratio 1.4 : 1.

A. R. POWELL.

**[Flotation] concentration of ores.** G. H. WIGTON (U.S.P. 1,783,206–7, 2.12.30. Appl., [A] 21.6.28, [B] 3.7.29).—Oxidised Pb ores are floated with the reaction product of  $\text{P}_2\text{S}_5$  and  $\alpha$ -naphthylamine in (A) BuOH, or (B) *o*-toluidine.

A. R. POWELL.

**Leaching and washing ores by the use of gas.** A. B. STEVENS (U.S.P. 1,783,591, 2.12.30. Appl., 26.1.29).—The ore is leached in a tank containing a perforated false bottom forming a chamber below the ore body and the leaching solution is passed upwards from this chamber through the ore mass. Before reaching the chamber part of the leaching liquor is by-passed through a mixing chamber in which fine jets of gas are intermingled with the liquor and the mixture is passed first through a centrifugal pump to ensure thorough mixing of gas and liquor, and thence through pipes into various parts of the supply chamber below the ore, the pipes being backwardly directed against the main flow of leaching liquor to effect thorough mixing therewith. A. R. POWELL.

**Complete washing of sulphur ores and other sulphur-containing substances.** O. SALADIN (B.P. 350,086, 21.3.30).—Pyrites is roasted in a multiple-hearth furnace and the charge in the zone of highest temp. is transferred to an intermediate zone, between the combustion and cooling zones, which is protected from rapid cooling by by-passing the whole or a part of the air for roasting from the cooling zone, where it enters the furnace, directly to the combustion zone. The charge is thus kept at the highest temp. with the access of little or no air until the whole of the S is expelled. A. R. POWELL.

**Lining compositions for permanent or chill moulds for magnesium and its alloys.** I. G. FARBERIND. A.-G. (B.P. 348,470, 24.7.30. Ger., 10.8.29).—Mould dressings for Mg alloy castings comprise a mixture of equal parts of  $H_3BO_3$  and  $MgO$ ,  $Fe_2O_3$ ,  $Al_2O_3$ , or  $MgCl_2$ . A. R. POWELL.

**Manufacture of cores for castings.** IMPERIAL CHEM. INDUSTRIES, LTD., and H. M. BUNBURY (B.P. 348,315, 29.3.30).—The cores are bonded with liquid rosin, with or without a drying oil and a catalyst, *e.g.*,  $Co$  linoleate. Glue or dextrin may also be added and the rosin may previously be esterified with glycerin. A. R. POWELL.

**Zinc chloride-base fluxes [for soft soldering].** (A) G. LUTZ, (B) W. K. SCHWEITZER, (C) E. A. TAYLOR, and (D) H. P. CORSON and E. S. RIDLER, Assrs. to GRASSELLI CHEM. CO. (U.S.P. 1,785,131, 1,785,147, 1,785,155, and 1,785,181, 16.12.30. Appl., 26.12.29).—The flux comprises a mixture of  $ZnCl_2$  solution in aq. EtOH and HCl with (A) sulphonated castor oil, (B) sulphonated mineral oil, (C) the Na salt of butylnaphthalenesulphonic acid, or (D) an aldehyde, *e.g.*, butaldehyde. A. R. POWELL.

**Soldering. Flux for soft soldering.** H. S. McQUAID, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,785,134—5, 16.12.30. Appl., 26.12.29).—The flux comprises (A) a hydroxyalkylamine, *e.g.*, ethanolamine, or (B) an org. amine soap, *e.g.*, ethanolamine oleate. A. R. POWELL.

**Soldering flux.** H. C. MOUGEY, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,783,925, 2.12.30. Appl., 19.8.29).—The flux comprises a solution of  $ZnCl_2$  and a  $H_2O$ -sol. oil solvent, *e.g.*, di-, tri-, or tetra-ethylene glycol, EtOH, BuOH, the monoacetates of mono- or di-ethylene glycol or of the Me or Et ethers of ethylene

glycol, Et lactate, Et hydroxybutyrate, or diacetone alcohol. A. R. POWELL.

**Soldering flux.** H. P. CORSON, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,785,180, 16.12.30. Appl., 26.12.29).—The flux comprises a sulphonated vegetable oil or one of its  $H_2O$ -sol. salts, *e.g.*, "Monopole" soaps. A. R. POWELL.

**Metal-clad shingles.** INTERNAT. COPPERCLAD CO., Assces. of W. MCL. SHAKESPEARE (B.P. 348,128, 28.12.29. U.S., 29.12.28).—The shingles comprise plates of a cellular fibrous material, *e.g.*, celotex, insulite, or masonite, coated with a waterproofing material or a water-repellent film and the outer surface is covered with a thin sheet of Cu which is trimmed round the edges. A. R. POWELL.

**Lead alloys, more particularly for sheathings of electric cables.** M. THIELERS (B.P. 351,449, 25.2.30).—See U.S.P. 1,779,784; B., 1931, 548.

**Electrodeposition of metals and alloys of the platinum group.** W. W. TRIGGS. From BAKER & CO., INC. (B.P. 348,919, 13.6.30).—See U.S.P. 1,779,436; B., 1931, 593.

**Annealing pots and the like.** E. L. JAMES (B.P. 352,061, 2.4.30).

**Electroplating apparatus [for depositing metal on small iron articles].** W. CANNING & CO., LTD., and G. A. POPE (B.P. 351,659, 9.5.30).

**Hydrocarbons during ore reduction.**—See II. Fe vessels for bleaching goods.—See VI. Recovery of cyanides [from Au working]. Pure  $Al_2O_3$ .  $H_2$  generator.—See VII. Annealing furnaces. Metallic-vapour rectifiers. Re filaments.—See XI.

## XI.—ELECTROTECHNICS.

**Relation between the magnetic induction of constructional steels and the chemical composition.** E. GEROLD (Stahl u. Eisen, 1931, 51, 613—615).—The magnetic induction of plain C steels decreases linearly with increasing C content, the slope of the curves decreasing with increasing field strength. A similar relation holds in the case of Si, but the curves are more nearly horizontal. For steels with 1% Al, Si, Cu, Mo, Mn, or Cr, the induction decreases hyperbolically with increase in field strength. From the curves given it is possible to calc. the magnetic induction of any steel containing small proportions of the above metals from the chemical analysis. A. R. POWELL.

**Advances in the construction and operation of coreless induction furnaces for production of steel.** N. BROGLIO (Stahl u. Eisen, 1931, 51, 605—613, 635—638).—A review of recent developments in the construction of the electric gear and melting crucibles is followed by tables and diagrams showing the energy balance in the manufacture of various types of steel in comparison with other methods of steel manufacture. The temp. of the charge can readily be measured and controlled within narrow limits during the whole of the melting operation, and the life of the crucibles is much longer than that of crucibles heated in fuel-fired furnaces as all the heat is generated in the metal. A. R. POWELL.



**Industrial and chemical research with X-rays of high intensity and with soft X-rays.** G. L. CLARK and K. E. CORRIGAN (*Ind. Eng. Chem.*, 1931, **23**, 815—820).—A modified Ott-Selmayr tube with a moving-picture camera will photograph diffraction patterns showing the stronger spots with exposures of less than 1 sec. This makes possible the study of such phenomenon as efflorescence, of unstable compounds such as  $KI_3$ , or of substances at the temp. of liquid air, and any rapidly proceeding chemical action can be followed. A tube capable of developing a ray of long wave-length is desirable for the study of complex substances. The tube, pinhole, specimen, and camera must be in one unit so that the beam travels in vac. throughout. This has been used on rubber and cellulose. Measurements support the long-chain micellar structure of cellulose. C. IRWIN.

**Some chemical and physical problems of light technology.** M. PIRANI (*Z. angew. Chem.*, 1931, **44**, 395—404).—A review of recent work on the production of light in filament bulbs and in gas-discharge tubes. A. R. POWELL.

**Electrical purification of blast-furnace gas by the Lurgi process at the Lübeck ironworks.** J. DREHER (*Stahl u. Eisen*, 1931, **51**, 577—582).—The plant is designed to treat 40,000 cu. m. of gas per hr. in two stages; most of the dust is precipitated from the hot gas (150—300°) in the first purifying chamber; the gas is then cooled to 20—30°, and passed through the second chamber in which the remainder of the dust and all the excess moisture are removed. The electrodes in both chambers are made of Armeo Fe, which have proved to have a longer life than Ni-Cr alloy wires. Working data and costs over a long period are given diagrammatically, and it is claimed that gas of high purity and low humidity is obtained independently of the method of operating the blast furnace and of the impurities present in the flue gas. A. R. POWELL.

**Thermal supervision of an electrical flue-gas purification plant.** S. HINRICHS (*Stahl u. Eisen*, 1931, **51**, 788—790).—Efficient electrical purification of flue gas depends to a large extent on the careful regulation of the temp. and pressure in the plant, the most satisfactory results being obtained when the gas enters the dust precipitator at 80°. Apparatus in use at a German steelworks for maintaining const. temps. throughout the purifiers, for maintaining the insulators at 225°, and for regulating the temp. (30°) and purity of the gas passing to the gas machines and the pressure in the filters is described with reference to diagrams. A. R. POWELL.

**Dielectric losses and electric dipole moment in transformer oil.** L. S. ORNSTEIN and G. J. D. J. WILLEMSE (*Z. tech. Physik*, 1930, **11**, 345—349; *Chem. Zentr.*, 1931, i, 2022).

$SnSO_4$ .—See VII. Cd.—See X.

#### PATENTS.

**Electrodes for electric furnaces.** SIEMENS PLANIWERKE A.-G. F. KOHLEFABRIKATE (B.P. 351,151, 26.4.30. Ger., 1.8.29).—Annular grooves and corresponding annular ribs are provided in the centre of the ring-shaped

face lying between the screw and socket and the periphery of the electrodes, so that an airtight joint which can be filled with fine graphite powder is made when the electrode is screwed into its socket. A. R. POWELL.

**Electric continuous annealing furnaces.** UGINE-INFRA (B.P. 351,176, 7.5.30. Fr., 8.5.29).—The furnace comprises a refractory wire-wound muffle into the floor of which at one end are inserted the pole-pieces of two electromagnets made of an alloy, e.g., ferrocobalt, which loses its magnetism only at a high temp. The magnets are energised by direct or alternating current in such a way that the articles to be annealed are drawn into the furnace as a magnetic chain, the forward movement of which pushes the annealed and demagnetised articles out at the other end of the furnace. A. R. POWELL.

**Electron-emitting cathodes.** W. W. TRIGGS. From Q.R.S.-DE VRY CORP. (B.P. 351,006, 15.3.30).—Ni, Ni-Si, or Pb cathodes are coated thinly with graphite, then with an oxide or carbonate of an alkali or alkaline-earth metal, and finally with another coating of graphite. The cathodes are then heated to incandescence in vac., whereby a tenacious vitreous coating of high electron-emitting power is produced. A. R. POWELL.

**[Glass for] luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., ASSCES. of PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 351,245, 24.6.30. Ger., 29.8.29).—The bulb comprises an outer envelope of ordinary glass, which is very absorbent for the infra-red and may be coloured or frosted, and an inner layer or independent bulb made from a borosilicate glass containing 4—5%  $Na_2O$ , 11—13%  $Al_2O_3$ , 10—11%  $CaO$ , 40—60%  $B_2O_3$ , and 10—25%  $SiO_2$ . Part, up to one half, of the  $SiO_2$  may be replaced by  $La_2O_3$  or  $Di_2O_3$ . The inner bulb is filled with Na vapour. A. R. POWELL.

**Manufacture of filaments and other bodies from rhenium.** L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 351,216, 4.6.30).—Fine Pt or W wires are heated in an atm. containing vapours of a Re halide. [Stat. ref.] A. R. POWELL.

**Metallic-vapour rectifiers.** INTERNAT. GEN. ELECTRIC CO., INC., ASSCES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 351,164, 2.5.30. Ger., 3.5.29).—The cathode receptacle is insulated from the Fe container by means of a ring or rings composed of Al or Mg or an alloy of these metals, the surface of the rings being coated with a film of the oxide of one of the constituent metals. A. R. POWELL.

**Products [filters] for the absorption of ultra-violet rays.** SOC. DES ÉTABL. GAUMONT (B.P. 351,029, 20.2.30. Fr., 14.3.29).—The filter comprises a solution of *p*-nitrophenol in  $H_2SO_4$  or  $KHSO_4$ . A. R. POWELL.

**[Cover for] electric accumulators.** BRITANNIA BATTERIES, LTD. (B.P. 352,055, 28.3.30. Ger., 28.3.29).

**[Plate supports for] electric storage batteries.** J. LUCAS, LTD., and G. D. SPENCER (B.P. 351,542, 27.3.30).

**Luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., ASSCES. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 352,352—3, 24.10.30. Ger., [A] 29.11.29, [B] 30.11.29).

**Light-sensitive cells.** F. H. CONSTABLE, and RADIOVISOR PARENT, LTD. (B.P. 351,468, 24.3.30).

**Electric gas- or vapour-filled arc-discharge tubes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 351,894, 28.11.30. Holl., 9.1.30).

**Combustion systems for fuel. Oil dehydrator.**—See II. **Pure  $Al_2O_3$ . Detecting  $O_2$  in  $H_2$ .**—See VII. **Magnetic material. Salt-bath furnace for steel. Annealing. Zn from its ore. Al and its alloys. Protecting Al etc. Mg from its compounds. Treatment of waste battery paste. Cr-plate.**—See X. **Insulating varnish films.**—See XIII.

## XII.—FATS; OILS; WAXES.

**Anti-oxidants in edible oil preservation. II. Resistance to oxidation attributable to presence of minute quantities of added or natural inhibitory catalysts.** F. C. VIBRANS (Oil & Fat Ind., 1931, 8, 263—264, 277).—Rates of  $O_2$  absorption (at  $90^\circ$ ) of lard containing anti-oxidants were observed; optimal concentrations of "Agerite" and thymol were 0.5 and 0.2%, respectively. E. LEWKOWITSCH.

**Report of Committee on the Kreis test for rancidity [of edible fats and oils].** A. S. RICHARDSON (Oil & Fat Ind., 1931, 8, 269—270).—The test is too erratic to be recommended for inclusion in the A.O.C.S. official methods: if used, quant. colorimetric measurement of the depth of tint is suggested. E. LEWKOWITSCH.

**Analysis of degreas.** M. AUERBACH (Collegium, 1931, 311—314).—A scheme for the analysis of degreas or moellon is outlined, the values determined including the contents of  $H_2O$ , unsaponifiable matter, oxidised and total fatty acids, and free acid. Tests are also described for indicating the presence of resin, wool fat, mineral oil, naphthenic acid, and resin sulphate in the emulsion. D. WOODROFFE.

**Oil seeds of Angola forest trees.** C. DE M. GERALDES [with A. D'ALMEIDA and C. DUARTE] (Bull. Mat. Grasses, 1930, 14, 332—342; Chem. Zentr., 1931, i, 2136).—*Allanblackia Floribunda*, Oliv., seeds contain 57.5% and the kernels 69.3% of yellowish-white, odourless fat,  $d_{25}^{15}$  0.9187 (0.8948),  $n_D^{20}$  1.4529, m.p.  $40.7^\circ$ , m.p. of fatty acids  $60$ — $62^\circ$ , f.p.  $39.5$ — $40.5^\circ$ , titre  $58$ — $60^\circ$ , saponif. val. 195, I val.  $33.3$ — $36$ , acid val.  $3.6$ — $7.5$ . *Allanblackia Sacleuxii*, Nua., seeds contain 68.9% and the kernels 72.1% of oil,  $d_{25}^{15}$  0.9194,  $n_D^{20}$  1.4551, m.p.  $41.8$ — $42.8^\circ$ , m.p. of fatty acids  $61$ — $63^\circ$ , f.p.  $41$ — $42^\circ$ , titre  $59$ — $61^\circ$ , saponif. val. 207.85, I val. 29.5, acid val.  $35.3$ . *Iringia Robur*, Mildbr., seeds contain 64.9% of fat. *Balanites Mayumbensis*, Exell., seeds contain 35.4%, and the kernels 39.1%, of a reddish-brown oil having a disagreeable odour,  $d_{25}^{15}$  0.9172,  $n_D^{20}$  1.4602, saponif. val. 204.61, I val. 101, acid val. 71.12. *Strimbosia Scheffleri*, Engl., kernels contain 18.1% of a similar oil,  $d_{25}^{15}$  0.9353,  $n_D^{20}$  1.4716, saponif. val. 216.2, I val. 83.6, acid val. 39.08. *Mimusops Ebolowensis*, Engl. et Krause, kernels contain 4.8% of oil. A. A. ELDRIDGE.

**Sapote (mammy apple) seed and oil.** G. S. JAMIESON and R. S. MCKINNEY (Oil & Fat Ind., 1931, 8, 255—256).—Sapote seed (*Calocarpum mammosum*) from Honduras contained 9.4%  $H_2O$  and 57% of oil, suitable for

edible purposes or soap manufacture. The bright yellow expressed oil, after 8 yrs. in cold storage, possessed a faint almond odour and pleasant taste, solidified at  $15^\circ$ , and deposited stearine at  $23$ — $27^\circ$ . It had:  $d_{25}^{25}$  0.9105,  $n_D^{25}$  1.4652, I val. (Hanus) 70.2, saponif. val. 189.5, acetyl val. 12.2, unsaponifiable matter 1.39%, Reichert-Meissl val. 0.15, Polenske val. 0.3, saturated acids (Pb salt— $Et_2O$  method) 30.37%, unsaturated acids 63.73%. The composition is given as (acids %): oleic 52.15, linoleic 19.8 (calc. from I val. etc.), palmitic 9.4, stearic 20.95, arachidic trace. E. LEWKOWITSCH.

**Decolorisation of green tallow.** F. E. CHAPMAN (Chem. Eng. Min. Rev., 1931, 23, Chem. Sect., 355—356).—The green colour of gut tallow, due to chlorophyll, may be removed by digesting with 10—15% of fuller's earth at  $130$ — $140^\circ$  for about 1 hr. and filtering (cost £1 per ton). Bleaching by air or  $H_2O_2$  is too expensive and chlorination is liable to proceed too far. E. LEWKOWITSCH.

**Use of fish oils in the soap industry. II.** ENGELHARDT (Proc. World Eng. Congr., 1929, 31, 105—118).—Processes for deodorising and hydrogenating fish oils are reviewed. The results of the Engelhardt process for deodorising fish oil fatty acids (preferential saturation of malodorous components) and the prep. of oleine from them are discussed. The  $d$  of deodorised fish oils ranges from 0.9494 to 0.9384, and of the acids from 0.9314 to 0.9373; the Tortelli-Jaffe reaction as a rule is positive. E. LEWKOWITSCH.

**Soap analysis Committee report.** H. P. TREVITHICK (Oil & Fat Ind., 1931, 8, 257—261).— $Et_2O$  is unsuitable for the determination of unsaponified and unsaponifiable material in soaps; extraction with light petroleum from solution in 50%  $EtOH$  is recommended and a standard (F.A.C.) procedure is detailed. E. LEWKOWITSCH.

**Effect of  $p_H$  on the detergent action of soap.** F. H. RHODES and C. H. BASCOM (Ind. Eng. Chem., 1931, 23, 778—780).—Actual washing tests (at  $40^\circ$ ) indicate that the detergent effect of soap solution (0.25%) increases and then decreases with increasing alkalinity (although the surface tension decreases continuously); the max. detergent effect is obtained at  $p_H$  10.7 with  $NaOH$ ,  $Na_2CO_3$ , and  $Na_3PO_4$ , but the magnitude of the increase of detergent power at the optimum appears to increase with the valency of the anion. Borax reduces the  $p_H$  of neutral soap solution and does not increase detergency. E. LEWKOWITSCH.

**Bleaching clay.**—See VII. **Determination of fat in leather and tanning products.**—See XV. **Soaps in spirituous preparations.**—See XX.

## PATENTS.

**[Stabilised] oleaginous compositions.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of F. M. CLARK (B.P. 351,171, 6.5.30. U.S., 6.5.29).—Addition of about 0.5% of phenyl- $\alpha$ -naphthylamine protects compositions of fatty or mineral oils, waxes, pitches, etc. from atm. oxidation at raised temp., e.g.,  $125$ — $150^\circ$ . E. LEWKOWITSCH.

**Fatty oil [stabilising] composition.** C. F. KÆGE-BEHN, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,784,360, 9.12.30. Appl., 31.1.29).—0.3—0.5% of a

phenol having an aryl group substituted in the nucleus, *e.g.*, 2- or 4-hydroxydiphenyl, is added to stabilise fats etc. against (atm.) oxidation. E. LEWKOWITSCH.

**Preparations for use as emulsifying and wetting-out agents or soaps, and products obtained with the aid thereof.** G. B. ELLIS. From CHEM. FABR. VORM. SANDOZ (B.P. 350,379, 6.12.29).—Monoaryl ethers of glycerol or glycol, mixed with aromatic, aliphatic, or hydroaromatic carboxylic or sulphonic acids, or their salts or substitution products (*e.g.*, the monoxylal glycerin ether and soaps or sulphonated oils), give clear solutions or stable emulsions, with which hydroaromatic hydrocarbons etc. may be incorporated. The products may be added to soaps to improve the wetting-out powers. E. LEWKOWITSCH.

**Production of soap powder from soap stock high in fatty acid.** C. K. STODDER (U.S.P. 1,785,054, 16.12.30. Appl., 28.8.28).—The semi-plastic, crude, acid soap stock is divided into small fragments, *e.g.*, by extrusion, mixed with powdered soda ash (*e.g.*, 50 wt.-%), and aged until the alkali has absorbed sufficient  $H_2O$  to leave the soap in a brittle condition suitable for powdering. E. LEWKOWITSCH.

**Detergents.** L. MELLERSH-JACKSON. From TWITCHELL PROCESS CO. (B.P. 350,505, 3.2.30).—A mixture of "mahogany sulphonates" (preferably oil-free) and sludge-layer mineral-oil sulphonates can be used in neutral, acid, alkaline, or hard water.

E. LEWKOWITSCH.

**[Paste] cleaning composition.** L. KIRSCHBRAUN, Assr. to F. L. BELKNAP (U.S.P. 1,786,249, 23.12.30. Appl., 9.2.20. Renewed 21.11.27).—An aq. emulsion of a volatile hydrocarbon, *e.g.*, gasoline, stabilised with soap, is passed through a homogeniser to yield a creamy paste which does not flash before an open flame.

E. LEWKOWITSCH.

**Softening of filaments. Waterproofing.**—See VI. **Soldering fluxes.**—See X. **Pine-tar oils etc. Esters for varnishes.**—See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Proportioning the oil in industrial paints.** P. DAUMER (Peint., Pig., Ver., 1931, 8, 1458—1462, 1482).—The quantity of any pigment which will give 100 kg. of paint of correct viscosity, when mixed with a linseed oil medium, may be calc. from the formula  $x = 100y/(y + 45)$ , where  $x$  is the wt. of pigment and  $y$  its sp. gr.  $\times 50$ . The medium consists of linseed oil 70%, liquid driers 15% of the oil, and the remainder thinners. Solid driers possess distinct disadvantages, but when used must be counted as pigment. Practical details of the prep. of paints and lacquer paints are given, together with a description of a simple method of obtaining the density of the dry colour. A diagram shows the quantities of all the constituents required for pigments of varied sp. gr.

F. C. HARWOOD.

**Relation between oil content and protective action [of paints].** H. WOLFF (Farben-Chem., 1931, 2, 297—300).—The "critical oil contents" of mixtures of each of two red leads with  $BaSO_4$  in increasing proportions were determined by the author's method, and

accelerated weathering tests were carried out on paints based on such mixtures. The two red leads show marked differences in behaviour in these tests, but the results of exposure show parallelism with the variation in "critical oil content." S. S. WOOLF.

**Action of ultra-violet rays on the ageing of paints.** H. MASSEILLE (Peint., Pig., Ver., 1931, 8, 1478—1482).—Coatings of paints under test are placed on plates of sheet Fe, 10 cm.  $\times$  12 cm., fixed above a watertight box through which cooling  $H_2O$  is circulated. The quartz Hg-vapour lamp used is generally placed at 20—30 cm. from the plates and the whole apparatus is housed in a closed container. Exposure for 15—20 hr. to ultra-violet rays is found to be analogous to 6 months' exposure to sunlight, when testing the superficial ageing of the film; for deeper ageing, where the elasticity, resistance, and adherence of the film are affected, exposure to ultra-violet rays for 24 hr. gives results analogous to 1 month's exposure to weather.

F. C. HARWOOD.

**Influence of physical properties of carbon black on its tinting strength.** E. P. W. KEARSLEY and G. L. ROBERTS (Ind. Eng. Chem., 1931, 23, 835—837).—The value of C black as a pigment does not depend entirely on the particle size, but varies with the thickness of the film of adsorbed gases. Heating C to even  $110^\circ$  to expel  $H_2O$  resulted in improved tinting strength, but complete displacement of gases is possible only by reducing the surface tension, to ensure complete wetting of the particles by the dispersing medium. The volatile matter present and oil-adsorption properties indicate the tinting characteristics. C. A. KING.

**Chemical processes during the formation and drying of linseed oil leather varnishes.** C. SCHIFFKORN (Chem. Umschau, 1931, 38, 169—175, 185—191).—The processes occurring during the heating of linseed oil in the presence of  $Fe^{+++}$  salts are represented as: (1) a dehydrogenation (oxidation in the Wieland sense) of the ethylenic linking of the unsaturated acids producing acetylenic linkings in their place: the  $Fe^{+++}$  salts act as H acceptors and are subsequently reoxidised by the air to the  $Fe^{+++}$  state, thus re-entering the cycle: (2) the acetylenic linkings are then oxidised to  $CO \cdot CO$  groups; and (3) such polyketonic acids condense with  $CH_2 \cdot CH_2$  groups in adjacent fatty acid chains with the elimination of  $H_2O$  and formation of unsaturated bridging linkings. These intra- or extra-mol. carbocyclic polymerides condition the colloidal nature of the product. During the drying of the solvent-free film of leather varnishes under the influence of ultra-violet light, activated O acts as H acceptor and removes (as  $H_2O_2$ ) activated H from (original?) unsaturated linkings, oxidation and condensation following as above.  $O_3$  in the air may produce ozonolysis of the fatty acids, whilst  $H_2O$  may add on to the double linkings instead of  $O_2$ , reducing the degree of oxidation and polymerisation.

E. LEWKOWITSCH.

**Initial condensation products of phenol with formaldehyde in presence of ammonia as catalyst.** T. SHONO (Proc. World Eng. Congr., 1929, 31, 533—536).—From a mixture of  $PhOH$ , aq.  $CH_2O$ , and 29% aq.  $NH_3$  after 24 hr. at  $35^\circ$  there are isolated 2:2'- and

4:4'-dihydroxydibenzylamines, m.p. 168° and 137—138°, respectively, and after heating to 140° the resin contains a compound,  $C_{14}H_{15}O_2N$ , which is probably  $\omega$ -amino-*o*-tolyl *o*-hydroxybenzyl ether. C. HOLLINS.

**Deterioration of structures.**—See IX. **Corrosion prevention.**—See X. **Varnishing of goloshes.**—See XIV.

## PATENTS.

**Production of [inert] pigment.** D. V. AULT (U.S.P. 1,784,411, 9.12.30. Appl., 4.2.29).—A clay-like material (found native in Manheim, Pa.) containing finely-divided sericite is dried and the sericite air-separated from grit. S. S. WOOLF.

**Ink.** C. E. BIVINS (U.S.P. 1,787,233, 30.12.30. Appl., 6.4.25).—Mixtures of colouring matter, ingredients for giving permanency to writings made with the ink, *e.g.*, tannic acid, gallic acid,  $FeSO_4$ , one or more cryst. org. acids, *e.g.*, citric or salicylic, for avoiding undue precipitation of sediment,  $BzOH$  as preservative, and  $H_2O$ ,  $EtOH$ , and glycerin to reduce the other constituents to paste are claimed. S. S. WOOLF.

**Converting turpentine and pine-tar oils into heavier oils.** E. B. SMITH, ASSR. to E. W. COLLEDGE (for NAT. TURPENTINE PRODUCTS CO.) and AMER. TURPENTINE & TAR CO., LTD. (U.S.P. 1,784,949, 16.12.30. Appl., 6.6.27).—Turpentine etc. is refluxed while allowing both the vapours and returning condensate to pass over fuller's earth until the requisite viscosity and sp. gr. are attained. The product has drying properties. E. LEWKOWITSCH.

**Treatment of rosin.** R. C. PALMER, ASSR. to NEWPORT CO. (U.S.P. 1,787,281, 30.12.30. Appl., 28.6.28).—An original rosin product in liquid phase, *e.g.*, a solution of wood rosin, is treated with caustic alkali in amount required to neutralise approx. 6% of the abietic acid content of the rosin. The homogeneous mixture of abietate and rosin obtained therefrom is free from crystallising tendency. S. S. WOOLF.

**Cracking of rosin.** G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,783,230, 2.12.30. Appl., 20.12.20. Renewed 15.3.28).—A mixture of rosin and  $H_2O$  is heated to above 370° under pressure (1000 lb./sq. in. or more), whereby at least 20% of the solid material is converted into oily constituents of  $d < 0.875$ . S. S. WOOLF.

**Improvement of agatho-copals.** W. DUX (B.P. 350,764, 10.7.30. Ger., 9.11.29).—Such copals, *e.g.*, Manila or kauri, are incorporated with polyhydroxy-fatty acids, *e.g.*, trihydroxystearic acid, by direct heating or by heating and then acidifying mixed solutions of the copal and fatty acid in alkali. The product is  $EtOH$ -sol. and can be used as a shellac substitute. S. S. WOOLF.

**Crystallising varnishes.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & CO. (B.P. 350,641, 9.4.30).—"Glyptal"-type resins, containing drying oil acids if desired, are dissolved in hydrocarbon solvents, a bodied (*e.g.*, blown) tung oil is added, and driers and volatile aliphatic thinners are incorporated as necessary. S. S. WOOLF.

**Drying of [insulating] varnish films.** P. B. COCHRAN, ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,786,824, 30.12.30. Appl., 16.1.28).—Fibrous insulated conductors are preheated in ozonised air (0.1–2%  $O_3$ ) at 70–135° for  $\frac{1}{2}$ –32 hr. and after cooling to 70° are impregnated with insulating varnish and the heating is continued until the varnish is dry. The  $O_3$ -saturated state of the fibre hastens the oxidation of the drying oil in the varnish. S. S. WOOLF.

**Coating composition.** C. H. SIEVER (U.S.P. 1,785,367, 16.12.30. Appl., 2.2.27).—Rubber is heated with an acid reagent of formula  $R \cdot SO_2 \cdot X$ , where R is an org. radical or a OH group, and X is a halogen or OH group. A solution in  $C_6H_6$  or PhMe of the artificial isomeride of rubber thus obtained is added to a solution of low-viscosity nitrocellulose, the solvent of which contains PhMe. S. S. WOOLF.

**Coating composition.** R. H. KIENLE and L. V. ADAMS, ASSRS. to GEN. ELECTRIC CO. (U.S.P. 1,783,364, 2.12.30. Appl., 29.1.27. Renewed 9.4.30).—Unslaked  $CaO$ , soda ash, soda-lime,  $Ba(OH)_2$ , etc. are suspended in granular form in solutions of "alkyd" resins in  $COMe_2$ ,  $EtOH-C_6H_6$ , glycol diacetate, butyl phthalate, etc. After agitation to permit neutralisation of the free acid constituents of the resin, the clear separated solution will not coagulate with basic or other pigments or fillers. S. S. WOOLF.

**Coating compositions.** IMPERIAL CHEM. INDUSTRIES, LTD., ASSEES. of E. F. ARNOLD (B.P. 350,463, 7.12.29. U.S., 7.12.28).—"Glyptal"-type resins of high drying oil content are incorporated with low-viscosity nitrocellulose in the absence of softeners, and pigments, solvents, and diluents are added if desired, the products being used as striping enamels etc. S. S. WOOLF.

**Resinous condensation products.** IMPERIAL CHEM. INDUSTRIES, LTD., and N. STRAFFORD (B.P. 350,896, 15.2.30).—Thermo-hardening compositions (*e.g.*, for stoving insulating varnishes) are produced by heating a phenol and  $CH_2O$  with tung or castor oil (with or without rosin) in a closed vessel at 100–150° in the absence of a catalyst, the reaction product being dehydrated by heating in vac. up to 100°. E. LEWKOWITSCH.

**Preparation of resitols.** A. NOWACK A.-G., and R. HESSEN (B.P. 350,427, 3.3.30. Ger., 7.2.30).—Solid  $PhOH-CH_2O$ -type condensation products in the "resol" stage are converted into "resitols" by heating in thin layers at 100–200° for one or more short periods, *e.g.*, 15–30 sec., and rapidly cooling. Most of the volatile constituents are removed, whilst the proportion of "resol" remaining assists the further working up of the "resitol." Hardening agents such as  $CH_2O$  or hexamethylenetetramine may be used if desired. S. S. WOOLF.

**Resinous composition.** T. F. BRADLEY, ASSR. to ELLIS-FOSTER CO. (U.S.P. 1,785,930, 23.12.30. Appl., 5.6.25).—A polyhydric alcohol, *e.g.*, glycerol, a polybasic carboxylic acid, *e.g.*, phthalic anhydride, and a hydroxy-fatty acid glyceride, *e.g.*, castor oil, are heated together at 200–300°. A monobasic acid, *e.g.*,  $BzOH$ , or rosin may also be incorporated. Pale resins compatible with nitrocellulose are obtained. S. S. WOOLF.

**Manufacture of condensation products [esters for varnishes etc.].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 350,992, 16.12.29).—Sorbitol, or a mixture of it with other polyhydric alcohols (excluding carbohydrates) containing more than three OH groups, is condensed with higher fatty acids; the products may be polymerised, oxidised, etc. as desired. The esters so obtained from linoleic acid have a high viscosity and dry more quickly than does linseed oil. E. LEWKOWITSCH.

**Moulding mixtures.** BAKELITE CORP., Assees. of C. A. NASH (B.P. 350,899, 7.3.30. U.S., 9.3.29).—Articles moulded from mixtures consisting of an absorbent filler and a phenolic resinoid, with which 3–5% of an insol. liquid, e.g.,  $H_2O$  (which is capable of swelling the material of the filler), has been incorporated, do not crack or distort when exposed to liquids.

E. LEWKOWITSCH.

**Treatment of filling and reinforcing materials used in plastic compositions.** IMPERIAL CHEM. INDUSTRIES, LTD., A. R. STEELE, and A. STEWART (B.P. 350,895, 14.2.30).—Wood meal etc. (free from  $CaO$  and hypochlorites etc.) is treated with a simple alkaline solution, e.g., aq.  $NaOH$ , and after removal or neutralisation of the free alkali is incorporated in a synthetic resinous product, giving a composition of improved bulk density. E. LEWKOWITSCH.

**Production of cellulose lacquer [lettered] surfaces.** C. S. JOHNSTONE (B.P. 351,183, 10.5.30).

**Sulphate-cellulose.**—See V. Cores for castings.—See X.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Behaviour of soot as a compounding ingredient for rubber relative to its adsorption of methyl-violet.** E. VON REUTER (Kautschuk, 1931, 7, 132–134).—Various forms of lampblack or C black influence the tensile strength of rubber not merely by surface phenomena; chemical influence on the vulcanisation process is also responsible. The surface activity of the C is measured most conveniently by adsorption of methyl-violet from aq. solution; the degree of “wetting,” however, in these circumstances is greater than that obtaining when such amorphous C is mixed into rubber in the ordinary manner, but the test with methyl-violet places a range of samples in substantially the same order as their reinforcing effect in rubber. Assuming that each type of amorphous C consists of cylindrical particles of uniform size, it is possible from the apparent density and the surface vol. (indicated by the adsorptive power) to calculate for each the diameter and length of the particles and the number per g. D. F. TWISS.

**New condensation products of rubber hydrocarbons with the aid of benzyl chloride.** F. KIRCHHOF (Kautschuk, 1931, 7, 128–132).—The application of the Friedel-Crafts reaction by the rapid addition of a suspension of  $AlCl_3$  in  $CCl_4$  to a cooled solution of rubber and benzyl chloride in the same solvents yields a pale yellow, amorphous, feebly thermoplastic product of  $d 1.10$ , approx. composition  $(C_{19}H_{20})_x$ , believed to be *benzylidenecaoutchouc*. If, however,

the benzyl chloride is introduced into a solution of rubber in  $CCl_4$  containing suspended  $AlCl_3$ , the product appears to be *cyclocaoutchouc*, the formation of which prevents further reaction with the benzyl chloride. The *benzylidenecaoutchouc* products contain between 3 and 5 benzylidene groups to one isoprene nucleus; their chemical, thermochemical, and physical properties are distinct from those of a mixture of polybenzylidene and *cyclocaoutchouc*. Structural formulæ are tentatively suggested for the polybenzylidene and *benzylidenecaoutchouc* hydrocarbons. D. F. TWISS.

**Varnishing of goloshes.** H. KOCH (Gummi-Ztg., 1931, 45, 1683–1686).—A description of the nature, production, and use of sulphurised linseed oil varnish for rubber over-shoes together with an indication of possible working difficulties and their remedies.

D. F. TWISS.

**X-Rays in research.**—See XI.

#### PATENTS.

**Vulcanisation of rubber.** S. M. CADWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,777,960, 7.10.30. Appl., 1.2.21).—Rubber is vulcanised at low temp. with the aid of a powerful combination of vulcanising ingredients introduced in such a way that only a part of the entire combination is present in any one portion of the rubber until after the preliminary mixing operations; the vulcanising combination is then completed and vulcanisation effected without further altering the form of the mass. E.g., two mixings may be made containing: (a) rubber 100,  $ZnO$  10, “oxy-*n*-butylthiocarbonic acid disulphide” 6, S 3 pts.; (b) rubber 100,  $ZnO$  10, S 3, aniline 4 pts. On bringing these mixings into contact when desired, e.g., by milling, or by superposing alternate layers of each, vulcanisation ensues in about 1 day at room temp. An alternative method of effecting vulcanisation is to submit the first of the above mixtures to an atm. containing aniline for 4 days or to treat it with liquid aniline for 1 day. Another pair of mixings suitable for joint use contains: (a) rubber 100,  $Zn$  butylxanthate 3 pts.; and (b) rubber 100,  $ZnO$  10, S 3, *p*-toluidine 4 pts. D. F. TWISS.

**[Metal-foil]-lined rubber tubing and its manufacture.** L. RADO (B.P. 352,198, 4.6.30. Ger., 7.11.29).

**Rubberised fabrics. Treatment of textiles.**—See VI. Coating compositions.—See XIII.

#### XV.—LEATHER; GLUE.

**Physical properties of sole leather.** C. SCHIAPARELLI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 94–199).—A general discussion, under the headings: generalities, mechanical testing, vegetable tanning, manufacture, “standard” sole leather,  $H_2O$ -solubles, permeability, resistance to wear and apparatus for measuring it, sp. gr., finishing, hardness, flexibility, cracking, secondary properties, tanning number, microscopical examination, and sampling. T. H. POPE.

**Free [mineral] acids in the analysis of vegetable [tanned] leathers.** I. V. KUBELKA and R. WOLLMARKER (Collegium, 1931, 96–111).—Free mineral acid was not found when the Innes, Procter-Searle, and Balland-Maljean methods for its determination

were applied (a) to leathers prepared from hides limed with  $\text{Na}_2\text{S}$ - $\text{CaO}$  liquors and/or delimed with  $\text{HCl}$  and tanned, respectively, with extracts free from sulphites, or (b) to similar leathers, which had absorbed  $< 0.5\%$   $\text{SO}_3$ . Positive results were given by the above methods of analysis on leathers tanned with synthetic tans and sulphited quebracho extract, or by the former leathers if they had been so treated as to contain  $> 0.5\%$   $\text{SO}_3$ . Negative results were obtained by the van der Hoeven (B., 1923, 109 A) and von Schroeder (B., 1930, 574) methods of determining free mineral acid on the leathers which had been tanned with sulphited extracts and synthetic tannins, but on which no  $\text{H}_2\text{SO}_4$  had been employed, and a higher result for the free mineral acid in leathers which had been treated with  $\text{H}_2\text{SO}_4$  was provided by these methods than by the former ones. The results were all lower than the actual amount of acid which had been absorbed by the leathers.

D. WOODROFFE.

**Determination of fat and moisture in leather.**

B. AVENATI-BASSI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 200—209).—For extracting fat from leather  $\text{CCl}_4$  serves as well as light petroleum. In drying the residue left after distillation of the bulk of the solvent from the fat, most of the solvent is eliminated during the first 5 hrs.' heating at  $100^\circ$ , increase in the wt. due to oxidation of the fat, followed by slight diminution to const. wt., then taking place. Chambard's method for determining the extraneous matters dissolved from the leather with the fat is modified by dissolving the extracted fat in light petroleum instead of in  $\text{COMe}_2$ ; the solution thus obtained may be washed with  $\text{H}_2\text{O}$ , which dissolves the extraneous matters, and two layers, readily separable, are formed.

T. H. POPE.

**Determination of fatty substances in products used in tanning and in finished tanned materials.** S. CAMILLA (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 236—239).—The modification of Marchand's method (B., 1931, 133) may be applied to the determination of fat in leathers and in egg-yolk, degreas, and other fatty materials used for treating hides. The material, dehydrated if necessary with calcined gypsum, is left in contact with  $\text{Et}_2\text{O}$  for 15—20 hr., 10 c.c. of the ethereal extract being then treated in the lacto-butyrometer with 10 c.c. of 95%  $\text{EtOH}$  and 10 c.c. of  $\text{H}_2\text{O}$  rendered alkaline with 2 drops of 2*N*- $\text{Na}_2\text{CO}_3$ . If 10 g. of the comminuted leather are used, failure of the ethereal fat layer to separate in the lacto-butyrometer indicates sole leather, in which only 1% of fat is allowed. The above procedure gives results in good agreement with those of the more prolonged official method, in which the material is extracted with light petroleum in a Soxhlet apparatus. T. H. POPE.

**Quantitative analysis of tanning substances.**

G. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 82—89).—Details of the procedure to be followed in determining insol. matters and non-tans are given, together with the results obtained on various tanning materials by different methods. T. H. POPE.

**Qualitative detection of sulphiting in tannin extracts.** F. STATHER and R. LAUFFMANN (Collegium, 1931, 314—315).—2 g. of the extract are dissolved in

25 c.c. of  $\text{H}_2\text{O}$ , treated with 5 c.c. of 30%  $\text{NaOH}$ , boiled for 10—15 min., neutralised with  $\text{H}_3\text{PO}_4$ , heated to boiling with 10 c.c. of dil.  $\text{H}_3\text{PO}_4$ , and the vapour passed through a mixture of 2 c.c. of 1-KI solution (1% I), 2 c.c. of  $\text{BaCl}_2$  solution (10%), and 10 c.c. of  $\text{H}_2\text{O}$  for 3 min. Sulphiting or the presence of sulphites in the extract is indicated by the formation of a turbidity or a ppt. of  $\text{BaSO}_4$  in the solution on keeping.

D. WOODROFFE.

**Acidity of tanning extracts.** F. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 223—235).—By means of apparatus and methods described, measurements of  $[\text{H}^+]$  and electrometric titrations of extracts of a number of tanning materials have been made.

T. H. POPE.

**Leather varnishes.**—See XIII.

## PATENTS.

**Obtaining gelatinising colloids in the form of little plates, sticks, or the like.** F. SELTSAM NACHFOLGER A.-G. FÜR CHEM. IND., G. SANDER, and T. HOFFMANN (B.P. 350,962, 15.3.30).—Glue, gelatin, dextrin, etc. are obtained in the form of small plates and the like by allowing thin jets of the colloid liquor to flow on to an endless, smooth, travelling surface free from grooves, notches, or ribs, passing round cooling drums; the plates etc. are cut off the solidified strips.

E. H. SHARPLES.

**Protecting animal fibre.**—See VI.

## XVI.—AGRICULTURE.

**Significance of the lime and acid status of soils and its determination by electrometric titration.** S. GOY (Ergeb. Agrik.-chem., 1930, 2, 35—52; Bied. Zentr., 1931, 60A, 234—235).—A summary and discussion of earlier papers by the same author.

A. G. POLLARD.

**Manurial experiment with cacao.** T. H. HOLLAND (Trop. Agric., 1931, 74, 263—268).—Phosphatic and potash manures had little influence on the crop yield of cacao or on the incidence of pod canker.

A. G. POLLARD.

**Fertilisation of grass with liquid manure. II.** E. RITTER (Landw. Jahrb. Schweiz, 1930, 44, 641—706; Chem. Zentr., 1931, i, 1961).—Clover was favoured by K and P; high N applications often caused the complete disappearance of clover and markedly increased the growth of grass. The K and P requirement was calc. from the composition of the crop. A. A. ELDRIDGE.

**Relation of composition of cultivated and wild green-manure plants to decomposition of the nitrogenous constituents.** H. MISU, I. OMAI, and T. HIBINO (Ann. Agric. Exp. Sta. Chosen, 1930, 5, 1—126).—The plants were fermented for 48 days under conditions simulating those in soil with upland and lowland systems of culture. Max. ammonification is favoured by increase in total N, ash,  $\text{CaO}$ , or  $\text{CaO} + \text{MgO}$ ; nitrification by that in total N,  $\text{K}_2\text{O}$ , and  $\text{CaO} + \text{MgO}$ ; and titratable acidity by that in total N and  $\text{CaO} + \text{MgO}$ . Disturbance in the attainment of maxima was caused by increase in crude fibre, N-free extract, and N-free extract/protein.

CHEMICAL ABSTRACTS.

**Changes in the nitrogen of green plants.** G. KLEIN (Ergeb. Agrik.-chem., 1930, 2, 143; Bied. Zentr., 1931, 60A, 200).—In special sterile-culture apparatus plants assimilated  $\text{NH}_4$  salts and utilised these directly for protein production. Amino-acids were absorbed by the plants, but were not equally utilisable, glycine and alanine being inferior to asparagine and aspartic and glutamic acids in this respect. Nitrates were, in part, reduced in the plant roots and the N was translocated in org. combination. Protein decomp. was of a varied nature. The principal N-decomp. product was  $\text{NH}_3$ , the toxic action of which was prevented by its combination with org. acids. Urea, principally as ureide, was also detected and amines occurred in the flowers and, to a smaller extent, in the leaves.

A. G. POLLARD.

**Possible effect of fungicides on the composition of apples.** W. A. DE LONG and A. D. PICKETT (Science, 1931, 73, 649–650).—A slightly lower reducing power has been detected in apples sprayed with S fungicides during the growing season as compared with unsprayed fruit or with that treated with Bordeaux mixture. Variations in acidity showed no definite trend.

L. S. THEOBALD.

**Fungicidal action of sulphur. II. Production of hydrogen sulphide by sulphured leaves and spores and its toxicity to spores.** S. E. A. McCALLAN and F. WILCOXON (Contr. Boyce Thompson Inst., 1931, 3, 13–38; cf. A., 1930, 1068).—Formation of  $\text{H}_2\text{S}$  from S occurred in the presence of leaf tissue and fungus spores (0.002 mg.  $\text{H}_2\text{S}$  per hr. per sq. dm. of leaf surface of strawberry and up to 9.8% of the wt. of spores in 12 hr.). The optimum temp. for  $\text{H}_2\text{S}$  production was  $35^\circ$  and the action ceased at  $60^\circ$ .  $\text{H}_2\text{S}$  was formed at  $p_{\text{H}}$  4.0–8.0 with no definite optimum. Actual contact of S and spores was not necessary, the action taking place through a membrane or over an air space of 3–4 mm. The gas appeared on the spores and not on the S. Substances having the S-reducing SH group were detected in certain fungus spores. The sensitivities of various spores to injury by S and by  $\text{H}_2\text{S}$  were of the same relative order. On the basis of units of toxicity ( $\text{H}_2\text{S}$  required to reduce germination to 50%) spores sensitive to S produce more, and S-resistant spores less, than 1 unit. The diffusion of S vapour from S to the spores and its reduction on or in the spores is postulated.

A. G. POLLARD.

**Efficiency of contact insecticides. I. Surface forces as related to wetting and tracheal penetration.** F. WILCOXON and A. HARTZELL (Contr. Boyce Thompson Inst., 1931, 3, 1–12).—Many spray liquids do not form a film over an insect nor penetrate the tracheal tubes in the absence of spreading agents. The toxicity of nicotine solutions to *Aphis rumicis* varied with the spreader used, the efficiencies being in the order Ca caseinate < Penetrol < Na oleate. The spreading coeffs. of the solutions as determined by measurements of surface tension and angles of contact were in the same order. Spray solutions, even when containing soap, did not penetrate the trachea of dead insects.

A. G. POLLARD.

**Fertilisation experiments with phosphorites.** G. TOMMASI and S. D. DI DELUPIS (Ann. Staz. chim.-

agrar. sperim. Roma, 1930, [ii], 13, 39 pp.; Chem. Zentr., 1931, i, 2104).

**Ammonia liquor.**—See II. Thermo-phosphates. —See VII.

## PATENTS.

**Superficial treatment of granular chemical fertilisers.** A. MENTZEL (B.P. 351,098, 26.3.30. Ger., 26.3.29).— $\text{NH}_4\text{HCO}_3$  crystals are fed from a hopper on to a rotating disc which throws the crystals against a spray of NaCl fed in an annular disperse stream round the disc so that the surface of the crystals becomes coated with a thin protective layer of  $\text{NaHCO}_3$  containing a little  $\text{NH}_4\text{Cl}$ . The remainder of the latter formed by the reaction is obtained as a solution when the treated crystals are drained.

A. R. POWELL.

**Apparatus for manufacture of nitrogenous fertilisers.** ADCO, LTD., and J. ASHEN (B.P. 341,425, 14.10.29).—The apparatus comprises a conveyor belt, on which is fed the org. material, e.g., straw, to be treated, and a hopper above the belt for delivering a thin even layer of nitrogenous material over the straw. The hopper is provided with two rollers, one of which is axially grooved to deliver the material in fine streams, and below the hopper is arranged a series of jets for spraying  $\text{H}_2\text{O}$  into the falling stream of N-material. The apparatus is intended for the manufacture of fertilisers from straw by the processes described in B.P. 152,387 and 219,384 (B., 1920, 827 A; 1924, 810).

A. R. POWELL.

**Insecticidal, insect-repelling, and fungicidal compositions.** C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 350,897, 15.2.30).—The prep. consists of petroleum white oil, an emulsifying agent such as an alkali sulphonate derived from petroleum, and an extract of a plant having insecticidal properties, e.g., "fish poisons" (*Derris*, cubé, etc.) or pyrethrum and the like. The product may be diluted with  $\text{H}_2\text{O}$  and anti-oxidation catalysts may be added.

E. H. SHARPLES.

## XVII.—SUGARS; STARCHES; GUMS.

**Oxidation of dextrose, lævulose, and sucrose with bleaching powder.** S. OCHI and M. NAKAMURA (Proc. World Eng. Congr., 1929, 31, 537–542).—At  $15$ – $45^\circ$  aq. bleaching powder oxidises dextrose to the extent of 94%, lævulose 37%, sucrose 43–46%; it is possible to oxidise dextrose selectively in admixture with lævulose (in invert sugar) or sucrose. C. HOLLINS.

**Influence of sodium chloride on the determination of starch by the fermentation method.** B. LAMPE and W. KILP (Z. Spiritusind., 1931, 54, 167–168).—When starch in potato flakes is determined from the yield of EtOH produced by a fermentation for 72 hr. of the starch hydrolysed by approx. 2.5% HCl and subsequently neutralised by NaOH, the value is approx. 2–3% lower than when the starch is hydrolysed by a malt infusion. The decreased yield of EtOH with the acid-inversion method is due to the inhibiting effect on the fermentation of the 3.5% of NaCl produced by the neutralisation. The yield of EtOH is not affected by higher rates of seeding yeast, but decrease in the amount of HCl and greater dilution of the mash before fermentation



yields an amount of EtOH equal to that given by the enzymic method.

C. RANKEN.

**Determination of the starch value and distillation value of potato flakes.** B. LAMPE and W. KILP (Z. Spiritusind., 1931, 54, 173—174).—Provided the potato flakes are fresh and normal, their starch values as determined from the yield of EtOH by fermentation of the flakes hydrolysed by the modified acid-inversion method are the same as those given by the mash method of hydrolysis (distillation values), but with old and especially with over-roasted flakes the yield of EtOH is 5—10% higher with the acid-inversion method, so that the distillation value cannot be calc. from the starch value.

C. RANKEN.

**Preparation of soluble starch with hypochlorites.** B. RASSOW (Textilber., 1931, 12, 468).—Solutions of NaOCl react with starch, thereby liberating heat and modifying the starch so that the viscosity of its aq. solution is much less than that of the untreated starch; the treated starch also has increased reducing and acidic properties. Untreated and treated starches may be well coloured by basic dyes (the untreated starch better than the treated), but acid and direct dyes have little affinity for either starch; treated starch gives very level shades with direct dyes.

A. J. HALL.

PATENT.

**Cryst. sugar.**—See I.

## XVIII.—FERMENTATION INDUSTRIES.

**Production of malt and wort.** L. R. BISHOP (J. Inst. Brew., 1931, 37, 345—359).—With "stocking malts" made under average malting conditions, the amount of permanently sol. N in the standard laboratory hot mash prepared from English 2-rowed malt is 35% of the total N content of the original dry barley. With 6-rowed barleys the value is approx. 29%. For "bulk malt" the corresponding values are 33 and 28%, respectively, for normal modification. The higher permanently sol. N of malts with higher N content is due to increased amounts of "peptide" and "undetermined" N. With rising mashing temp. the contents of ammonia-, amide-, and amino-N are const., whilst that of peptide-N and, consequently, the total sol. N increases to a broad optimum zone at 50°. The amount of "apparent maltose" in the wort increases rapidly from 40° to a sharply defined optimum at 60°, and then rapidly falls. The content of dextrin rises more slowly to an optimum above 70°. The content of "peptide N" obtained from 6-rowed barley is smaller than that from 2-rowed barley.

C. RANKEN.

**Metallic contamination of beer.** G. HAGUES (J. Inst. Brew., 1931, 37, 366—372).—The first-runnings of beer through a bottling plant constructed of Cu contained a max. of 1.9 grains Cu per gal. due to the corrosion of the Cu piping and Cu gauze in the filter, which were kept bright by sand and H<sub>2</sub>SO<sub>4</sub>. In the later runnings the Cu content decreased to 0.1 grain per gal. Corrosion was diminished if the plant was cleansed by brushing and sterilised by steam. Ni fermentation vessels were corroded where the yeast head was in contact with the metal. The resulting beers contained 0.12—0.17 grain Ni per gal. and the yeast 0.14—1.0 grain per lb. With fermentation vessels constructed of monel metal the content of Ni in the beers was reduced to 0.02—0.04

grain per gal. and that of the yeast to 0.05—0.11 grain per lb.

C. RANKEN.

**Solid carbon dioxide from by-product fermentation gas.** C. L. JONES (Ind. Eng. Chem., 1931, 23, 798—800).—The waste gases from starch fermentation contain 60% CO<sub>2</sub> and 40% H<sub>2</sub>. They are stripped from solvents in an adsorption purifier, the CO<sub>2</sub> is absorbed in H<sub>2</sub>O under pressure, and the remainder used for MeOH synthesis. Odour is removed by the Reich system from the separated CO<sub>2</sub>, which is of 96—97% purity, and H<sub>2</sub> from the top of a refrigerated fractionating column. The plant is designed for all-the-year-round operation with a large storage for the winter's output. The evaporation loss from this when full is less than 0.1% per day. The supply of these waste gases is much in excess of the present demand for solid CO<sub>2</sub>.

C. IRWIN.

**Determination of lactic acid in wine in presence of sugar.** P. BERG and G. SCHULZE (Wein u. Rebe, 1931, 12, 433—445; Chem. Zentr., 1931, i, 2131).—Use of BaCO<sub>3</sub> instead of Ba(OH)<sub>2</sub> during evaporation avoids formation of lactic acid from sugar, but permits conversion of anhydride into acid. In ashing the alcoholic solution after removal of Ba, ignition at too high a temp. must be avoided. Tillmans and Weil's stepwise method of titration is criticised.

A. A. ELDRIDGE.

**Dehydration of alcohol under pressure.** W. SCHNACK (Z. Spiritusind., 1931, 54, 179—180).—Abs. EtOH is manufactured economically by heating the raw spirit with CaO in an autoclave at 4 atm. and subsequently distilling in a vac. the EtOH from the residual CaO. By suitable modifications only 50 kg. of steam are required to yield 100 litres of abs. EtOH. In the azeotropic method, the dehydrating power of benzene and benzol is doubled at 10 atm., and less of these liquids is required. With 94% raw spirit the amount of steam required for the manufacture of 100 litres of abs. EtOH is reduced from 160 to 130 kg. by the use of pressure.

C. RANKEN.

**Determination of starch. Starch in potato flakes.**—See XVII.

PATENT.

**Protection of enzymes against the damaging action of metals.** S. SOKAL. FROM KALLE & Co. A.-G. (B.P. 351,315, 22.8.30).—Enzymes are protected against the inhibiting action of metals by the addition of compounds having the atomic grouping ·N·C·S, e.g., thio-carbamide or KCNS.

C. RANKEN.

## XIX.—FOODS.

**Proteins of vitreous barley.** E. EHRLICH and E. KNEIP (Z. ges. Brauw., 1931, 54, 1—7, 9—13; Chem. Zentr., 1931, i, 2129).—The hordein content appears chiefly to determine the vitreous condition.

A. A. ELDRIDGE.

**Wheat, flour, and their by-products.** P. BRUÈRE (J. Pharm. Chim., 1931, [viii], 14, 5—22).—Methods of milling, as carried out in France, are explained, and the chemistry and analysis of wheat and flour are described. For the determination of cellulose 5% HCl, followed by 10% KOH, is employed, and for that of acidity the flour is extracted with 90% EtOH.

T. McLACHLAN.

**Composition of kitchen waste.** B. THOMAS and J. HARGRAVE (J. Min. Agric., 1931, 38, 366—373).—

Kitchen refuse from different sources varies considerably in composition, but represents broadly a food of the same type, *i.e.*, a medium protein concentrate containing excessive amounts of oil. E. LEWKOWITSCH.

**Corrosion of tinsplate by fruit juice.**—See X. **Rancidity test for fats etc. Edible oil preservation. Sapote oil.**—See XII. **Manures for cacao.**—See XVI. **Air conditioning.**—See XXIII.

## PATENTS.

**Pasteurising.** F. B. DEHN. From BARRY-WEH-MILLER MACHINERY Co. (B.P. 350,627, 2.4.30).—In a process of pasteurising milk in bottles on a zig-zag chain conveyor, a large quantity of  $H_2O$  is used to effect rapid cooling, and part only of this  $H_2O$  passes on to the pasteurising and preheating compartments, counter-current to the milk. B. M. VENABLES.

**Milk preparation and its production.** W. R. B. ST. J. GATES, J. TAVROGES, and COW & GATE, LTD. (B.P. 350,670, 5.5.30).—Milk powder, sugar, and  $H_2O$ , with or without an emulsifying agent, are mixed and evaporated in vac. to a consistency suitable for pressing into moulds. E. B. HUGHES.

**Production of solid milk and cream preparations.** P. BORN, Assr. to N.V. NEDERLANDSCHE GRUYÈRE-BLOKMEK FABRIEK (U.S.P. 1,786,559, 30.12.30. Appl., 26.2.26. Ger., 27.2.25).—Milk or cream, with or without added sugar, is condensed in vac. to form a mass capable of being pressed into blocks, which may be coated with a protecting material such as cacao butter. E. B. HUGHES.

**Continuous production of ice-cream and other [edible] plastic substances.** VOGT INSTANT FREEZERS Inc., Assees of C. W. VOGT (B.P. 350,660, 22.4.30. U.S., 13.5.29).—Ice-cream (etc.) is produced from the liquid state by passing along tubes subjected to the action of a temp.-changing medium, the temp. of which is controlled by the resistance offered by the plastic substance being treated. E. B. HUGHES.

**Manufacture of chocolate.** V. WAYAFFE (B.P. 351,928, 30.12.29).

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Improvement of tobacco. Taste improvement and bleaching.** H. JORDT (Chem.-Ztg., 1931, 55, 462—463).—Attempts to bleach tobacco after fermentation either are too costly ( $H_2O_2$  or  $O_3$ ) or spoil the flavour ( $SO_2$ ,  $HNO_3$ ,  $Cl_2$ ). Removal of  $SO_2$  etc. by warming the treated tobacco slowly to 70—90° (rapid fermentation temp.) failed to remove the S compounds, which are apparently a cause of the bad flavour. On the other hand, fermentation in presence of  $O_3$  gives good results. Under carefully regulated conditions of moisture, temp., and  $O_3$  concentration, a golden-yellow to clear brown tobacco with a mild, pleasant flavour was obtained. It is possible that nicotine is partly or wholly eliminated. In absence of  $O_3$  the tobacco was black-brown. Tobacco fermentation (which is almost without effect on the essential tobacco constituents) appears to include a reducing process resulting in the production of dark colouring matters difficult to remove subsequently; the formation of these is prevented by

the  $O_3$ . The drying of the green tobacco leaf in presence of  $O_3$  before fermentation is also suggested.

C. HOLLINS.

**Examination of myrrh.** P. BOHRISCH (Pharm. Ztg., 1931, 76, 787—790, 800—802).—The history, origin, properties, purification, and uses of myrrh are described together with the physical and chemical characteristics and colour reactions of 9 different varieties and of Bisabol myrrh and Bdellium. Methods for their differentiation are suggested and compared.

E. II. SHARPLES.

**Preparation of Tinctura Chinæ composita by diacolation.** II. BREDDIN (Pharm. Ztg., 1931, 76, 802).—Procedure for successful diacolation is described.

E. II. SHARPLES.

**Examination of soap-containing spirituous [medicinal] preparations.** W. MEYER (Chem.-Ztg., 1931, 55, 518—519).—Exact and routine methods for the analysis of such preparations are described. Commercial samples are shown to vary considerably in composition; some contained linseed oil instead of olive oil, although conforming outwardly to the scanty requirements of the D.A.B. More stringent tests should be officially specified (cf. B., 1930, 1045).

E. LEWKOWITSCH.

**Hungarian coriander oil.** P. S. JÓNÁS (Magyar Gyóg. Társas. Ert., 1931, 7, 24—29; Chem. Zentr., 1931, i, 2129).—The average essential oil content is 0.17—0.49%. For the determination 500 g. are distilled with steam and the oil is weighed; the method of D.A.B. VI gives low results. A. A. ELDRIDGE.

## PATENTS.

**Manufacture of quinoline and acridine compounds [bactericides].** I. G. FARBENIND. A.-G. (B.P. 347,515, 29.5.30. Ger., 10.6.29).—Benz-thiazoles, -oxazoles, and -iminazoles carrying in 2-position a substituted quinoline or acridine residue, and having at least one  $NH_2$  group, are valuable bactericides, especially against spherobacteria. 4-Chloro-6-(6'-methyl-2'-benzthiazolyl)quinaldine, m.p. 196—197°, obtained from dehydrothiitoluidine by condensation with Et acetoacetate followed by treatment with  $POCl_3$ , is condensed with alcoholic  $NH_3$  to give the 4-amino-compound, m.p. 297—298°; the 4-ethylamino- (m.p. 204°), piperidino- (m.p. 166°),  $\gamma$ -diethylamino- $\beta$ -hydroxy-*n*-propyl- (m.p. 180°), *p*- $\beta$ -diethylaminoethoxyanilino- (m.p. 215°), and carboxymethylamino- [hydrochloride, m.p. 300° (decomp.)] compounds are similarly prepared. Other examples are: 4-amino-6-(6'-methoxy-2'-benzthiazolyl)quinaldine, m.p. 272°, and the 4-isoamylamino-compound, m.p. 149—150°; 4-amino-6- and -7-(6'-ethoxy-2'-benzthiazolyl)quinaldines, m.p. 268° and 236°, respectively; 10-amino- (m.p. above 300°) and 10- $\gamma$ -diethylamino- $\beta$ -hydroxy-*n*-propylamino- (m.p. 172°) derivatives of 8-(6'-methyl-2'-benzthiazolyl)acridine; 10-amino-8-(6'-ethoxy-2'-benzthiazolyl)acridine, m.p. above 300°; 2-amino- (m.p. 188°) and 2- $\gamma$ -dimethylamino- $\beta$ -hydroxy-*n*-propylamino- (m.p. 137°) derivatives of 4-(6'-ethoxy-2'-benzthiazolyl)quinoline; 4-amino-3-(6'-ethoxy-2'-benzthiazolyl)quinoline, m.p. 248°; 4-amino-6-(6'-methyl-2'-benzoxazolyl)quinaldine, m.p. 288°; 2-ethylamino-4-(6'-methyl-2'-benziminazolyl)quinoline, m.p. 145°;

2-ethylamino-4-(6'-ethoxy-2'-benzthiazolyl)quinoline, m.p. 191°; 8-amino-6-(6'-ethoxy-2'-benzthiazolyl)quinoline, m.p. 176°; 4-amino-6-(6'- $\beta$ -diethylaminoethoxy-2'-benzthiazolyl)quinoline, m.p. above 300°; and the 6'-allyloxy- (m.p. 220—222°) and 6'-isoamyloxy- (m.p. 236—237°) compounds; 4-methylanilino-6-(6'-methyl-2'-benzthiazolyl)quinoline, m.p. 199°; 4-diethylamino-6-(nitro-6'-methyl-2'-benzthiazolyl)quinoline, m.p. 110° (decomp.), and the corresponding amine, m.p. 108°; the 4-amino-compounds, m.p. 270° (decomp.) [glycollate, m.p. 228° (decomp.)] and 325° (decomp.), respectively; 10-amino-8-(nitro-6'-methyl-2'-benzthiazolyl)acridine, m.p. above 300°, and the corresponding diamine, m.p. above 300°. The various intermediates are described. C. HOLLINS.

**[Manufacture of] barbituric acid derivatives.** COMP. DE BÉTHUNE (B.P. 348,140, 5.2.30. Fr., 5.2.29).—Barbituric acids having  $\Delta^2$ -cyclopentenyl and aryl or alkyl groups in position 5 are synthesised by the usual methods. Examples are the Et, m.p. 161—162°, allyl, m.p. 139—140°, phenyl, m.p. 183—184°, compounds and mono- (m.p. 197—198°) and di- (m.p. 156—157°)  $\Delta^2$ -cyclopentenylbarbituric acids. [Stat. ref.] C. HOLLINS.

**Manufacture of solutions of cholesterol or its esters or mixtures of these.** F. PASSER (B.P. 348,082, 28.11.29. Addn. to B.P. 328,922; B., 1930, 626).—Glycol or glycerol is added to solutions prepared as in the prior patent in order to improve the effect on the skin. C. HOLLINS.

**Manufacture of a colloidal silver iodide compound.** H. S. KEELAN, Assr. to E. R. SQUIBB & SONS (U.S.P. 1,783,334, 2.12.30. Appl., 13.1.27).—Gelatin is hydrolysed by boiling with 4% of NaOH in a 3% solution and, after filtration, equiv. quantities of aq. AgNO<sub>3</sub> and NaCl solutions are added simultaneously with const. stirring to produce a colloidal solution of AgCl which is then treated with the equiv. of NaI to convert the AgCl into AgI. The solution is dialysed through collodion membranes to remove Na salts and then evaporated to dryness in vac. The product forms yellow scales readily sol. in H<sub>2</sub>O to give a colloidal solution of high germicidal power. A. R. POWELL.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

**Manufacture of [photographic] bleaching-out layers.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 348,232, 18.2.30).—The light-sensitiveness of azine, oxazine, and other dyes for photographic layers is regulated by hydrogenation or substitution in an aromatic residue external to the chromogenic ring system, so that dyes in a multi-coloured system bleach out at the same or any desired rates. Introduction of H, halogen, CN, CNS, NO<sub>2</sub>, CHO, COR, or CO<sub>2</sub>R increases the sensitiveness; alkyl, aryl, OH, OAr, OR reduce it. In both cases substitution *ortho* to the linking with the chromogen is more effective than *meta*, and *meta* than *para*. To correct the reddish greys due to insufficient sensitiveness of phenylrosinduline (purple component) and too great sensitiveness of 5-anilino-9-dimethylaminopheno- $\beta$ -naphthazoxonium chloride (blue-

green component) in a mixture of these with phenanthra-phenazine methonitrate (yellow component), the first is replaced by *m*-chlorophenylrosinduline, the second by the 5-*o*-anisidino-compound. C. HOLLINS.

**Photochemical production of screen diapositives for production of photomechanical printing surfaces.** DR. BEKK & KAULEN CHEM. FABR. G.M.B.H. (B.P. 351,201, 26.5.30. Ger., 27.5.29).—A AgBr (etc.) gelatin emulsion is reduced in sensitivity by pretreatment with an alkali dichromate solution, so that exposures up to 4 min. may be given. After development of the Ag image the screen points are reduced, but, if necessary, may be re-enlarged by intensification after washing out the unhardened gelatin. Reduction and enlargement are repeatable. J. LEWKOWITSCII.

**Photomechanical screens.** J. HEIDENHAIN (B.P. 351,626, 14.4.30. Ger., 10.8.29).—A transparent plate (glass, celluloid, or "Cellon") is coated with a PbS mirror on which an albumin-(NH<sub>4</sub>)<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> layer is applied. The plate is then exposed through a ruled plate, and developed and etched in the normal manner. J. LEWKOWITSCII.

**Coloured photographic pictures.** H. VON PASSOW and L. BANG (B.P. 352,192, 30.5.30. Ger., 31.5.29).

**[Filter-screen supports for] colour photography and cinematography.** P. W. SMITH (B.P. 351,498, 26.3.30).

**Filters for ultra-violet rays.**—See XI.

## XXII.—EXPLOSIVES; MATCHES.

**Air conditioning.**—See XXIII.

### PATENTS.

**Blasting fuses.** W. ESCHBACH (B.P. 351,930, 22.2.30).

**Nitrocellulose.**—See V.

## XXIII.—SANITATION; WATER PURIFICATION.

**Loss of ammonia nitrogen from [sewage] trickling filters.** W. RUDOLFS and N. CHAMBERLIN (Ind. Eng. Chem., 1931, 23, 828—830).—The loss of NH<sub>3</sub> not accounted for by microbial re-assimilation was not uniform for the different levels of the filters nor affected by the time of contact with the different filtering media. The loss in an experimental stone filter was 14—70% and from a wire-mesh filter 9—41%, a comparatively small quantity of which escapes into the air as NH<sub>3</sub>; the greater loss is in the form of volatile NH<sub>4</sub> compounds and volatile oxides indicated by the decrease in the biochemical O demand. C. A. KING.

**Rise of air conditioning [in chemical operations].** W. L. FLEISHER (Ind. Eng. Chem., 1931, 23, 732—735).—The influence which the control of temp. and humidity of the atm. exerts, not only on human comfort, but on industrial processes, e.g., manufacture of confectionery, matches, textiles, etc., is outlined. C. A. KING.

**Corrosion prevention.**—See X.

### PATENTS.

**Filtration systems. Treatment of flue gases etc.**—See XI. **Glauconite.**—See VII. **Insecticides.** See XVI. **Bactericides.**—See XX.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 11 and 18, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Sight feed for centralising control of distillation equipment.** D. & F. OTHMER (Chem. Met. Eng., 1931, 38, 415—416).—The liquor passes to the column through an orifice in the bottom of a sight feed box. One end of an inverted U-pipe is below the liquor level in the feed box and the other is carried to the central control level and connected with a manometer. By pumping air through a connexion to the pipe the liquor level is indicated on the manometer. A pipe on the feed box acts as a vent for the air.

D. K. MOORE.

**Measurement of small particles.** W. L. GADD (Cement, 1931, 4, 763—767).—A direct method is actually to measure the diameter of a number of particles in one direction only on a microscope slide without reference to the orientation of the particles; the average diameter being obtained measured in several directions, the law of averages equalises the orientation. For fine particles which may be held in suspension in a suitable viscous liquid, *e.g.*, medicinal paraffin or a sugar solution, a definite vol. of the powder suspended in the solution is allowed to flow into a vertical calibrated glass tube, which is illuminated from beneath until opacity is attained, at which point the height of the suspension is noted. The standard normal suspension for comparison consists of 0.5 g. of pulverised quartz sand suspended in 100 c.c. of sugar solution (150 g. of sugar in 100 c.c.  $H_2O$ ). This method is not suitable for cement particles  $>200$  mesh/linear inch.

C. A. KING.

**Influence of the proximity of a solid wall on the consistency of viscous and plastic materials. III.** R. K. SCHOFIELD and G. W. S. BLAIR (J. Physical Chem., 1931, 35, 1212—1215; cf. B., 1930, 844).—Theoretical. A solid wall may modify the consistency of the material at an appreciable distance from it.

L. S. THEOBALD.

**Problems in the conduction of heat.** G. GREEN (Phil. Mag., 1931, [vii], 12, 233—255).—Mathematical.

**Polariscopes.**—See VIII. **Electrical gas purification.**—See XI.

See also A., Aug., 928. **Ebullioscope for testing purity of liquids.**

### PATENTS.

**Regenerative reverberatory furnace.** O. SCHWITZER, and EISEN- U. STAHLWERK HOESCH A.-G. (B.P. 351,297, 6.8.30).—A form of inlet-outlet passage and removable angle piece between the furnace and regenerators (which are provided for air only) is described.

B. M. VENABLES.

**Furnaces for supplying heating and drying gases.** J. CHATWIN. From SCHMITZ & Co., G.M.B.H. (B.P. 351,625, 14.4.30).—A supply of moderately hot gases is produced by causing cold compressed air to induce a downdraught through the bottom of the furnace; the induced air enters at the top of the furnace but at once passes into an annular space surrounding the fuel and is admitted into and passes through the fuel at a considerable distance below the top.

B. M. VENABLES.

**Rotatable drying drums of the class divided into compartments.** F. HAAS (B.P. 351,130, 8.4.30).—An arrangement of lifting and distributing blades is described. [Stat. ref.]

B. M. VENABLES.

**Rotary dryer.** W. A. HARTY and F. W. MOORE, Assrs. to HARMOR & Co., Inc. (U.S.P. 1,787,197, 30.12.30. Appl., 5.5.28).—A rotary dryer in which the material travels in an annular space between a hollow core and the outer shell has the former extended at the feed end and a burner placed therein. A worm conveyor is either attached to the core or separately driven below it. The worm runs in a casing having an open top to receive the feed.

B. M. VENABLES.

**Drying or concentrating of raw sludge substances.** L. MELLERSH-JACKSON. From F. KRUPP GRUSONWERK A.-G. (B.P. 351,328, 3.9.30).—Sludge, *e.g.*, cement slurry, is lifted into the path of hot gases, *e.g.*, kiln exhaust gases, on the faces of a number of rotating discs in one or more stages, from the earlier of which it falls back into the feed trough and from the last of which it is scraped. Filtration may be effected before, intermediately, or after thickening by heat, and the filter cake may be formed into blocks and calcined in a shaft furnace.

B. M. VENABLES.

**Heat-exchange apparatus.** C. F. HAMMOND and W. SHACKLETON (B.P. 351,020, 19.3.30).—Apparatus of the type comprising a pile of plates provided with spiral or other channels on both sides is operated with heat exchange through the thickness of the plates only by the provision of separating plates of insulating material which reduce the conductivity of the pile as a whole and improve the countercurrent effect. The ribs touching both sides of an insulating plate are arranged to be opposite each other.

B. M. VENABLES.

**Heat interchanger.** A. E. PEW, JUN., Assr. to SUN OIL Co. (U.S.P. 1,787,086, 30.12.30. Appl., 10.6.26).—A heat exchanger of the bundle-of-tubes type having return flow has one tube plate fixed in the casing and the other free to slide. The casing is constructed of a number of pairs of semi-cylindrical sections.

B. M. VENABLES.

\* The remainder of this set of Abstracts will appear in next week's issue.

**Heat-exchanging apparatus for liquids.** R. A. LISTER & Co., LTD., and P. H. WATTS (B.P. 351,587, 2.4.30).—A method of connecting the edges of corrugated sheets to end-pieces without leaving sharp corners is described.

B. M. VENABLES.

**Ball crushing mills.** S. A. WARD (B.P. 350,964, 18.3.30).—The material is dried by hot air in an annular passage between the fixed outer wall and rotating inner wall of the mill, slides down fixed funnel-shaped members, and is alternately caught and flung out by rotating dish members, falling at the bottom into the path of the rotating balls, and after crushing is drawn up the central passage by the same air current.

B. M. VENABLES.

**Apparatus for separating or sorting crushed material.** S. A. WARD (B.P. 351,129, 8.4.30).—A grinding mill, which may be of the ball-race type, is covered by a plate provided with a central aperture for an updraught conveying all the crushed material, and, nearer the periphery, sets of apertures for supply of hot air, supply of new material, and return of oversize. In a separator above, the updraught is deflected downwards with deposition of coarse material into shoots leading to the apertures, which are preferably closed by doors automatically opening when material has accumulated. The draught is diverted upwards again through an annular space of much larger area than the central uptake and passes to a fan at the top which induces all the draught and is driven from the mill by a shaft running up through the hollow main shaft.

B. M. VENABLES.

**Extracting and sampling dust from dust-laden air or gas flowing in a conduit.** DAVIDSON & Co., LTD., and J. WHITMORE (B.P. 351,584, 2.4.30).—The sampling tube faces the main current of gas and serves also as a Pitot tube to measure the pressure and velocity conditions in both the main and sample stream. A pair of smaller tubes are run along the wall of the sample tube, one inside, the other outside; both have closed ends and small side apertures and are used to measure the static pressure. In use the rate of flow of the sample aspirated through a filter is adjusted so that the two static pressures are equal; the velocity of the sample is then equal to that of the main stream, and the cross-section of the sample tube is a definite fraction of that of the main stream, so that calculation is simple.

B. M. VENABLES.

**Reducing and classifying materials.** H. G. LYKKEN (B.P. 351,069, 22.3.30. U.S., 29.3.29).—The material is fed into the bottom of a vertical casing by a mechanical device which also keeps it gently agitated. It is crushed by impact against the corrugated walls, a vortical motion being produced by a lower rotor with an air supply just above. The air-borne material rises to an upper chamber, where a second impeller produces a vortical motion for purposes of classification, the oversize falling back to the grinding compartment through controlled ports near the wall of the casing.

B. M. VENABLES.

**Pneumatic separators.** E. BARTHELMUSS (B.P. 350,932, 7.3.30. Ger., 7.3.29).—An apparatus is described in which coarse material is separated by simple

deflexion with subsequent separation of finer products in whirling streams of air.

B. M. VENABLES.

**Freezing of colloidal liquids.** B. P. TSITOVITSCH (B.P. 351,132, 8.4.30. Esth., 8.4.29).—Liquids comprising or containing biocolloids are rapidly frozen by applying them to surfaces, which are much colder than the lowest f.p. of any constituent, in the form of a thin layer, in drops or in an atomised condition. The frozen layer is removed by scraping or other means and the material kept at a low temp. until thawing is required; when this is effected the original qualities of the material will be recovered unchanged.

B. M. VENABLES.

**Centrifugal-bowl separator.** C. SCHMITZ (U.S.P. 1,786,921, 30.12.30. Appl., 22.4.29. Ger., 27.4.28).—A detachable cover embodying collecting vessels and supply and delivery pipes is described.

B. M. VENABLES.

**Washing slimy-pulpy, finely-divided, or liquid products.** L. ALTPETER, and GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 351,192, 17.5.30).—The material and the wash liquor are supplied in turn to a centrifuge, the lighter liquid, usually the wash water, first, so that they change places in the bowl. After the lighter liquid has collected inwardly it is removed by an overflow device that does not cause agitation.

B. M. VENABLES.

**Filter-leaf construction.** W. H. BACHELDOR, Assr. to STANDARD OIL Co. (U.S.P. 1,787,100, 30.12.30. Appl., 5.8.29).—The leaf is constructed of coarse wire mesh or other spacing material supported in a frame, which is considerably thicker. Finer wire gauze and filter cloth are clamped outside the frame and metallic strips are placed round the interior of the frame to preserve a clear passage for filtrate all round the circumference.

B. M. VENABLES.

**Prevention of incrustation in boilers.** H. MENZ (B.P. 351,331, 8.9.30).—A device containing rolled strips of fabric coated with a tannin extract and a water-softening salt is inserted in the boiler.

B. M. VENABLES.

**Saturation of superheated steam.** I. G. FARBER, Assr. to A.-G. (B.P. 351,296, 1.8.30. Ger., 1.8.29).—Superheated steam is bubbled into water through a perforated pipe or other device and the bubbles are further broken up by beds of balls, rings, etc. in one or more stages.

B. M. VENABLES.

**De-scaling of tubes.** F. ABLARD, J. JOHNSTON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 350,922, 17.1. and 15.2.30).—Scale is removed from the exterior of tubes by internal high-speed hammering effected by collars loosely mounted on the cranks of a crankshaft rotating at at least 10,000 r.p.m. The cranks have small throw, and either may be rigid for straight tubes or may consist of flexible shafts eccentrically mounted in bearings which are rounded off so as easily to enter the tubes.

B. M. VENABLES.

**[Acoustic] means for indicating changes in gases.** A. MEISSNER, Assr. to GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (U.S.P. 1,789,369, 20.1.31. Appl., 30.6.27. Ger., 10.8.26).—A piezo-electric crystal is arranged in the input circuit of a thermionic oscillator

and is provided with two electrodes, one of which is spaced from a face of the crystal a distance equal to an odd number of half wave-lengths of sound corresponding to the natural period of the crystal in a surrounding standard gas. Any change in the gas is indicated by deflexion of the pointer of an ammeter inserted in the output circuit of the oscillator. J. S. G. THOMAS.

**Colloid [for cleaning etc.].** E. L. LEASMAN (U.S.P. 1,790,272, 27.1.31. Appl., 20.6.27).—Finely-divided clay or bentonite is mixed, in quantity sufficient to form a paste, with a hot solution of  $\text{Na}_3\text{PO}_4$ .

H. ROYAL-DAWSON.

**Constructing and applying lagging and other heat-insulating means.** E. KREWS and H. W. FRANKLIN (B.P. 352,640, 19.5.30).

**Fin tubing for heat-exchange apparatus.** GRISCOM-RUSSELL Co., Assees. of R. C. JONES and R. COLSTON (B.P. 353,251, 23.7.30. U.S., 1.8.29).

**Cleaning apparatus for the discs of centrifugal bowls.** AKTEBOLAGET SEPARATOR (B.P. 352,886, 12.12.30. Swed., 20.12.29).

**Apparatus for the production of foam [for fire extinguishing etc.].** PYRENE Co., LTD. (B.P. 352,849, 20.10.30. Ger., 18.10.29).

**Thermostats [of diaphragm type] for refrigerating and like apparatus.** METERS, LTD., and C. C. APPLETON (B.P. 353, 225, 5.7.30).

**Lubricant.**—See II. **Separating gaseous mixtures.**—See VII. **Kilns.**—See IX. **Feed-water.**—See XXIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Ash, ultimate composition, and calorific value of coal.** F. SCHUSTER (Gas- u. Wasserfach, 1931, 74, 629—635).—The methods that have been proposed for determining the true content of mineral constituents of a coal, as distinguished from the ash, fall into three classes: (a) those taking into account the reactions occurring in the ash during combustion of the coal, e.g., oxidation of pyrites, loss of  $\text{H}_2\text{O}$  of hydration of the silicates, etc., (b) Brinsmaid's method in which the calorific val. is determined as a function of the ash content after separating the coal into fractions of different ash content (J. Ind. Eng. Chem., 1909, 1, 65), and (c) methods depending on the extraction of all or part of the mineral constituents with acid (cf. Mayer, B., 1929, 966). The literature of the subject is critically summarised. It is shown that if the ash is regarded as identical with the mineral constituents the results of the ultimate analysis when referred to pure coal may be in error by amounts greater than the limits of error of the analysis. Brinsmaid's method is considered to be the best for determining the calorific val. of the pure coal.

A. B. MANNING.

**Analysis of coals.** BRUÈRE (Ann. Falsif., 1931, 24, 268—273).—Detailed methods are given for the determination of moisture, ash, and coke yield, and calculation of the results, from which calorific power can be obtained by the aid of formulæ and tables given. The character of the coke is also noted and typical figures are given for various coals. E. B. HUGHES.

**Caking power and swelling of coal. II—V.** II. A. J. PIETERS (Rec. trav. chim., 1931, 50, 851—855, 856—859, 860—861, 865—873; cf. B., 1930, 308).—II. The crushing strength of a coke is diminished if the coal is plastic during gas evolution, which thus forms bubbles. The plasticity is affected by the rate of heating of the coal, an optimum rate existing for each kind of coal, and can, if large, be diminished by admixture with a non-caking coal. The crushing strength of the coke may thus be varied from 0 to 30—80 kg./sq. cm.

III. An apparatus for measuring accurately the swelling of coal at accurately known temp. is described. The swelling is shown to be influenced by the rate of heating, to be greater and to occur at a lower temp. if the coal is finely powdered, to occur at a lower temp. but to be less under an applied external pressure, and to be suppressed by addition of sand.

IV. An apparatus for measuring the permeability of coal to gases during heating is described. Passage of a gas through coal prevents the formation of a coherent coke, carrying away tarry matter, which acts as a "primary binder." Experiments with an intermittent gas stream show that the period of suppressed permeability is short. Addition of high-temp. coal tar, or oxidised coal tar, as a binder or source of binder, to a non-caking coal (19.6% volatile matter) caused formation of a coherent coke, the best results (judged by a shatter test) being obtained with 10% of ordinary tar, though 2.5% was sufficient. Oxidised tar was more effective than high-temp. tar. The function of the binder is to coat the coal particles when fluid, and by rapid decomp. to cement them together.

V. An apparatus for determination of the rate of evolution of gas from coal heated at a const. rate is described, and the following conclusions are reached. (1) Water is evolved between 80° and 120°. (2) Evolution of gas commences at 320°, and with good caking coals reaches a large max. at 480°. (3) With non-caking coals evolution of gas is more gradual, and the max. less pronounced and at a slightly higher temp. (4) With both kinds of coal a small secondary max. occurs at about 700°, at which temp. shrinkage of the semi-coke occurs. With banded coal, separated by crushing and sieving into (A) vitrain, (B) durain, and (C) fusain fractions, it was found that A and C showed the gas evolution characteristic of caking and non-caking coals, respectively, B being intermediate. Permeability experiments showed that A and B form a plastic mass at 420—460°, which soon becomes porous, while C is unchanged. A and B begin to soften at 360°; A swells at 420°, and becomes very soft; B swells (somewhat less) at 420°, but remains viscous. C neither swells nor softens.

R. S. CAHN.

**Influence of the constituents of coal on its coking properties.** J. M. PERTIERRA (Anal. Fís. Quím., 1931, 29, 374—385).—The pressures exerted during the coking of the vitrain and durain constituents of a coal were 590 and 325 g./sq. cm. Analyses are given of the isolated constituents and of the coke and other products. The durain had m.p. 380° and the vitrain 403°. The plasticity curves rise rapidly at 410° and 415° respectively, and thereafter fall at different rates which indicate that semi-coke is formed more rapidly from durain than

from vitrain; the former yields a denser and less porous coke. Primary solidification is complete at 450° and 480° respectively. The separation of coal into constituents suitable for coking is discussed.

H. F. GILLBE.

**Apparatus for fuel chemistry.** O. LAUE (Brennstoff-Chem., 1931, 12, 253—255).—Apparatus shown at an exhibition at Essen is briefly described.

A. B. MANNING.

**High-low-temperature carbonisation.** H. T. WRIGHT (Gas J., 1931, 195, 32—34).—The Bussey retort (heated by a portion of the charge, of oval section; the coal rests on a grate and air for combustion is supplied around the periphery some distance above) when tested showed great flexibility. The coke, a good smokeless fuel, passing down the retort is cooled by a water-jacket, quenching being eliminated. The tar is separated into two fractions: (a) a light paraffinic spirit containing 52% of aromatic compounds, (b) a fuel oil; the liquor is disposed of. The coke is made at 1000°, and the outlet gas temp. is 50°, hence it is neither a high- nor low-temp. process.

A. H. EDWARDS.

**Evolution of gases from coal and coke.** G. E. FOXWELL (Gas J., 1931, 195, 91—93).—Measurement of the gas evolution from a S. Yorks coal heated rapidly to above 700° and maintained at a given temp. indicates that the decomp. above 700° is a unimol. reaction and, by analogy with other gas-solid reactions, is probably largely influenced by surface conditions. Examination of the gas evolved from coal at various temps. and at various times after charging into horizontal retorts shows that the thermal yield decreases progressively as the carbonisation time increases, the final amounts of gas requiring disproportionately large amounts of heat. It is suggested that reduction of carbonising time will increase the capacity of the plant, reduce capital and fuel costs by utilising fewer retorts, and will increase the combustibility of the coke when 3—4% of volatile matter is left in the charge.

H. E. BLAYDEN.

**Dilution of straight coal gas.** W. M. CARR (Gas J., 1931, 195, 103—105).—At Stretford gas works coal gas is diluted by producer gas which is drawn from the producers feeding the setting and cleaned. The dilution system and the automatic method of control are described. The quantity of producer gas utilised does not exceed 15 vol.-% of the straight coal gas and the mixed town gas has an average inert content of less than 12%. The installation requires only a small expenditure and no additional labour, and maintenance costs are negligible.

H. E. BLAYDEN.

**Dilution [of gas] by steaming in vertical retorts.** J. E. BLUNDELL (Gas J., 1931, 195, 157—159).—The effect of steaming is to increase the CO<sub>2</sub> and decrease the calorific val. of the gas. Given the yield of gas per ton at one calorific val., the yield at any other can be calc. The efficiency of steamed vertical retorts is compared with that of retorts in which the gas is mixed with water-gas to obtain the same calorific val. Results of tests show increased yields of products, and the ash of the coke is not increased.

A. H. EDWARDS.

**Dilution of horizontal-retort gas.** J. H. CLEGG (Gas J., 1931, 195, 159—161).—A gas of 565 B.Th.U.

was reduced to 499 B.Th.U. by diluting with flue gas from the settings, the admission being regulated by a butterfly valve. At the inlet to the exhauster it is further reduced to 475 B.Th.U. by admission of blue water-gas. The resulting gas contains CH<sub>4</sub> 21.6, H<sub>2</sub> 39.8, N<sub>2</sub> 19.9, CO<sub>2</sub> 3.9, O<sub>2</sub> 0.6, CO 9.9, C<sub>n</sub>H<sub>m</sub> 4.3%.

A. H. EDWARDS.

**The wet sulphur purifier of the Hamburg gas-works.** H. MÜLLER (Gas- u. Wasserfach, 1931, 74, 653—657).—The gas is washed by aq. K<sub>3</sub>Fe(CN)<sub>6</sub> and K<sub>2</sub>CO<sub>3</sub> or KHCO<sub>3</sub>. The S resulting from oxidation of the H<sub>2</sub>S is separated by filtration and the washing liquid is continuously regenerated by electrolytic oxidation and recirculated through the washer. S of 99% purity and H<sub>2</sub> are obtained as by-products. Details of the plants are given and the efficiency of the process is discussed.

H. E. BLAYDEN.

**[Aromatic and hydroaromatic compounds of lignite tar.]** J. HERZENBERG and E. VON WINTERFELD (Ber., 1931, 64, [B], 1911).—The authors' experiments (B., 1931, 706) were performed with a benzine obtained by a particular process and the results are not necessarily applicable to all lignite-tar benzenes.

H. WREN.

**Coal-tar oils for internal-combustion engines.** J. D. SMITH (Gas J., 1931, 95, 41—42).—A blend of 90% of light creosote oil (washed) and 10% of 90—190° water-white solvent naphtha gave satisfactory results when tested on an omnibus provided with two carburetors. The engine was warmed by running with petrol, and thereafter run on tar oil. The blended oil has a closed flash point of 54°; distillation commences at 148°, and 90% distils at 273°. The exhaust gas contained 0.3% CO, compared with 4.2% on petrol.

A. H. EDWARDS.

**Classes of hydrocarbons and some properties of representative Japanese gasoline fractions.** M. MIZUTA (Proc. World Eng. Congr., 1929, 31, 31—48).—The physical consts. and chemical composition of a number of Japanese gasolines have been determined. The relation between aniline point and aromatic content where the latter substances are present in large quantities has been redetermined and is found not to be strictly linear. It was found advisable to cut fractions at 97°, 120°, and 150° for fractions containing C<sub>6</sub>H<sub>6</sub>, PhMe, and xylene. Unsaturated substances are found in small quantity (<12%), whilst gasolines from the southern portion of the Japanese Archipelago are rich in aromatics, those of Formosa containing up to 50% and little naphthenes. The oils from the more northerly districts are richer in naphthenes and poorer in aromatics. The oils from Formosa show laevorotation whilst those from the north are generally dextrorotatory.

T. A. SMITH.

**Pyrogenic decomposition in tube furnaces.** H. I. WATERMAN and T. W. TE NUYL (Petroleum, 1931, 29, 539—542).—The temp. rise in a refined vaseline oil,  $\eta$  0.8681, circulating slowly through an experimental furnace was found to be quite irregular. No decomp. occurred up to 425° and only a small alteration in viscosity was observed; decomp. was vigorous at 600°. Results of 10 tests at different temps. are recorded.

E. DOCTOR.



**Refining process for making high-grade gasoline.**

F. W. HEATH (Petroleum, 1931, 29, 542—544).—To prevent coloration by exposure to air and light, gasoline is treated with aq. alkaline  $K_3Fe(CN)_6$  and subsequently with  $H_2SO_4$ . Unsaturated hydrocarbons are scarcely affected by this method. E. DOCTOR.

**Solvent extraction of lubricating oils.**

S. W. FERRIS, E. R. BIRKHIMER, and L. M. HENDERSON (Ind. Eng. Chem., 1931, 23, 753—761).—A number of solvents have been used to separate the naphthenic and paraffinic constituents of lubricating oils. The solvents have been compared by plotting the relation vols. undissolved oil/vol. solvent against the viscosity-gravity const.  $a$  of the undissolved oil (cf. Hill and Coats, B., 1928, 592). A high value of  $a$  indicates high naphthene content. The selectivity of a solvent may be expressed as the difference between the  $a$  values of the dissolved and undissolved oils. For these relationships to hold extractions should be carried out at least 25° below the crit. solution temp. Many of the solvents were superior to  $SO_2$ ; the best were  $PhCN$ ,  $PhNO_2$ , and  $PhCHO$ . Generally, cyclic compounds were superior to open-chain compounds, and addition of aliphatic chains to the ring reduced the efficiency. Efforts were also made to correlate the extractive value of the solvents with other physical properties such as dielectric const. and internal pressure. A high internal pressure was found to be associated with a high miscibility temp. T. A. SMITH.

**Alinement chart for estimation of viscosity index of oils.** L. NEWELL (Ind. Eng. Chem., 1931, 23, 843).—The viscosity index as defined by Dean and Davis (B., 1929, 1039) may be determined by the aid of the given alinement chart. Viscosities at three temps. and the viscosity index are given. The value of the index for an oil is determined by alining the viscosities as obtained for two temps. and observing where this line cuts the viscosity index scale; this index also indicates the relative paraffinic-naphthenic character of a lubricant as described by Davis and McAlister (B., 1931, 376). T. A. SMITH.

**Regeneration of used mineral oils.**

E. AZZARELLO (Proc. World Eng. Congr., 1929, 31, 1—14).—A scheme is outlined and costs are discussed for the purification of used railway axle oils. It is estimated that 20% of the oil used for this purpose can be recovered, together with the material used as wicks in lubricators and some bearing metal which may be separated by mechanical means from the wicks. A lay-out for the recovery of 10,000 kg. per month is described. After pressing from the fibrous material, the oil is passed through screens, then allowed to settle for 10 days at 85—90°, and treated with limed kieselguhr at the same temp. for a further 20 days and again decanted. The clear liquid is filtered through more kieselguhr and cotton shreds, then through fine coal. After centrifuging, the oil is again filtered through cotton under pressure. The recovered oil is suitable for reuse. T. A. SMITH.

**Acetyl value of oxidised mineral oils.**

T. YAMADA (J. Soc. Chem. Ind. Japan, 1931, 34, 188—189 B).—"Technical white oil" and a switch oil were oxidised in air at 130°; the acid val., sap. val.,

and sap. val. of the acetylated product increase almost linearly with the time, but the acetyl val. of the unsaponifiable fraction shows a definite max. Alcohol and acid formation is small in the case of the less refined switch oil. E. LEWKOWITSCH.

**Determination of soluble bitumen and total organic matter in bituminous rocks.** I. UBALDINI (Annali Chim. Appl., 1931, 21, 222—228).—After the rock has been moistened with EtOH and treated with dil. HCl, the whole is shaken with about 50 c.c. of  $C_6H_6$  and centrifuged. The aq. layer, separated from the clear  $C_6H_6$  solution, is washed with several quantities of  $C_6H_6$  until this is no longer coloured. The total  $C_6H_6$  solution is evaporated and the residue of almost pure bitumen dried and weighed. Org. matter other than bitumen is determined by filtering the aq. layer through calcined, washed asbestos on a Gooch crucible, evaporating the filtrate to dryness, and either subjecting the dried residue to elementary org. analysis or determining its loss of wt. on ignition. T. H. POPE.

**Heating asphalt by diphenyl vapour.** G. H. MONTILLON, K. L. ROHRBACH, and W. L. BADGER (Ind. Eng. Chem., 1931, 23, 763—769).—The heat transfer coeffs. for the heating of asphalt by means of diphenyl vapour have been determined by means of a circulatory apparatus. The liquid-film coeff. increased with the velocity through the heating tube, whilst viscosity had no effect. The average heat-transfer coeffs. were: overall 30—45, asphalt liquid film 39—55, and diphenyl vapour 225—400 B.Th.U. per hr. per sq. ft. per °F. A table of the physical characteristics of diphenyl vapour is given. The only changes in the asphalt due to continued heating under the conditions were such as could be attributed to loss of volatile constituents. T. A. SMITH.

**Petrol-alcohol-benzol mixtures.** C. BARON, C. BOULANGER, and R. LEGRAIN (Compt. rend., 1931, 192, 1383—1385, and Bull. Assoc. Chim. Sucr., 1931, 48, 295—296).—Using aviation petrol,  $d$  0.698, a petrol-benzol mixture containing not more than 15% benzol is stable at -40°, but one containing 25% or more deposits solid at -5°. EtOH (99%) and petrol deposit at -16° with 10% EtOH, at -30° with 40%. Mixtures of petrol and benzol in the ratios 8:1, 7:2, and 6:3 with 1 pt. of EtOH deposit at -27°, -30°, and -43° respectively. C. A. SILBERRAD.

**Spectroscopic studies of engine combustion.**

L. WITHROW and G. W. RASSWEILER (Ind. Eng. Chem., 1931, 23, 769—776).—The normal flame front, the rapid combustion during detonation, and the afterglow or re-illumination of the gases in an engine cylinder have been examined spectroscopically by means of an engine cylinder fitted with an observation window and stroboscopic disc. The flame-front spectrum shows that light is emitted from CH and  $C_2$  mols., whilst the afterglow is due to the combustion of CO. In the detonating flame front the lines due to CH and  $C_2$  are missing, but are re-established when knocking is suppressed by  $PbEt_4$ . Spectroscopic analysis indicates that the difference between knocking and non-knocking explosions is confined to the nature of the combustion of the last portion of the charge. At Pb and  $PbO$  are shown to exist in the flame front at the time when

knocking would occur if  $\text{PbEt}_4$  had not been added. The spectra of a number of fuels other than gasoline are given.

T. A. SMITH.

**Photographic determination of the detonating tendency of petrol.** M. AUBERT and R. DUCHÊNE (Compt. rend., 1931, 192, 1633—1635).—More detailed study of previous methods (cf. A., 1928, 248) confirms the diminution of knock caused by increased turbulence of the air-gas mixture. Actual detonation is indicated by a brilliant spark succeeding diminished light emission, indicating a diminished initial rate of normal combustion. For any given petrol gas-air mixture detonation begins when the mixture is heated to a definite temp.; the higher this is the less is the tendency of the petrol to knock. Knocking is a max. when the initial (normal) combustion is a min. The effect of  $\text{PbEt}_4$  is confirmed (cf. A., 1928, 715; B., 1928, 5). C. A. SILBERRAD.

**Decomposition and polymerisation of the olefines.** G. EGLOFF, R. E. SCHAAD, and C. D. LOWRY, JUN. (J. Physical Chem., 1931, 35, 1825—1903).—A survey of published work relating to the action of heat, light, high pressure, chemical reagents, electric discharges, and  $\alpha$ -particles on the olefines.

H. F. GILLBE.

**Description of plant for preparing coal for pulverised-fuel-fired and stoker-fired boilers at Synthetic Ammonia and Nitrates, Ltd.** G. H. C. CORNER (Fuel Econ., 1931, 6, 337—343).

**Ethyl alcohol. Hexoic acid. Humic acid derivatives.**—See III. Dielectric loss in oils.—See XI. Oil acidity.—See XII. Brown coal and crops.—See XVI.

See also A., Aug., 903, Adsorption of  $\text{PhOH}$  by charcoal. 904, Adsorption of  $\text{H}_2$  and  $\text{CO}$  on  $\text{ZnO-Cr}_2\text{O}_3$  catalysts. 913, System  $\text{NH}_3\text{-H}_2\text{SO}_4$ . 926, Determination of  $\text{CO}$ . 932, Catalytic oxidation of mineral oils in vapour phase. 972, Colour reactions of phenols.

#### PATENTS.

**Coal-washing apparatus.** W. C. MENZIES (U.S.P. 1,790,107, 27.1.31. Appl., 26.2.27).—Special means of controlling the delivery of coal, discharging refuse from the coal, and separating coal from refuse are detailed. To the washing box constructed on the upward flow of water principle is fitted a non-clogging screen.

A. H. EDWARDS.

**Production of coke.** K. WIRGES, and G. WIPPERMANN MASCHINENFABR., STAHLWERK U. EISENGIESSEREI G.M.B.H. (B.P. 351,854, 8.10.30).—Coking coals of high volatile matter content are mixed with ground coke slack consisting of sharp grains of 0—3 mm. diam. and the mixture is carbonised. The coke slack is preferably crushed between rollers until 80—90% is below 1 mm. and none is above 2 mm. in diam.

A. B. MANNING.

**Coking retort oven.** J. VAN ACKEREN, ASSR. to KOPPERS Co. (U.S.P. 1,787,963, 6.1.31. Appl., 23.5.25).—In ovens of the Koppers and Becker type a pair of smaller horizontal flues in each heating wall is substituted for the usual single horizontal flue, and each horizontal flue is subdivided into a number of flues which serve (say) five vertical flame flues. This modification permits an upward extension of the flame flues which decreases the

time of coking, strengthens the construction, and prevents decomp. of the gases.

A. H. EDWARDS.

**Removal of smoke from coke ovens.** J. VAN ACKEREN, ASSR. to KOPPERS Co. (U.S.P. 1,785,748, 23.12.30. Appl., 27.6.25).—A bridge member with a gas passageway on its under side is provided across the bottom of each feed opening in the oven. These permit the gas and smoke to pass to the offtakes across the streams of coal entering through the feed openings during the charging operation.

A. B. MANNING.

**Treatment of coal and like substances.** W. HECKEL, ASSR. to C. H. ELLSWORTH (U.S.P. 1,789,549, 20.1.31. Appl., 30.1.21).—By heating coal or bituminous substances at about  $500^\circ$ , a tar containing lubricating oils free from  $\text{C}_{10}\text{H}_8$ , a gas rich in aliphatic hydrocarbons, and a low-temp. fuel are obtained. The products are condensed successively at  $130^\circ$ ,  $65^\circ$ , and  $35^\circ$ .

A. H. EDWARDS.

**Apparatus for carbonising coal.** J. MITCHELL, ASSR. to CHARCOLITE CORP. (U.S.P. 1,784,676, 9.12.30. Appl., 7.8.25. Cf. B.P. 256,038; B., 1926, 812).—The apparatus comprises a number of vertical, cylindrical, double-walled retorts, the annular spaces between the walls forming heating baths of fusible salts. The retorts are supported in a structure having vertical division walls forming separate heating chambers for each retort. Hot combustion gases from suitably placed burners are circulated through the chambers. Closure means are provided at the upper and lower ends of the retorts, and the upper ends are provided also with gas offtakes. The lower ends of the retorts are cooled sufficiently to prevent by solidification any leakage of the fused salts in the jackets.

A. B. MANNING.

**Apparatus for carbonising and distilling carbonaceous materials.** W. W. ODELL, ASSR. to NORTHERN LIGNITE COAL Co. (U.S.P. 1,785,645, 16.12.30. Appl., 17.10.24. Can., 30.5.24).—The retort consists of a vertical shaft, rectangular in plan, formed of a series of superposed chambers each converging downwardly. Horizontal tuyères are arranged in pairs on opposite walls of the shaft, at the junctions of the superposed chambers. Each tuyère supports a baffle which extends into the shaft and co-operates with centrally arranged baffles in constricting the diameter thereof. A horizontal gas offtake pipe having openings in its under side is provided in the shaft above the level of the tuyères. The fuel is carbonised as it passes through the hot zone produced by combustion of part of the charge with the air blown in at the tuyères. The coke is discharged after traversing a cooling zone, by means of a rotating drum with radial blades.

A. B. MANNING.

**Apparatus for distilling solid carbonisable materials.** F. T. SNYDER (U.S.P. 1,785,565, 16.12.30. Appl., 8.6.25. Can., 14.5.25).—The material is carbonised in a vertical distillation chamber in which it passes successively through an upper evaporating zone, an intermediate carbonising zone, and a lower cooling zone. The products of distillation pass through a heat exchanger and a water-cooled condenser in succession. Part of the gas leaving the condenser is reheated in the heat exchanger and is then subdivided into three currents, two of which pass through the evaporating

and cooling zones respectively of the distillation chamber, whilst the third passes through a heating stove to the carbonising zone of the chamber. Provision is made also for injecting oil, or tar, and air into the bottom of the carbonising zone.

A. B. MANNING.

**Apparatus for gasifying coal etc.** C. DAVIES (U.S.P. 1,784,985, 16.12.30. Appl., 31.5.22).—The apparatus comprises a carboniser, a water-gas generator, and a fuel preheater etc. The carboniser is preferably of the rotary type having baffles on the interior wall which continuously lift and drop the fuel through the hot gas stream. The carbonised material is transferred directly to the water-gas generator, and the hot water-gas from the latter is led to the carboniser, its sensible heat being utilised for carrying out the carbonisation. The blow gases, after passing through the regenerators, serve to preheat the fuel.

A. B. MANNING.

**Production of pure carbon.** C. B. EDWARDS and H. R. HORNER, Assrs. to P. C. REILLY (U.S.P. 1,789,380, 20.1.31. Appl., 2.8.26).—Coke from coal-tar pitch or petroleum coke is fed into an elliptical retort, wherein it is heated by the gases from a combustion zone (temp. 1930°) half way down the retort where the volatile hydrocarbons are removed. The coke passes into a cooler zone and is removed by conveyor screws from the bottom of the retort. The product (0.05—0.07 ohm resistance) is suitable for electrode carbons.

A. H. EDWARDS.

**Gas producer.** J. F. ROGERS, Assr. to WELLMAN ENG. CO. (U.S.P. 1,784,882, 16.12.30. Appl., 2.11.30. Cf. U.S.P. 1,572,040; B., 1926, 351).—A gas producer has a body and an ash pan which normally rotate together but can be automatically given a relative motion at suitable intervals. Improved means of supporting the two parts of the apparatus and of producing the desired motion are described.

A. B. MANNING.

**Carburetted water-gas apparatus.** W. J. MCCLURG (U.S.P. 1,788,400; 13.1.31. Appl., 25.10.24).—The generating chamber is provided with a hollow shaft and both are divided into four symmetrical compartments. Those in the chamber open into a common chamber which communicates with the carburetting shaft; this connects with a column fitted with baffles, the mixed gas and vaporised oil passing to a steam generator and then to storage. The steam and compressed air are conducted through valves to the bottom of each angular compartment and to the common chamber, permitting up- and down-blasting operations.

A. H. EDWARDS.

**Oil-gas making.** F. J. NOLAN, Assr. to THERMO INDUSTRIES, INC. (U.S.P. 1,790,166, 27.1.31. Appl., 28.4.27).—Gas is produced from heavy petroleum oil without residue by feeding measured quantities of heated oil and water into a short hot tube through a central tube open at its end and fitted with baffling plates to mix the vapours. A generator containing several such tubes is heated by oil, the gas being compressed either as liquid or gas into metal containers.

A. H. EDWARDS.

**Gas-generating apparatus.** W. DARBY, and CARBIC, LTD. (B.P. 351,179, 8.5.30).—An apparatus, e.g.,

for the generation of  $C_2H_2$  from  $CaC_2$  in cake form, is provided with baffles to prevent the water from rapidly flowing away from the carbide when the apparatus is disturbed.

B. M. VENABLES.

**Storage and transport of acetylene, more particularly for use for the autogenous working of metals.** A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 351,634, 22.4.30).—The gas is stored in the form of compacted  $C_2H_2$  snow, produced, e.g., by compressing the snow, formed from the gas by known means, in a wooden cylinder by means of a wooden ram.

A. B. MANNING.

**Gas purification.** G. E. SEIL, Assr. to KOPPERS CO. (U.S.P. 1,786,509, 30.12.30. Appl., 11.6.26).—A carbhydrate, e.g., starch, is oxidised, preferably with  $HNO_3$ , and the product is mixed with an Fe compound. A wash liquor for removing  $H_2S$  from coal gas etc. is prepared by dissolving the mixture in water and adding alkali, the liquor preferably containing 0.06—0.1%  $Fe_2O_3$ , 0.1—2.0% of org. material, and about 1% of alkali.

A. B. MANNING.

**Removal of naphthalene and carbon disulphide from gases.** G. E. SEIL, Assr. to KOPPERS CO. (U.S.P. 1,785,365, 16.12.30. Appl., 6.5.26).—The gas is washed with a solvent, e.g., kerosene oil, which will absorb both  $C_{10}H_8$  and  $CS_2$ , preferably by the Sperr process (U.S.P. 1,578,687; B., 1926, 430), and recirculated solvent is treated, e.g., with an aq. or alcoholic solution, to remove  $CS_2$ .

A. B. MANNING.

**Fractional extraction of mineral oils.** S. W. FERRIS, Assr. to ATLANTIC REFINING CO. (U.S.P. 1,788,569, 13.1.31. Appl., 16.7.27).— $PhNO_2$  is dissolved by heating in a viscous oil, liquid at room temp. and containing paraffinic and naphthenic hydrocarbons. On cooling the mixture forms two layers, the upper richer in paraffinic and the lower richer in naphthenic hydrocarbons. The layers are separated, the solvent is removed by distillation, and the process repeated if desired until oils entirely paraffinic and entirely naphthenic in nature are obtained.

H. S. GARLICK.

**Treating hydrocarbons.** J. C. BLACK (U.S.P. 1,789,510, 20.1.31. Appl., 1.8.28).—Hydrocarbon oil is passed in heat-exchange relationship with cold treated oil before passing through pipe coils externally cooled by liquid  $NH_3$  to approx. 8°. The cooled oil is continuously passed through an absorber in countercurrent flow to  $SO_2$  gas liberated from already treated oil. The oil, containing condensed and dissolved  $SO_2$ , is further cooled and then passed through a treater in counterflow to streams of liquid  $SO_2$ , cooled to approx. the same temp., and in sufficient quantity to dissolve from the oil the products to be removed. The treated oil and liquid  $SO_2$  containing extracted material are separated and continuously subjected in separate chambers to pressure distillation at temps. sufficient to vaporise the major portion of  $SO_2$ , which is collected and condensed under the same pressure. The remaining  $SO_2$  is vaporised, condensed, and absorbed in a hydrocarbon oil to be subsequently treated with liquid  $SO_2$ , by a second distillation in separate containers at pressures <10 lb.

H. S. GARLICK.

**Polymerisation of hydrocarbon distillates.** R. C. OSTERSTROM, JUN., J. HYMAN, and C. R. WAGNER, ASSTS. to PURE OIL Co. (U.S.P. 1,789,413, 20.1.31. Appl., 25.1.30).—Gum-forming compounds are removed from cracked hydrocarbon distillate by passing the oil under pressure, in a continuous stream of restricted cross-section, through a vaporising heater and simultaneously thoroughly mixing it with a polymerising agent consisting of Na sulphonates in a polymerised hydrocarbon, the latter being obtained in a preceding operation of the process and returned after treatment with acid and alkali. The light oils are separated from the high-boiling polymerised compounds and separately condensed and collected. H. S. GARLICK.

**Cracking of hydrocarbons.** E. C. HERTHEL and H. L. PELZER, ASSTS. to SINCLAIR REFINING Co. (U.S.P. 1,787,981, 6.1.31. Appl., 11.6.27).—Oil is circulated to and from a supply held in a vaporising zone through heating tubes where it is heated to a cracking temp. A bed of finely-divided solid material, *e.g.*, fuller's earth, is maintained within the main body of oil so that the circulating oil passes through it. Vapours are removed from the vaporising zone under superatm. pressure, subjected to refluxing action, and the reflux condensate together with raw oil is circulated through the heating tubes into the vaporising zone. Heating gases for the cracking operation are passed first over separate heating tubes through which reflux condensate and new oil are circulated, and then over the first mentioned heating tubes. Pitch-laden residual oil is discharged direct from the bottom of the vaporising zone. H. S. GARLICK.

**Method for transforming and distilling hydrocarbons.** A. A. F. M. SEIGLE (U.S.P. 1,779,828—9, 28.10.30. Appl., 14.1.26 and 9.8.27).—Preheated heavy hydrocarbons are passed through a retort consisting of a spiral passage surrounding a heating flue, and the vapours are cracked at 500—700° in presence of metal turnings or other catalysts. The vapours from the retort are then passed through two expansion and cooling chambers where they are cooled to 250—450° in presence of further catalyst, the heat being used to vaporise water under pressure. T. A. SMITH.

**Pressure still for cracking oils.** D. A. YOUNG, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,788,006, 6.1.31. Appl., 15.7.27).—The unvaporised residue-discharge line of a pressure still extends for a substantial distance within the still and below the normal liquid level and is controlled by a valve at its inner end having an operating extension outside the still and passing within the discharge line. H. S. GARLICK.

**Oil-refinery condenser.** J. PRICE (U.S.P. 1,789,880, 20.1.31. Appl., 15.4.29).—A pair of tube sheets are mounted opposite each other on the periphery of a cylindrical shell, so arranged to provide flexible portions to take up changes in length of a bundle of tubes connecting the tube sheets. Baffles are arranged longitudinally at the sides of the tube bundle, and means are provided for preventing the liquid by-passing around the tube bundle, between the baffles and the shell. H. S. GARLICK.

**Treating saturated hydrocarbons and gaseous mixtures rich in saturated hydrocarbons.** Soc.

D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTRI., ASSEES. of E. VOITURON (B.P. 349,067, 14.1.30. Ger., 14.1.29).—Saturated hydrocarbons (*e.g.*,  $\text{CH}_4$ ) in gaseous or atomised form are passed with steam and  $\text{O}_2$  through a reaction chamber maintained at 1300° or above. Sufficient  $\text{O}_2$  is employed to destroy substantially the whole of the hydrocarbon, *i.e.*, 1 mol. of  $\text{C}_n\text{H}_{2n+2}$  to 0.5*n* mol.  $\text{O}_2$ . The vol. rate of flow of the mixture per hour, without taking into account the steam, should be 50,000—100,000 times the vol. of the reaction chamber. Under these conditions the saturated hydrocarbons yield considerable amounts of  $\text{C}_2\text{H}_2$  in addition to CO and  $\text{H}_2$ . R. W. L. CLARKE.

**Conversion of heavy petroleum oils into lubricating oils by treatment with hydrogen at high pressures and temperatures.** STANDARD OIL DEVELOPMENT Co., ASSEES. of E. M. CLARK (B.P. 349,342, 19.6.30. U.S., 18.7.29).—High-grade lubricating oils are obtained in large yield from heavy unrefined petroleum oils which contain small quantities of asphaltic, gummy, or resinous impurities by treatment with  $\text{H}_2$  in presence of catalysts containing W or Mo at temps. above 370° and at pressures above 25 atm. The production of oils boiling below 205° is limited to less than 20% of the oil treated. The process may also be carried out at temps. between 370° and 455° and at pressures above 200 atm. and the yield of oil boiling below 205° limited to 5—10%. Considerable reduction in viscosity and a marked improvement in Conradson C value of the oil accompany the destructive hydrogenation of the impurities. R. W. L. CLARKE.

**Manufacture of lubricating oils and other hydrocarbon products.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,071, 10.2., 18. and 19.8.30).—Solid or semi-solid open-chain hydrocarbons containing at least 13.5 pts. (preferably 15 pts.) of H to 100 of C and having a mean mol. wt. of at least 170 (and preferably 250) and only slight lubricating properties are treated with agents capable of introducing exchangeable substituents, such as Cl, O, and S, in presence of suitable catalysts, preferably  $\text{AlCl}_3$ , and, if desired, in presence of cyclic hydrocarbons or olefines. The exchangeable substituents may be eliminated from the products before treatment with condensing agents, *e.g.*, by steam-distillation in vac. A 50—60% yield of good-quality viscous lubricating oil can be produced from paraffin wax and the like. R. W. L. CLARKE.

**Gear lubricant.** S. D. WHITE and C. L. KNOPP, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,789,614, 20.1.31. Appl., 18.8.28).—Light (Gulf Coast) lubricating oil (100 sec. Saybolt viscosity at 38°) is blended with a heavy Gulf Coast flux oil (1000—5000 sec. at 99°) in proportions that produce a base blend of Saybolt viscosity of 55—62 sec. at 99°. This mixture is heated to a temp. at which it is freely fluid (120—135°) and approx. 25% of caustic bottoms from the redistillation of Gulf Coast lubricating oil over NaOH, preheated to a corresponding temp., are gradually mixed in. The composite stock is then heated to approx. 150° and blown (4—8 hr.) with dry air until the product has a MacMichael viscosity of 55—100 (preferably 85—90) poises at 38°, Knott viscosity not exceeding 12 oz.

at 18°, and a Knott adhesiveness not exceeding 75 oz. at 18°.

H. S. GARLICK.

**Testing bituminous mixtures.** H. W. SKIDMORE and G. ANSON, Assrs. to CHICAGO PAVING LABORATORY, INC. (U.S.P. 1,789,846, 20.1.31. Appl., 5.3.28).—The material to be tested is placed in a stationary die and a movable die aligned with and put adjacent to it. The movable die is actuated so as to subject the cylinder of bitumen (1—4 in. diam.) to an increasing shearing force, an indicator showing the amount and rate of force applied. The dies are mounted in a const.-temp. bath.

A. H. EDWARDS.

**Removal of oxygen from gases.** F. PORTER, Assr. to KAY COUNTY GAS CO. (U.S.P. 1,787,795, 6.1.31. Appl., 19.8.26 and 21.9.27).—Natural gas mixed with air is passed over porous refractory material at 800—2000°. When the product, now containing CO and H<sub>2</sub>, is mixed with a further quantity of natural gas and passed over a suitable catalyst, e.g., Cu or Fe, at 300—500° the CO and H<sub>2</sub> combine with the O<sub>2</sub>.

D. K. MOORE.

**Elimination of combustible constituents from the products of combustion of an internal-combustion engine.** J. C. W. FRAZER (U.S.P. 1,789,812, 20.1.31. Appl., 12.8.29).—An Fe chromite catalyst (approx. Fe<sub>2</sub>O<sub>3</sub>.3Cr<sub>2</sub>O<sub>3</sub>) is deposited on screens of heat-conducting metal placed in the passage of a mixture of air and exhaust gases in such a manner that it is kept heated at 450°, either electrically, or by the reaction.

A. H. EDWARDS.

**Removal of carbon deposits [from internal-combustion engines].** W. G. LOVELL and T. A. BOYD, Assrs. to GEN. MOTORS RES. CORP. (U.S.P. 1,787,789, 6.1.31. Appl., 24.8.28).—A mixture of an amine of b.p. >65° (e.g., isoamylamine, benzylamine) and a varnish solvent is injected into a hot engine, and after 1 hr. the engine is started and the loosened C ejected through the exhaust.

A. H. EDWARDS.

**Domestic gas plant for waste vegetable products.** J. K. GOLD (B.P. 353,221, 2.7.30. U.S., 9.9.29).

**Powdered-fuel burners.** A. DOCKING and J. MCCLEARY (B.P. 352,516, 12.2.30).

**Oil-fuel burners.** BABCOCK & WILCOX, LTD., and A. FRANKISH (B.P. 352,816, 20.9.30).

**Burners [for hot-blast stoves].** W. W. TRIGGS. From FREYN ENGINEERING CO. (B.P. 352,812, 19.9.30).

**Filters [for liquid fuel].** INVENTIONS DEVELOPMENTS, LTD., and W. J. TURNER (B.P. 352,465, 8.4.30).

**Sulphuric acid. Acid sludge.**—See VII. Pre-serving hydrocarbons.—See XIV.

### III.—ORGANIC INTERMEDIATES.

**Ethyl alcohol, a product of high-pressure syntheses.** G. T. MORGAN and R. TAYLOR (Proc. Roy. Soc., 1931, A, 131, 533—540; cf. A., 1930, 867).—From a mixture of H<sub>2</sub> and CO (2:1 by vol.) maintained at 200 atm., in presence of 75 c.c. of a catalyst prepared from Co(NO<sub>3</sub>)<sub>2</sub> and Zn(MnO<sub>4</sub>)<sub>2</sub> and heated to 380—410°, 75 c.c. of liquid product were obtained per hr. This contained approx. 9.8% EtOH. Two acetals, ethylidene dimethyl ether and propylidene dimethyl

ether, were identified in the crude liquid product. Appreciable quantities of EtOH were also obtained by the use of other catalysts.

L. L. BIRUMSHAW.

**Hydrogenation of cyclic compounds. I.** G. ROBERTI (Annali Chim. Appl., 1931, 21, 217—221).—Hydrogenation of PhOH yields cyclohexane, the intermediate product being cyclohexanol, not C<sub>6</sub>H<sub>6</sub>. Hydrogenation of NH<sub>2</sub>Ph gives hydrogenated cyclic hydrocarbons, NH<sub>3</sub>, and other products.

T. H. POPE.

**Pale cresylic acid and lysol.** N. GLASS and A. J. JONES (Pharm. J., 1931, 127, 77).—Impurities in the cresol or the use of an excess of soap causes lysol to gelatinise on dilution. Samples of commercial cresols are examined as to impurities (hydrocarbons, pyridine, etc.), solubility in NaOH, b.-p. curve, percentage of *m*-cresol, and properties of the lysol produced. The presence of fractions boiling above 205°, the proportion of *m*-cresol, ratio of *m*-cresol to high-boiling fractions, and proportion of fatty acids to *m*-cresol are important in standardising cresol and lysol.

E. HOPKINS.

**γ-Methyl-αγ-pentadiene.** H. L. FISHER and F. D. CHITTENDEN (Ind. Eng. Chem., 1930, 22, 869—871).—γ-Methyl-αγ-pentadiene produced from MeCOEt and MeCHO by condensation to methyl β-hydroxy-α-methyl-propyl ketone, followed by successive hydrogenation and dehydration of the resulting glycol, did not polymerise satisfactorily to an elastic resilient product. Polymerisation of αγ-diolefines to rubber-like products is, consequently, not dependent merely on the presence of a vinyl group.

D. F. TWISS.

**Syntheses from natural-gas hydrocarbons. I.**  
**Hexoic acid from pentane.** H. B. HASS and J. R. MARSHALL (Ind. Eng. Chem., 1931, 23, 352—353).—The acids are prepared through the nitriles, which are themselves obtained by treating the alkyl chlorides with NaCN. A 70% yield of nitrile with a recovery of 25—28% of unchanged chloride can be obtained on treating amyl chloride with NaCN, and this yield can be increased to 90% if NaI be used as the catalyst. Secondary alkyl chlorides and bromides give poor yields (about 30%) and *tert*-amyl chloride or bromide affords no nitrile. The reactions are carried out in alcoholic solution, 80% EtOH being best. Equimol. quantities of alkyl chloride and NaI are used together with a 10% excess of NaCN. Hydrolysis to hexoic acid is effected by refluxing the nitrile with an equimol. proportion of H<sub>2</sub>SO<sub>4</sub> (d 1.80) and is complete in 30 min.

T. A. SMITH.

**Methylglycol derivatives of humic acids.** W. FUCHS and O. HORN (Brennstoff-Chem., 1931, 12, 251—252. Cf. A., 1929, 1282; 1930, 1408).—Humic and nitrohumic acids prepared from brown coal react with methylglycol in presence of HCl to form derivatives in which 2 or 4 mols. of methylglycol are combined with 1 mol. of humic acid. The derivatives are not carboxylic esters, for, like the original acids, they form salts by interaction with KOAc in alcoholic solution. It is suggested that addition takes place by the opening of a ring containing O.

A. B. MANNING.

**Lactic acid. Hydrolysis of acetylsalicylic acid.**—See XX.

See also A., Aug., 894, Conductivity of pure liquids. 919, Catalysts for synthesis of MeOH. 949, Mono-sulphonic acids of 1-methylnaphthalene. 950, Prep. of chloronitroanilines. 951, 1-Diazo- $\beta$ -naphthol-4-sulphonic acid. 955, Intermediates in synthesis of di- and tri-arylmethane dyes. 971, Detection of org. compounds. 972, Colour reactions of phenols.

## PATENTS.

Catalytic vapour-phase oxidation of aromatic organic compounds. SELDEN Co., Assecs. of A. O. JAEGER (B.P. 348,604, 8.2.30. U.S., 8.2.29).—The oxidation of  $C_6H_6$ , phenols, PhMe, acenaphthene,  $C_{10}H_8$ , etc. is moderated by mixing the vapours with  $H_2$  or with a suitable oxidisable org. compound (other than a homologue or isomeride) before passing with air or  $O_2$  over the catalyst; e.g.,  $C_6H_6$  or PhMe is mixed with MeOH or other aliphatic alcohol, an aldehyde being recovered as by-product. C. HOLLINS.

Concentration of aqueous formic acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,944, 5.7.30).—A mixture of formamide and aq. (e.g., 32–97%) formic acid is treated with gaseous mineral acid (HCl) or conc.  $H_2SO_4$  at 60–80°, filtered from  $NH_4$  salts, and distilled in vac. Preferably the formamide used should be equiv. to the  $H_2O$  present. C. HOLLINS.

Production of primary alcohols [by reduction of esters]. H. T. BÖHME A.-G. (B.P. 346,237, 10.7.30. Ger., 23.9.29).—The reduction of esters with Na and alcohols is effected in  $H_2$  under pressure, e.g., 15–20 atm. C. HOLLINS.

Production of ketones from secondary alcohols. (A, B) RHEINISCHE KAMPFER-FABR. G.M.B.H. and (B) H. SANDKUHL (B.P. 347,931 and Addn. B.P. 347,933, [A, B] 4.6.30. Ger., [A] 14.6.29).—The sec.-alcohol vapour is led with steam over a dehydrogenating catalyst (Ni, Co, Cu, with or without alkali or alkaline-earth oxides or hydroxides) below the b.p. of the alcohol, usually 120–300°. Examples are the prep. of (A) menthone, cyclohexanone, (B) camphor (from isoborneol) and acetophenone. Yields are 97–99%. C. HOLLINS.

Manufacture of condensation products [aryl 2-benzthiazolyl disulphides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 348,682, 12.2.30).—A 2-thiolbenzthiazole is condensed with a  $\cdot SCl$  or  $\cdot SBr$  derivative of  $C_6H_5$  or  $C_{10}H_5$  in an inert solvent, e.g., dry  $C_6H_6$ ; halogen,  $NO_2$ , alkyl, or alkoxy substituents may be present. Examples are the products from *p*-nitrochlorothiobenzene,  $NO_2\cdot C_6H_4\cdot SCl$ , and 2-thiol- (m.p. 137–138°), 6-chloro-4-methoxy-2-thiol- (m.p. 172–173°), and 4:6-dichloro-2-thiol- (m.p. 181–182°)-benzthiazoles, and from *o*-nitrobromothiobenzene and 2-thiolbenzthiazole, m.p. 110°. C. HOLLINS.

Production of [4-chloro-]derivatives of phthalic acid. E. G. BECKETT, P. F. BAUGHAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 348,632, 15.11.29).—Phthalic acid or anhydride is chlorinated in presence of aq.  $Na_2CO_3$ ,  $K_2CO_3$ , alkali bicarbonates or acetates. C. HOLLINS.

Manufacture of double compounds of the acridine series. A. CARPMAEL. From I. G. FARBENIND.

A.-G. (B.P. 348,047, 27.12.29. Addn. to B.P. 328,212; B., 1930, 741).—3:7-Diacetamidoacridine or other hydrolysable diaminoacridine derivative is alkylated in a solvent (nitro- or dichloro-benzene) to the extent of 50%, e.g., with 1 mol. of Me or Et toluene-*p*-sulphonate at 140°, Ac groups (etc.) being subsequently removed.

C. HOLLINS.

Recovery of sulphites.—See VII.

## IV.—DYESTUFFS.

See A., Aug., 908, Colloid chemistry of dyes. 950 and 952, Use of polychlorobenzenes in synthesis of dyes. 951, Hydrogen sulphite compounds of azo dyes. 955, Synthesis of greater di- and tri-arylmethane dyes. 957, Basic properties of hydrazones (dye derivatives).

## PATENTS.

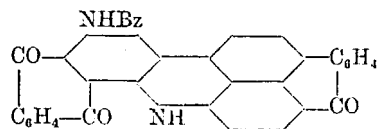
Production and use of anthraquinone derivatives [guanidinoanthraquinones as dyes for acetate silk and wool]. E. G. BECKETT, P. F. BAUGHAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 348,661, 12.11.29).—A di- or poly-aminoanthraquinone hydrohalide is heated with dicyanodiamide, or the mixed bases are treated with hydrogen halide and heated at 150–200°. Products (which may contain unchanged  $NH_2$  groups) from 1:4-diaminoanthraquinone (yellow on acetate silk), 1-amino-4-hydroxyanthraquinone (red on acetate silk, brown-red on wool), 2:3-dichloro-1:4-diaminoanthraquinone, and 1:3:8-triamino-2-methylanthraquinone are described. C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 348,811, 3.4.30).—A 2:3-hydroxynaphthoic arylamide is coupled in substance or on the fibre with a diazotised 6-halogeno-3-amino-1:4:5-trimethylbenzene. Bright red shades fast to boiling NaOH (without pressure) and to light are obtained. Examples are 2:3-hydroxynaphthoic 5-chloro-*o*-anisidide, 4-chloro-2:5-dimethoxyanilide, 2:3- and 2:5-dimethoxyanilides, *o*-toluidide, *o*-anisidide, 4-chloro-2:3:5-trimethylanilide, 6-chlorocresidide,  $\alpha$ - and  $\beta$ -naphthylamides, 4-methoxy- $\alpha$ - and 3-methoxy- $\beta$ -naphthylamides, and *o*-phenetide. C. HOLLINS.

Manufacture of [direct] azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 348,680, 11.2.30).—Diazo-compounds containing mordant groupings are coupled with 1-aminophenylpyrazolones and the products are phosgenated or thiophosgenated; a nitrophenylpyrazolone may be used and the  $NO_2$  group reduced after coupling. Examples are: 3- or 5-aminosalicylic acid, 3-*p*-aminobenzamidosalicylic acid, 4:4'-diamino-3:3'-dicarboxydiphenylcarbamide, 4-nitro-*o*-aminophenol, 1:2-aminonaphthol-4-sulphonic acid, or 2:3-aminonaphthol-6-sulphonic acid  $\rightarrow$  1-*p*-aminophenyl-3-methylpyrazolone, phosgenated. The shades (yellow, orange, red) on cotton are made faster to washing and AcOH by after-coppering. C. HOLLINS.

Manufacture of vat dyes [of the benzanthrone series]. I. G. FARBENIND. A.-G. (B.P. 348,706 and 349,115, 13.2.30. Ger., 13.2.29. [A] Addn. to B.P. 305,679; B., 1930, 755).—(A) 1-Amino-4-(3'-benzanthranyl amino)anthraquinone is aroylated (e.g.,

benzoylated), halogenated (*e.g.*, dibrominated), and finally oxidised by heating in  $\text{PhNO}_2$  to give a yellow-green vat dye (as annexed formula).



(B) A green vat dye of similar structure free from the aroyl-amino-group is obtained by chlorinating or brominating in the 2-position a 1-(3'-benzanthronylamino)anthraquinone (which may already contain halogen in other positions) and condensing the product in  $\text{H}_2\text{SO}_4$  or oleum. [(B) Stat. ref.]

C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 348,671, 18.12.29).—The product described in Example 1 of B.P. 24,604/08 (see B., 1909, 596) is chlorinated or brominated and condensed with a suitable amine, *e.g.*, with 1-aminoanthraquinone for a brown-olive vat dye, becoming olive-green on further condensation in  $\text{H}_2\text{SO}_4$ , or with 1-amino-5-benzamidoanthraquinone (olive-green to olive-brown), aminodibenzanthrone (grey to black), dichloroaminoanthraquinoneacridone (green), or the carbazole from 5:5'-diamino-1:1'-dianthraquinonylamine (brown). The product of alkaline fusion of 1-(9-chloro-3-benzanthronylamino)anthraquinone is chlorinated and condensed with 1-amino-5-benzamidoanthraquinone for an olive-green. C. HOLLINS.

**Dehalogenation of [cyclic] organic halogen compounds [vat dyes of the anthraquinoneacridone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,676, 24.9.29 and 19.6.30).—Halogenated anthraquinoneacridones and other cyclic compounds are partly or completely dehalogenated by treatment with a reducing agent free from metal (*e.g.*, hydrazine or formic acid) in a diluent (*e.g.*, pyridine or quinoline) and in presence of preferably 1 equiv. of Cu or other metal forming reducible oxides or of a reducible salt or oxide of such metal. 2:4:8-Trichloro-6:7-phthaloylacridone is reduced with hydrazine and Cu in boiling pyridine to the 2:4-dichloro-compound (clear red vat dye); the same product results when the hydrazine is replaced by phenylhydrazine, hydrazobenzene, dioxindole, or quinol. In the following 6:7-phthaloylacridones halogen is similarly removed first from position 8 and then from position 1 (the 2-chloro-compound may be reduced with hydrazine under pressure): 1- and 2-chloro-, 1:2- and 1:3-dichloro-, 1:2:3- and 1:2:8-trichloro-, 1:2:3:4-, 1:2:3:8-, and 1:2:4:8-tetrachloro-, 1:2:3:4:8-pentachloro-, 2-chloro-4:8-dibromo-, 2:4-dichloro-1-bromo-, 1:3-dichloro-2:4-dibromo-, 1:2:3-trichloro-4-bromo-, 1:2:3-trichloro-4:8-dibromo-, 2:4:8-tribromo-, 4:8-dichloro-2-methyl-, 4:8-dichloro-2-amino-, 1:2:3-trichloro-8-amino-. The nitro-group is reduced in 1:2:3-trichloro-8-nitro-6:7-phthaloylacridone, but not in the 4:8-dichloro-2-nitro-compound. Other dehalogenations described are: tetra- to di-bromopyranthrone, dichlorotetrabromo- to dibromo-dibenzanthrone, di- to mono-bromobenzanthronepyrazolanthrone, 8-bromo-5:6-phthaloylquinoline to phthaloylquinoline, hexa- to tetra-bromodihydroxydinaphthazine, 1:3-dibromo- to 3-bromo-2-aminoanthraquinone, m.p. 305–310°, 9:10-dichloroanthrac-

ene tetrachloride to 9:10-dichloroanthracene, perchloro- to hexachloro-naphthalene, m.p. 202–204°, 1:3:6-trichloro-2:4-dinitrobenzene to a compound deflagrating at 345°, and octabromocyclohexane, m.p. 178–182°, to gaseous olefines. C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Thermal insulating properties of fabrics.** M. C. MARSH (J. Text. Inst., 1931, 22, τ 245–273).—The chief factor which determines thermal insulating value is the thickness of the fabric, excluding projecting fibres. There is no direct relation between this property and wt. per unit area of the fabric. Comparison of materials of different composition is difficult, and the results indicate that a fabric of a given insulating value could be made from any of the textile materials by choosing the right thickness and closeness of structure. In general, woollen cloths have higher insulating values than worsted, whilst rayon materials with a very shiny surface tend to have high values. B. P. RIDGE.

**Hollow artificial [silk] fibres.** M. HESSENLAND and F. FROMM (Z. angew. Chem., 1931, 44, 630–631).—Instead of extruding the cellulose solution into a coagulating bath in the ordinary way, a coagulating solution of the usual composition is forced through a nozzle into the spinning liquid, whereby hollow threads which are coagulated from inside outwards are formed. These are stretched and passed through a fixing bath. The method is applicable to any cellulose wet-spinning process and the filaments obtained are hollow throughout.

B. P. RIDGE.

**Laundry "winter damage."** J. B. WILKIE (Bur. Stand. J. Res., 1931, 6, 593–602).—"Winter damage" of fabrics dried out of doors is caused by  $\text{H}_2\text{SO}_4$  formed by the oxidation of atm.  $\text{SO}_2$  absorbed by the damp material. The oxidation is accelerated by traces of Fe, spent bleach liquor, and  $\text{AcOH}$ . The damage may be minimised by adding  $\text{Ca}(\text{HCO}_3)_2$  to the final wash water, by eliminating Fe and spent bleach liquor as far as possible from the laundered fabric, and by making the drying time as short as possible. C. W. GIBBY.

**Swelling of cellulose and its affinity relations with aqueous solutions. Preferential absorption of sodium hydroxide from dilute solutions as a characteristic property of cellulose, and an indication of previous mercerisation or other swelling treatment.** S. M. NEALE (J. Text. Inst., 1931, 22, τ 320–338).—The manner in which preferential absorption by various types of cellulose from  $\text{NaOH}$  solutions up to 5*N* varies with  $\text{NaOH}$  concentration is shown. In opposition to Rumbold (A., 1930, 749), it is found that cotton that has been mercerised loose gives a smooth curve of the characteristic "adsorption" type, whilst the curve for purified cotton smoothly approaches an asymptote at about 2*N* concentration, rises rapidly as swelling occurs, and then approaches a higher asymptote at 5*N*. In aq.  $\text{NaOH}$  of any chosen concentration below 2*N*, the absorptions characteristic of various samples of cellulose bear a const. ratio to each other. Investigation of the reactivity of cotton that has been treated with  $\text{NaOH}$  solutions of varying concentration by determining its capacity for absorbing dil. alkali shows that with



NaOH above 15% the product has a fairly uniform level of activity, but immersion of the cotton for more than 3 min. is required to attain the max. effect when the NaOH concentration exceeds 40%. With NaOH solutions more conc. than 25% a less active mercerised product is obtained if the NaOH is washed off with brine than when water only is used, and it is concluded that the former prevents the transient swelling that occurs when cellulose in equilibrium with conc. NaOH is suddenly immersed in water. Mercerisation does not consist essentially in the development of an imperfectly cryst. arrangement due to rapid coagulation of cellulose from a state of semi-solution. B. P. RIDGE.

**Detection of oxycellulose in bleached cotton goods.** H. HEINRICH (Textilber., 1931, 12, 113—114).—The presence and relative amount of oxycellulose are ascertained by boiling 1 g. of the bleached fabric in 20 c.c. of 6% NaOH for 1 hr. (the vol. is maintained const. by additions of water), then diluting the product to 50 c.c., and comparing its yellow colour with that similarly obtained from cotton free from oxycellulose. The results agree with those obtained by the Kauffmann method. The yellower is the extract the higher is the amount of oxycellulose present and the more susceptible the fabric to after-yellowing in storage. A. J. HALL.

**Test for distinguishing oxy- and hydro-celluloses.** R. HALLER (Textilber., 1931, 12, 257).—Cotton fabric freed from impurities is immersed for 1—2 hr. in cold aq.  $\text{SnCl}_2$ , washed, and immersed in a solution of 1 or 2 drops of  $\text{AuCl}_3$  per litre of water; the presence of oxycellulose is indicated by development of a purple colour, whereas hydrocellulose produces no distinctive coloration. The test is effective even when the cotton has been previously boiled with NaOH. A technically bleached fabric usually gives a pink colour in the above test. A. J. HALL.

**Hygroscopic moisture of cellulose.** II. S. OGURI and S. TERUI (J. Soc. Chem. Ind. Japan, 1931, 34, 182—186 B; cf. B., 1930, 898).—The absorption of water vapour by cellulose in an enclosed vessel is independent of temp. (12—30°) if R.H. is kept const. A. A. LEVI.

**Theory of milling. I. Method for measuring the scaliness of wool fibres.** J. B. SPEAKMAN and E. STOTT (J. Text. Inst., 1931, 22, T 339—348).

**Macromols. and micelles.**—See XIII.

See also A., Aug., 905, **Cellulose acetate solutions**. 928, **X-Ray fibre photography**.

#### PATENTS.

**Disinfection of raw wool.** NORDDEUT. WOLLKÄMMEREI & KAMMGARNSPINNEREI (B.P. 352,279, 28.7.30. Ger., 27.8.29).—Wool is degreased with an org. solvent and, after removal of the latter, is treated with known gaseous disinfectants, whereby its sortability is preserved. F. R. ENNOS.

**Opening and preparing artificial staple fibre for spinning.** M. F. THOMA (U.S.P. 1,785,823, 23.12.30. Appl., 8.2.29).—After treatment with softening, crinkling, and hygroscopic agents (cf. B.P. 282,776; B., 1929, 429), the fibres are pressed into flat cakes and stacked in piles 5 ft. or more in height for about 10 hr. The

cakes are then opened up and dried at 50—93°. Static electricity developed during these processes is eliminated by exposing the opened-up fibres to live steam for about 48 hr. Yarn spun from the resulting fibres has a soft silky feel, and shows 25—40% higher strength than yarn made from untreated fibres. D. J. NORMAN.

**Manufacture of crêpe fabric.** BRIT. CELANESE, LTD. (B.P. 352,113, 14.4.30. U.S., 12.4.29).—Highly twisted yarns of degummed silk, which may be sized with a  $\text{H}_2\text{O}$ -sol. material, are incorporated in the fabric, which is afterwards treated with hot aq. liquids. F. R. ENNOS.

**Manufacture of textile materials.** BRIT. CELANESE, LTD. (B.P. 352,058, 31.3.30. U.S., 2.4.29).—Fabrics containing org. derivatives of cellulose, which may be superficially hydrolysed if desired, are treated with an org. substance (other than an animal or vegetable oil), e.g., waxes, fatty acids or their Na salts fixed by a coagulant, which causes scorching at a temp. below that at which the cellulose derivative begins to melt. F. R. ENNOS.

**Manufacture of artificial silk.** W. HARRISON (B.P. 351,527, 25.3.30).—Filaments, spun from solutions of cellulose derivatives (viscose, cellulose acetate or nitrate) in suitable coagulating baths so as to form a skin of cellulose, are treated with a solvent, e.g.,  $\text{Na}_2\text{CO}_3$ , aq.  $\text{CO}_2$ , which causes the core to swell to a greater extent than the outer layers. A diametrical expansion of the threads is thus produced with consequent disposition of the crystallites of the outer layers at right angles to the axes of the threads. F. R. ENNOS.

**Production of artificial filaments etc.** BRIT. CELANESE, LTD. (B.P. 351,718, 6.6.30. U.S., 17.6.29).—The upward dry-spinning of solutions of org. derivatives of cellulose in volatile solvents is initiated by use of a liquid coagulating bath, containing  $\text{H}_2\text{O}$ , MeOH, EtOH,  $\text{C}_6\text{H}_6$ ,  $\text{CCl}_4$ , or kerosene together with soap, which is afterwards withdrawn. F. R. ENNOS.

**Manufacture of matt artificial silk.** GLANZSTOFF-COURTAULDS G.M.B.H. (B.P. 351,395, 21.2.30. Ger., 22.2.29).—Viscose containing finely-divided insol. substances such as metallic compounds, fatty oils, waxes, hydrocarbons etc. is further treated with a  $\text{H}_2\text{O}$ -sol. sulphonated fatty oil (Turkey-red oil) before spinning. F. R. ENNOS.

**Production of artificial [viscose] silk yarn of diminished lustre.** NAAML. VENN. HOLLANDSCHE KUNSTZIJDE IND. (B.P. 348,910, 6.6.30. Holl., 25.10.29).—Viscose emulsions containing fats and oils which yield dull-lustre silk when spun and are so stable that they do not "cream" even when stored up to the point of spontaneous coagulation are obtained by employing as emulsifying agent small quantities of the derivatives obtained by the elimination of  $\text{H}_2\text{O}$  from saturated or unsaturated hydroxy-fatty acids, e.g., ricinoleic acid. A. J. HALL.

**Manufacture of viscose solutions.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 351,401, 17.3.30. Ger., 26.3.29).—Cellulose of a low degree of polymerisation which shows a Cu viscosity (as determined by the described method) of less than 5, after freeing from hemicellulose and moist-grinding, is treated first with part or all of the required

quantity of aq. NaOH of mercerising concentration and then with  $\text{CS}_2$ . The resulting xanthate is dissolved in  $\text{H}_2\text{O}$  together with that portion of the NaOH which was not added at the earlier stage. F. R. ENNOS.

**Manufacture of nitrocellulose.** H. C. HEIDE. From E. TSCHUDIN (B.P. 351,608, 8.4.30).—Prior to nitration, cellulose in a comparatively dense form (cf. B.P. 322,998 and 323,019; B., 1930, 184) is treated with a gas-generating substance, e.g., a bicarbonate, either in solid form or in aq. solution, to facilitate penetration of the nitrating acids. F. R. ENNOS.

**Materials made of or containing organic esters of cellulose.** BRIT. CELANESE, LTD., and S. M. FULTON (B.P. 351,417, 24.3.30. Addn. to B.P. 316,521 and 318,468; B., 1930, 1023).—Saponification of the traveling filaments is carried out during their passage from the spinning machine to the collecting device or during the warping or sizing operations. F. R. ENNOS.

**Esterification of cellulose materials with vapours of lower fatty acids.** KODAK, LTD. From EASTMAN KODAK Co. (B.P. 352,054, 28.3.30).—Cellulose material is heated in the vapour of a fatty acid containing 2–8 C atoms at 100–200° in absence of both  $\text{O}_2$  and catalyst until the product contains at least 4% of the acyl group. It is then further esterified in the usual way, whereby economy in fatty anhydride is effected. F. R. ENNOS.

**Soluble nitrocellulose and coating composition containing it.** E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,788,406, 13.1.31. Appl., 26.1.26).—Cellulose nitrate (10–11.5% N) is digested at 110–140° under at least 50 lb. per sq. in., and washed with water to produce a stability of 30 min. (German test). The resulting ester is dissolved in 95% EtOH, in which it is 99% sol. F. R. ENNOS.

**Phosphoric acid solution of cellulose.** G. W. MILES and C. DREYFUS, Assrs. to CELANESE CORP. of AMER. (U.S.P. 1,787,542, 6.1.31. Appl., 30.12.25).—Cellulose is kept at 0–10° with 75–100%  $\text{H}_3\text{PO}_4$  for several hrs. until dissolved, with simultaneous or subsequent addition of 30–50 vol.-% of EtOH or AcOH. F. R. ENNOS.

**Preparation of aqueous emulsions of cellulose derivatives.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 351,444, 19.12.29).—A cellulose derivative (pyroxylin) is colloided with oil (linseed) and treated with a solvent, and the base material so formed is mixed with an emulsion of Na oleate, gasoline, and  $\text{H}_2\text{O}$ . The resulting water-in-oil emulsion is inverted to an oil-in-water emulsion by vigorous stirring in presence of a large quantity of  $\text{H}_2\text{O}$  determined by the particle size required. F. R. ENNOS.

**Treating cotton and other porous or absorbing vegetable pulps or fibres.** C. W. ANDREWS (U.S.P. 1,784,566, 9.12.30. Appl., 5.10.29).—To prepare a filtering medium for tobacco smoke, raw cotton fibre is steamed at 42 lb./sq. in., boiled with dil. aq. NaOH, washed, dried, combed, and sprayed first with aq.  $\text{H}_3\text{BO}_3$  and then with milk of magnesia. Sweetening agents or EtOH may be introduced. D. J. NORMAN.

**[Electrically] drying cellulose articles such as pulp, cotton cloth, etc.** G. E. COBLENS and A. W. MORRIS (B.P. 351,170, 6.5.30).—The material is interposed between two perforated or wire-mesh electrodes made from, e.g., nichrome or other high-resistance alloy, and the whole is submitted to high pressure to embed the electrodes in the surface of the material. These electrodes are then heated electrically. Drying may be accelerated by passing a.c. or d.c. at, e.g., 100 volts through the material to promote electro-osmosis. D. J. NORMAN.

**Pulpmaking and apparatus therefor.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,784,849, 16.12.30. Appl., 18.9.28).—The digestion liquor is heated to a temp. and pressure slightly above that required for cooking in a separate vessel which can be connected to give a closed system with any digester in the battery. The hot liquor is then introduced at the bottom of the digester and displaces the air therefrom into the heating vessel. The process is particularly suitable for kraft cooking and reduces the time required to reach the cooking temp. from about 2 hr. to 15–20 min. D. J. NORMAN.

**Refining cellulose pulp.** G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN Co. (U.S.P. 1,784,161, 9.12.30. Appl., 27.5.24).—Unbleached pulp, particularly sulphite pulp, is agitated for about 20 min. with a small quantity of an oxidising agent [1–2%  $\text{Ca}(\text{OCl})_2$  on the wt. of pulp], washed, and digested at atm. pressure for 2–6 hr. with, e.g., 0.1–0.5% NaOH solution. After further washing the pulp is bleached, preferably with a mixture of  $\text{Ca}(\text{OCl})_2$  and NaOH or  $\text{Na}_2\text{CO}_3$  to prevent local overbleaching. The resulting product contains 99.6% of cellulose, 93–94% of  $\alpha$ -cellulose, and has Cu no. 1.7. D. J. NORMAN.

**Digester for sulphite wood pulp.** E. MORTERUD (U.S.P. 1,785,591, 16.12.30. Appl., 26.2.29. Nor., 25.1.29).—Intake and outlet pipes from the external heating and circulating system enter the digester adjacently at the middle and communicate with sieve boxes at the top and bottom of the digester. These pipes, which may be made from non-corrosive steel alloy, consist of at least 2 loosely fitting sections, and each section is fixed to the digester wall at one point only to permit free expansion and contraction in any direction. D. J. NORMAN.

**Chemical pulp papermaking process.** F. A. JOHNSON, Assr. to J. H. O'CONNELL, J. D. HASKELL, and DILTS MACHINE WORKS, INC. (U.S.P. 1,786,538, 30.12.30. Appl., 15.10.27).—Highly hydrated stock suitable for the manufacture of grease-proof or glassine paper is obtained by grinding chemical pulp in a pulp grinder under a pressure of 20–80 lb./sq. in. The freeness of the stock is controlled by adjusting the grinding pressure, the  $\text{H}_2\text{O}$  sprays, and the sharpness of the stone. When run on the machine the sheet should be formed quickly, and hence the number of suction boxes should be increased to 9. D. J. NORMAN.

**Filtering and decolorising paper of acid character [for oils etc.].** W. S. BAYLIS, Assr. to FILTROL Co. (U.S.P. 1,784,509, 9.12.30. Appl., 5.8.29).—The paper contains 10–25% of an acid activated clay of the

bentonite or smectite type containing 0.15—0.50% of free  $\text{H}_2\text{SO}_4$ , *e.g.*, "Filtrol" prepared by a process similar to that of U.S.P. 1,397,113 (B., 1922, 5 A).

D. J. NORMAN.

**Manufacture of fibrous articles.** N. E. BROOKES. From BEMIS INDUSTRIES, INC. (B.P. 352,275, 23.7.30).—Pulp is introduced through a moving perforated distributing duct into a form of the desired shape having a foraminous outer wall, which is subjected to a differential pressure to cause deposition of the fibres thereon and removal of the liquid. The article is afterwards partly dried by a current of air introduced through the duct.

F. R. ENNOS.

**Apparatus and process for removing the grease from and cleaning garments and textiles.** R. FABRE (B.P. 352,503, 10.4.30. Fr., 11.4.29).

**Dry-cleaning [plant].** BRIT.-AMER. LAUNDRY MACHINERY CO., LTD. From AMER. LAUNDRY MACHINERY CO. (B.P. 352,666, 29.5.30).

**[Feeding means for] spinning apparatus for artificial silk.** MARTIN HÖLKEN G.M.B.H. (B.P. 352,761, 30.7.30. Ger., 30.7.29).

**Apparatus for washing artificial silk spinning bobbins.** BARMER MASCHINENFABR. A.-G., O. BOCHMANN, and W. ZINSELMAYER (B.P. 353,197, 20.6.30)

**[Flow boxes for] manufacture of paper, pulp-board, etc.** W., H., and H. VOITH [J. M. VOITH] (B.P. 353,133, 13.5.30).

**Cotton fibres etc. Cellulose derivative fibres. Yarns or filaments.**—See VI. Mouldable materials. —See XIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Swelling of cellulose. Oxycellulose. Oxy- and hydro-cellulose.**—See V.

### PATENTS.

**Bleaching textile goods and fibrous substances of all kinds with hydrogen peroxide.** H. E. POTTS. From G. ADOLPH (B.P. 351,217, 5.6.30).—Bleaching is effected by impregnating the textile material with a solution of  $\text{H}_2\text{O}_2$  of suitable concentration and alkalinity (*e.g.*,  $\text{H}_2\text{O}_2$  0.05—0.5,  $\text{NaOH}$  0.5—5,  $\text{H}_2\text{O}$  100), and then maintaining the impregnated material at a suitable temp. and avoiding evaporation until bleaching is complete.

A. J. HALL.

**Dyeing of loaded silk.** SOC. CHEM. IND. IN BASLE (B.P. 351,400, 15.3.30. Switz., 16.3.29).—Loaded silk is dyed in a neutral or fatty soap bath with a metal (Cu or Cr or both) compound derived from an azo dye made from an *o*-hydroxy- or *o*-carboxy-diazo component and an aminonaphtholsulphonic acid (*e.g.*, 2:5:7, 1:8:4, 2:8:6) or an *N*-derivative of such a sulphonic acid, or with a further conversion product of a reduced dye containing Cr obtainable from diazotised 5-nitro-2-aminophenol and a 6-arylamino- $\alpha$ -naphthol-3-sulphonic acid.

A. J. HALL.

**Coloration [with aniline-black] of [cellulose acetate] textile materials.** BRIT. CELANESE, LTD., A. MELLOR, and D. T. McLELLAN (B.P. 351,577, 2.4.30).

—Mixed material containing a cellulose ester or ether, particularly cellulose acetate, and a vegetable fibre such as cotton is dyed black by first treating it with an aq. suspension of *p*-aminodiphenylamine or 2:4-diaminodiphenylamine which is absorbed by the cellulose acetate but not the cotton, then padding with an aniline-black liquor containing  $\text{NH}_3\text{Ph}$ ,  $\text{HCl}$ ,  $\text{KClO}_3$ , and  $\text{CuSO}_4$ , drying, ageing at 40—50° for development of a black on both fibres, and finally after-chroming at room temp. for 1 hr. with aq.  $\text{Na}_2\text{Cr}_2\text{O}_7$  (*d* 1.005—1.015).

A. J. HALL.

**Coloration [by discharge printing] of materials made of or containing cellulose esters or ethers.** BRIT. CELANESE LTD., and G. H. ELLIS (B.P. 351,457, 20.3.30).—Coloured discharges on cellulose acetate etc. materials dyed with azo dyes are obtained by using a discharge paste containing  $\text{SnCl}_2$  or  $\text{Sn}(\text{CNS})_2$ , an anthraquinone dye, and a swelling agent for the silk such as phenol, quinol, or an alcohol. Anthraquinone dyes sensitive to hyposulphites are usually unaffected by  $\text{Sn}^{II}$  salts.

A. J. HALL.

**Treating cotton fibres.** W. PICKARD and J. WEST (B.P. 351,248, 26.6.30).—Cotton of shorter staple than hitherto is spun into very strong yarn of fine counts after a treatment in which it is sprayed with a solution of rubber in naphtha, allowed to dry, and rendered fire-proof by spraying with a solution containing 4 oz. of  $\text{NaHCO}_3$  and 1 oz. of  $\text{H}_2\text{SO}_4$  per gal.

A. J. HALL.

**[Linen-like] yarn produced from cotton or other vegetable fibres or derivatives thereof.** D. HUNTER, and LINUM PRODUCTS SYNDICATE, LTD. (B.P. 348,305, 25.3.30).—A permanent stiffness is conferred on cotton yarn by impregnation with a solution of cellulose (*e.g.*, cotton dissolved in cuprammonium solution), which may or may not be capable of attacking the yarn, followed by treatment with a coagulating agent such as  $\text{NaOH}$  of mercerising concentration.

A. J. HALL.

**Manufacture or treatment of textile or other material [delustred cellulose derivative fibres].** BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 349,980, 5.3.30. Addn. to B.P. 332,231; B., 1930, 944).—The relustring process previously described is effected with steam having a degree of superheat of at least 20°, and at ordinary or (preferably) higher pressure.

A. J. HALL.

**Process and apparatus for treating [tinting and lubricating] yarns or filaments.** BRIT. CELANESE, LTD. (B.P. 350,056, 14.3.30. U.S., 14.3.29).—Yarns of cellulose acetate or other cellulose derivative are tinted and made more suitable for knitting and weaving by impregnation either successively or simultaneously with a fugitive dye, *e.g.*, Acid Green G, and a lubricant, *e.g.*, a mineral oil or olive and castor oils, together with one or more polyhydric alcohols, *e.g.*, ethylene glycol, glycerin, and diethylene glycol.

A. J. HALL.

**Production of coated fabric material resistant to the growth of fungus.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 350,833, 5.9.30).—See U.S.P. 1,779,258; B., 1931, 585.

**Vat for wet-treatment of lengths of fabric.** B. SCHROERS (B.P. 353,355, 14.11.30).

# VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Titration [of sodium hydroxide and carbonate] by Winkler's and by Warder's method.** A. SUCHIER (*Z. angew. Chem.*, 1931, 44, 534—536).—The applicability of the two methods for the determination of carbonate in fairly pure (93—97%) NaOH has been studied. The average deviation from the true val. for  $\text{Na}_2\text{CO}_3$  by the Winkler method is +0.2%, and by the Warder method +0.7%; for the NaOH determination the deviations are +0.1% and -0.2%, respectively. Curves for obtaining the correct from the experimental val. are reproduced. H. F. GILLBE.

**Factors influencing the stability of hypochlorite solutions and a proposed formula for a modified Dakin's solution.** H. DAVIS (*Pharm. J.*, 1931, 127, 81—82).—The rate of decomp. of Eusol B.P.C. and Dakin's solution at room temp. is measurably affected by light, but exposure to the air has little effect. At 55° Dakin's solution is much the more stable. Solutions from Daufresne's, Dakin's, and the U.S.P. X formulae are in descending order of stability, and the first two have  $p_{\text{H}}$  10.23 and 9.71, respectively. A modified formula in which the amounts of  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  and  $\text{H}_3\text{BO}_3$  used depend on the % of available Cl in the Ca hypochlorite gives a uniform product, relatively stable and less alkaline ( $p_{\text{H}}$  9.53) than the earlier solutions. E. HOPKINS.

**Deterioration of sodium hypochlorite solution.** A. KLING and R. SCHMUTZ (*Compt. rend.*, 1931, 192, 1655—1657).—Decomp. according to (a)  $\text{NaOCl} = \text{NaCl} + \text{O}$  is of practical consequence only in samples of chlorometric degree above 20; in ordinary samples of degree below 12 only (b)  $3\text{NaOCl} = \text{NaClO}_3 + \text{NaCl}$  is of importance. To distinguish deterioration due to (b) from fraudulent addition of  $\text{H}_2\text{O}$  the amount of  $\text{Cl}_2$  evolved on addition of HCl is determined. The quantity evolved in cases of genuine deterioration is unchanged:  $\text{NaOCl} + 2\text{HCl} = \text{NaCl} + \text{H}_2\text{O} + \text{Cl}_2$  and  $\text{NaClO}_3 + 6\text{HCl} = \text{NaCl} + 3\text{H}_2\text{O} + 3\text{Cl}_2$ . C. A. SILBERRAD.

**Conversion of calcium cyanamide into cyanide.** P. PASCAL and (MLLE.) BERNHEIM (*Compt. rend.*, 1931, 192, 1425—1427).—The best yield is obtained by heating 1 pt. of Ca cyanamide (pure) with 0.2 C and 4 NaCl at 1000°. C. A. SILBERRAD.

**Method of bringing tricalcium phosphate into solution.** P. JOLIBOIS and G. CHAUDRON (*Compt. rend.*, 1931, 192, 1650—1652).— $\text{Ca}_3(\text{PO}_4)_2$  (e.g., 3 kg. of Morocco phosphate) is suspended in  $\text{H}_2\text{O}$  (50 kg.) slightly acidified with HCl (0.5 kg.), and  $\text{PbCl}_2$  (6 kg.) is added. The  $\text{Ca}_3(\text{PO}_4)_2$  is converted into insol.  $\text{PbCl}_2 \cdot 3\text{Pb}_3(\text{PO}_4)_2$ , which is separated, the Pb remaining in solution being precipitated by  $\text{Ca}(\text{OH})_2$ . The chlorophosphate is then treated with  $\text{HNO}_3$  of  $d$  1.32 (6 kg.), when most of the Pb separates as  $\text{Pb}(\text{NO}_3)_2$ , which is converted by KCl into  $\text{PbCl}_2$  and  $\text{KNO}_3$ . The solution is neutralised with  $\text{NH}_3$  when the rest of the Pb separates as  $\text{Pb}_3(\text{PO}_4)_2$ , and the mixed  $(\text{NH}_4)_3\text{PO}_4$  (1.6 kg.) and  $\text{NH}_4\text{NO}_3$  (0.1 kg.) are obtained on evaporation. C. A. SILBERRAD.

**Dangers in refining radioactive substances.** H. SCHLUNDT, W. MCGAVOCK, JUN., and (Miss) M. BROWN (*J. Ind. Hygiene*, 1931, 13, 117—134).—Satisfactory methods of reducing the intensity of radiations from radioactive materials are described; potential danger exists from  $\gamma$ -rays transmitted by the screens used and from  $\alpha$ -particles produced from emanation in the air. H. F. GILLBE.

**Recovery and utilisation of sulphur dioxide in the extraction of sulphur.** P. LEONE (*Annali Chim. Appl.*, 1931, 21, 238—244).—To utilise the  $\text{SO}_2$  of the gases issuing from the earth in the Sicilian S regions, it is suggested that the gases be passed over NaCl at 450—500° and thus converted into HCl and  $\text{Cl}_2$ . This mixture is washed with  $\text{H}_2\text{O}$  and the resulting aq. HCl used to dissolve the S-containing calcareous gangue and so enrich it in S. The  $\text{Cl}_2$  may be used for making bleaching powder. T. H. POPPE.

**Moisture content of liquid sulphur dioxide.** A. K. SCRIBNER (*Ind. Eng. Chem. [Anal.]*, 1931, 3, 255—257).—When the  $\text{H}_2\text{O}$  content of liquid  $\text{SO}_2$  is less than 65 p.p.m. absorption by  $\text{P}_2\text{O}_5$  is recommended for its determination. Corrections are given for changes in temp. and pressure. E. S. HEDGES.

**Removal of atmospheric carbon dioxide by means of active charcoal.** S. YAGI (*J. Soc. Chem. Ind. Japan*, 1931, 34, 203—205 B).—Experiments designed to investigate the possibility of removing  $\text{CO}_2$  from air by means of active charcoal instead of caustic alkali solution in the prep. of  $\text{N}_2$  by the liquefaction process show that below -90° the  $\text{CO}_2$  is almost completely adsorbed by active charcoal, but that below -100° to -120° the  $\text{O}_2$  adsorption, which is small or negligible at higher temp., suddenly increases to high values. This may be connected with the fact that the crit. temp. of  $\text{O}_2$  is -118°, below which capillary liquefaction is possible. The adsorption capacity of the charcoal at -90° for  $\text{CO}_2$  in air is much less than that for pure  $\text{CO}_2$ , and this is thought to be due to the adsorption of  $\text{O}_2$  and  $\text{N}_2$  in the former case. The effects of pressure, heat of adsorption, and velocity of gas flow are considered. N. H. HARTSHORNE.

**S purifier.**—See II. Na silicate. Boric acid and  $\text{Al}_2\text{O}_3$ .—See VIII. Metals and alloys with  $\text{H}_3\text{PO}_4$ . Rubber for pickling baths.  $\text{H}_2\text{S}$ .—See X.  $\text{ZnSO}_4$ .—See XI. S in black powder.—See XXII.

See also A., Aug., 893, Phosphorescent substances. 902, Cyclic separation of two salts. 904, Adsorption of  $\text{H}_2$  and CO on  $\text{ZnO}-\text{Cr}_2\text{O}_3$  catalysts. 907, Hydrous  $\text{Al}_2\text{O}_3$  sols. 911, Recrystallisation of  $\text{CaHPO}_4$ . 916,  $\text{CO}_2$  absorption. 919, Ni catalysts for hydrogenation. 921, Pure  $\text{KNO}_3$  and  $\text{NaNO}_3$ . Refractory carbides, nitrides, and borides. 923, Standard  $\text{U}_3\text{O}_8$ . 926, Determination of CO, and of small quantities of alkalis in  $\text{H}_2\text{O}$ -insol. acids ( $\text{H}_2\text{WO}_4$ ). 927, Microanalysis of Be silicate rocks. 929, New cell for electroanalysis. 986, N fixation and  $\text{NH}_3$  production by *Azotobacter*. Nitrite formation by soil bacteria.

## PATENTS.

**Sulphuric acid chambers.** FISON, PACKARD, and PRENTICE, LTD., CHANCE & HUNT, LTD., and R. T. MAUDSLEY (B.P. 352,016, 28.3.30).—Two chambers with

vertical or almost vertical walls and of semicircular or semi-elliptical cross-section are built up with the flat surfaces separated only by the necessary supporting framework etc.; the chambers are provided with means for  $\text{H}_2\text{O}$ -cooling over the whole of the exterior surface, with connecting pipes near the top, and with an inlet and outlet at the lower ends of the first and second chambers, respectively.

L. A. COLES.

**Process for utilising waste gases from contact plants for sulphuric acid manufacture.** METALLGES. A.-G. (B.P. 351,825, 5.9.30. Ger., 3.10.29).—The gases are used as combustion air for roasting pyrites, Zn blende, etc. in blast-roasting apparatus.

H. ROYAL-DAWSON.

**Apparatus for concentrating sulphuric acid.** W. C. MAST, ASSR. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,790,507, 27.1.31. Appl., 22.9.27).—The dil. acid, especially that from the acid treatment of oils, flows through a series of compartments in which it is treated with a countercurrent of hot gases led successively into each compartment below the acid level.

L. A. COLES.

**Recovery and purification of dilute sulphuric acid from acid sludge.** F. E. KIMBALL (U.S.P. 1,790,049, 27.1.31. Appl., 30.9.29).—Water is added to the sludge obtained by the action of  $\text{H}_2\text{SO}_4$  on petroleum oils, and the acid layer is separated and treated to  $150^\circ$  under pressure. After keeping, the acid separates from the oil.

D. K. MOORE.

**Process and apparatus for converting sulphur dioxide into trioxide.** C. B. CLARK, ASSR. to GEN. CHEM. CO. (U.S.P. 1,789,460, 20.1.31. Appl., 10.11.26).—Temp. control is maintained by withdrawing part of the gas leaving each converter, cooling it, and then re-introducing it with the gas entering a subsequent converter. If the  $\text{SO}_3$  is absorbed from the withdrawn gas before its reintroduction, the overall conversion of  $\text{SO}_2$  into  $\text{SO}_3$  is increased.

D. K. MOORE.

**Apparatus for manufacturing nitrogen oxide.** F. W. DE JAHN, ASSR. to F. A. BOWER (U.S.P. 1,789,580, 20.1.31. Appl., 15.6.28).—A mixture of air and  $\text{NH}_3$ , obtained by passing air, heated by the gases from the converter, up a column down which aq.  $\text{NH}_3$  flows, is passed through a heat interchanger and a converter provided with cooling coils.

D. K. MOORE.

**Manufacture of boric acid.** A. KELLY (B.P. 351,810, 20.8.30).—Borax in any degree of hydration is added to a hot solution of  $\text{Na}_2\text{SO}_4$  and  $\text{NaHSO}_4$ , and the liquor is boiled and separated from the  $\text{Na}_2\text{SO}_4$ .  $\text{H}_2\text{SO}_4$  is added, and the liquor is cooled to  $30^\circ$ .

H. ROYAL-DAWSON.

**Manufacture of sodium carbonate.** H. B. KIPPER (U.S.P. 1,789,235, 13.1.31. Appl., 22.5.28).— $\text{CO}_2$ ,  $\text{NH}_3$ , and purified brine are simultaneously introduced into a reaction chamber fitted with stirring and cooling devices, and the ppt. of  $\text{NaHCO}_3$  is separated from the aq.  $\text{NH}_4\text{Cl}$ , and converted into  $\text{Na}_2\text{CO}_3$  by heating with the hot gases from a lime kiln.

D. K. MOORE.

**Production of potassium sulphate and soda.** C. T. THORSELL (U.S.P. 1,787,497, 6.1.31. Appl., 25.2.30. Ger., 5.3.29).—The mother-liquor from the

Solvay process is mixed with  $\text{CaSO}_4$  or  $\text{SrSO}_4$ , and after separating the carbonate, the remaining liquor is treated with crude sylvinic  $\text{NaCl}$  and  $\text{NH}_3$ ; after filtering, the mother-liquor is cooled to remove the  $\text{NH}_4\text{Cl}$ , the remaining liquor being used again in the cycle and the residue treated with  $\text{H}_2\text{O}$  for conversion into  $\text{K}_2\text{SO}_4$ .

H. ROYAL-DAWSON.

**Recovery of alkali sulphites from fusion liquors.** C. L. MASTERS, ASSR. to ELKO CHEM. CO. (U.S.P. 1,788,955, 13.1.31. Appl., 4.6.27).—An aromatic sulphonate is fused with alkali hydroxide and the phenolic compound liberated by the addition of  $\text{SO}_2$  and separated. Any phenol remaining in the solution of alkali sulphite is removed by distillation. The sulphite solution is cooled and crystallised. The mother-liquor is conc. and the steam produced used for the distillation.

D. K. MOORE.

**Separation of potassium aluminates and phosphates.** I. G. FARBERIND. A.-G. (B.P. 351,877, 31.10.30. Ger., 23.11.29).—Gaseous  $\text{NH}_3$  is passed at  $15$ – $20^\circ$  through a solution of  $\text{K}_3\text{PO}_4$  and K aluminate (such as is obtained by heating Fe Al phosphates with aq.  $\text{KOH}$ ) to saturation point, whereby the liquid separates into two layers, the bulk of the phosphate being in the lower layer, whilst the upper contains the aluminates. The upper layer is drawn off and the lower layer is diluted with  $\text{H}_2\text{O}$  and further separated by treatment with  $\text{NH}_3$ .

H. ROYAL-DAWSON.

**Manufacture of (A) monophosphates of alkali metals and alkaline earths, (B) ammonium phosphate.** I. HECHENBLEIKNER, ASSR. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,790,502—3, 27.1.31. Appl., 1.7.27).—(A)  $\text{Ca}_3(\text{PO}_4)_2$ ,  $\text{CaO}$ , or a Na salt or (B) aq.  $\text{NH}_3$  is treated with dil.  $\text{H}_3\text{PO}_4$ ,  $d$  1.2–1.26, and the solution obtained is dried by atomisation in a current of hot gas. The residual gases are scrubbed with the dil.  $\text{H}_3\text{PO}_4$  for use in the process to recover (A) dust, (B)  $\text{NH}_3$ .

L. A. COLES.

**Concentration of solutions of magnesium chloride.** S. B. HEATH, ASSR. to DOW CHEM. CO. (U.S.P. 1,789,385, 20.1.31. Appl., 20.3.29).—The solutions are heated indirectly by steam passing through a Ni coil kept in contact with Al bars which are preferably cleaned and sand-blasted between each operation; the product contains  $<0.002\%$  Ni.

L. A. COLES.

**Manufacture of phosphorus oxychloride and thionyl chloride.** J. G. SCHUDEL, ASSR. to ELKO CHEM. CO. (U.S.P. 1,788,959, 13.1.31. Appl., 13.6.28).—When a mixture of  $\text{SO}_2$  and  $\text{Cl}_2$  (preferably as liquids) reacts with  $\text{PCl}_3$ ,  $\text{SOCl}_2$  and  $\text{POCl}_3$  are produced. The mixture may be separated by fractional distillation.

D. K. MOORE.

**Production of hydrogen from steam and carbon monoxide.** J. S. BEEKLEY, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,789,538, 20.1.31. Appl., 12.11.26).—The activity of Cr oxide gel as a catalyst for the production of  $\text{H}_2$  from steam and CO is increased during its prep. and maintained while in use by preventing air from coming into contact with it at temp. above  $120^\circ$ .

D. K. MOORE.

**Process for separating mixed liquefied gases.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 351,599,

4.4.30. Holl., 19.4.29).—C, SiO<sub>2</sub> gel, or other selective adsorbent is used to remove impurities from O<sub>2</sub> or other gas while liquid. The mixed impurities, *e.g.*, Kr and X, are afterwards separated by fractional absorption or distillation, or by chemical means. B. M. VENABLES.

**Separation of mixtures of rare gases.** I. G. FARBERIND. A.-G., Assecs. of K. PETERS and K. WEIL (B.P. 351,898, 7.1.31. Ger., 7.1.30).—Gaseous mixtures adsorbed on suitable agents are expelled fractionally under greatly reduced pressure, and at temps. at which the tension of one of the constituents is almost zero, whilst that of the other is sufficient for its removal. *E.g.*, a mixture of A, Kr, and X may be separated by pumping off the A at  $-129^{\circ}$  to  $-92^{\circ}$ , the Kr at  $-92^{\circ}$  to  $-78^{\circ}$ , and the X at higher temp., all at about 0.1 mm. pressure. H. ROYAL-DAWSON.

**Oxygen from gases.**—See II. **Formic acid.**—See III. **Colloidal Pb.**—See X. **Electrolysis.**—See XI. **Fertilisers.**—See XVI.

## VIII.—GLASS; CERAMICS.

**Advantages of electric heating applied to glass lehrs.** R. M. CHERRY (Chem. Met. Eng., 1931, 38, 400—402).—Electrically heated lehrs for glassware and plate glass are described and their performances given.

D. K. MOORE.

**Polariscopes for use in glass factories.** C. D. SPENCER and S. JONES (J. Amer. Ceram. Soc., 1931, 14, 512—517).—The advantages of using the reflexion type of instrument together with binocular analysers are discussed and the importance of matching the field (tint plate) colours is emphasised. A tint plate with a retardation of 575 m $\mu$  is recommended.

J. A. SUGDEN.

**Viscosity of glass between the strain point and the melting temperature.** H. R. LILLIE (J. Amer. Ceram. Soc., 1931, 14, 502—511).—An apparatus for measuring the elongation of a glass fibre under load is described. Viscosity measurements were made on three simple Na<sub>2</sub>O—CaO—SiO<sub>2</sub> glasses both at low temp. and also in a rotating-cylinder viscosimeter, between the softening point and the m.p. The data from 500° to 1400° lie on a smooth curve. J. A. SUGDEN.

**Index of refraction of some soda-lime-silica glasses as a function of composition.** C. A. FAICK and A. N. FINN (J. Amer. Ceram. Soc., 1931, 14, 518—528).—Measurements were made on a series of pure Na<sub>2</sub>O—SiO<sub>2</sub> and Na<sub>2</sub>O—CaO—SiO<sub>2</sub> glasses melted in Pt from pure batch materials. From the results and the analytical figures it is possible accurately to predict *n* from the composition and also the composition of the various glasses having the same *n*. Evidence is given that *n* may be a simple function of certain Na<sub>2</sub>O—SiO<sub>2</sub> compounds present. J. A. SUGDEN.

**Devitrification of "pyrex" glasses.** G. W. MOREY (J. Amer. Ceram. Soc., 1931, 14, 529—531).—Devitrification was studied by the quenching method in the case of three glasses which have a lower liquidus temp. than any other known mixture having so high a SiO<sub>2</sub> content. The glasses with *n* 1.471, 1.485, and 1.473 had liquidus temps. 1077°, 1042°, and 1036°

respectively. In all cases tridymite is the primary phase and cristobalite was never observed.

J. A. SUGDEN.

**Effects of body composition and firing treatment on salt glazes.** R. K. HURSH and E. C. CLEMENS (J. Amer. Ceram. Soc., 1931, 14, 482—489).—The firing treatment has a greater influence on the glaze than has the body composition. The addition of various salts has no effect except MgCO<sub>3</sub> and CaCO<sub>3</sub> (above 1%), which give a darker and matt glaze. Increase in Al<sub>2</sub>O<sub>3</sub> content gives lighter colour and egg-shell texture and increases the tendency to devitrify. Increase in SiO<sub>2</sub> content improves the gloss and colour and decreases the tendency to devitrify. Addition of Fe<sub>2</sub>O<sub>3</sub> only darkens the colour and increase in flux content diminishes the tendency to devitrify. Reducing conditions during firing increase the thickness of the glaze and darken the colour (except in high-Al<sub>2</sub>O<sub>3</sub> bodies), and the glazes are more fluid as shown by the greater tendency to crystallise. J. A. SUGDEN.

**Separation and determination of boric acid and alumina.** Application to glasses, enamels, etc. MALAPRADE and SCHNOUTKA (Compt. rend., 1931, 192, 1653—1655).—To separate Al from a solution in which H<sub>3</sub>BO<sub>3</sub> is to be determined the solution is saturated in the cold with SO<sub>2</sub>, when the Al is precipitated quantitatively as an indefinite basic sulphite (*cf.* A., 1931, 452), and all the H<sub>3</sub>BO<sub>3</sub> remains in the solution, which is then acidified with HCl and boiled until all SO<sub>2</sub> and CO<sub>2</sub> are driven off. The Al ppt. is dissolved in HCl. Both can then be determined as usual. For glass or enamel the sample is fused with KOH and the filtered solution of the melt treated with SO<sub>2</sub>, when the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> both separate, though the presence of SiO<sub>2</sub> does not interfere with the titration of mannoboric acid with KOH. C. A. SILBERRAD.

**Sodium silicate, a new enamel raw material.** M. E. MANSON (J. Amer. Ceram. Soc., 1931, 14, 490—494).—The addition of SiO<sub>2</sub> to an enamel as an easily fusible silicate (*e.g.*, Na<sub>2</sub>SiO<sub>3</sub>) decreases the smelting temp. and time required to bring about complete dissolution of the SiO<sub>2</sub>. When all the SiO<sub>2</sub> is added as quartz, the enamel, although apparently well smelted, contains much undissolved SiO<sub>2</sub>. The presence of undissolved SiO<sub>2</sub> decreases the workability and acid-resistance. The results of laboratory and plant tests are recorded. J. A. SUGDEN.

**Control of draining consistencies of enamels by addition of sodium silicate.** D. G. BENNETT (J. Amer. Ceram. Soc., 1931, 14, 495—498).—The addition of 0.25—0.5% Na<sub>2</sub>SiO<sub>3</sub> greatly improves the draining property of an enamel. The dry wt. per unit area (and not the wet wt.) is the only accurate method for controlling the consistency of wet-coat enamels. J. A. SUGDEN.

**Refractories for use at high temperature.** W. H. SWANGER and F. R. CALDWELL (Bur. Stand. J. Res., 1931, 6, 1131—1143).—Crucibles of ThO<sub>2</sub> have been made from material which has been pre-fused in the C arc in an atm. of O<sub>2</sub> and may be used at temps. up to 2200°. Commercial MgO volatilises rapidly between 2000° and 2800°, and crucibles may be used only up

he 1800°; pure MgO crucibles may be employed for toating pure metals at 2000° without danger of contamination of the metal. Crucibles of commercial  $ZrO_2$  containing a small quantity of  $SiO_2$  may be used at 2000°, but only at atm. pressure and in an oxidising atm. BeO when in contact with C at 2000° appears to volatilise less than does MgO under the same conditions. Details are given of the production of crucibles, suitable for melting pure metals, from any of the four oxides; the firing temp. should be 1600—1800°, but  $ThO_2$  and  $ZrO_2$  must be kept out of contact with C at temps. above 1200°.

H. F. GILLBE.

**Machinery and methods of manufacture of sheet glass.** W. E. S. TURNER (Proc. Inst. Mech. Eng., 1930, 1077—1127).

**Electric enamelling furnace.**—See XI. **Glass for wines.**—See XVIII.

## PATENTS.

**Glass and method of making.** M. METH, Assr. to I. R. STEWART (U.S.P. 1,789,658, 20.1.31. Appl., 4.2.27).—The presence of 1% BeO may reduce the coeff. of expansion of a glass by half (larger amounts produce a negligible coeff.) and also increases the tensile strength. The BeO is added as a mineral containing approx. 66%  $SiO_2$ , 20%  $Al_2O_3$ , and 14% BeO. J. A. SUGDEN.

**Zirconium opacifier.** C. J. KINZIE, Assr. to TITANIUM ALLOY MANUFACTURING CO. (U.S.P. 1,789,311, 20.1.31. Appl., 7.9.28).—Na Zr silicate, prepared by roasting finely milled Zr silicate and soda ash, is claimed as opacifier in enamels smelted below 1100°. Above this temp. the advantages are almost wholly lost. If less than the theoretical quantity of  $Na_2CO_3$  required to form the normal silicate is used, the product is non-caking and can be stored in the finely ground state. A typical prep. contains 55%  $ZrO_2$ , 28%  $SiO_2$ , 14%  $Na_2O$ , traces of  $Fe_2O_3$ ,  $TiO_2$ ,  $Al_2O_3$ ,  $P_2O_5$ ,  $CO_2$ , etc.

J. A. SUGDEN.

**Manufacture of articles of silica [astronomical mirrors etc.].** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of E. THOMSON (B.P. 352,375, 28.1.31. U.S., 29.1.30).—The articles comprise a lower layer of quartz sand heated to coalescence in a mould, an intermediate layer built up of a mosaic of, e.g., square or hexagonal pieces of fused quartz and heated to fusion in an electric furnace at a temp. such as to cause a softening of the lower layer, and an upper layer of quartz particles sprayed on by a high-temp. flame to form a smooth, glossy surface.

L. A. COLES.

**Continuous preparation [purification] of kaolin and clay products.** E. MÜLLER (B.P. 352,036, 4.1.30).—The washing apparatus is supplied continuously with water, with Na and/or K water-glass,  $d$  1.33—1.36, and with the raw material, in proportions such that streaks are formed on the surface of the suspension (which usually has to be maintained at  $d$  1.08—1.09); the suspension flowing away is neutralised with HCl and filter-pressed.

L. A. COLES.

**Refractory.** S. M. PHELPS and M. C. BOOZE, Assrs. to AMER. REFRACTORIES INST. (U.S.P. 1,788,123, 6.1.31. Appl., 25.4.28).—The addition of up to 10% of certain substances (e.g., Al salts, alunite,  $Na_2SiO_3$ , cryolite,

$Mg_2P_2O_7$ , MgO, apatite, phosphate rock, CaO,  $CaO \cdot Al_2O_3$ , etc.) to high- $Al_2O_3$  materials before they are pre-calcined greatly reduces the shrinkage in subsequent making up and gives a denser and tougher product.

J. A. SUGDEN.

**Fireproof tile.** C. W. HALL (U.S.P. 1,790,418, 27.1.31. Appl., 14.2.29).—A silicious and carbonaceous mixture (e.g., cinders) is pressed in a mould. A typical analysis is  $SiO_2$  24.4%,  $Fe_2O_3$  1.4%, CaO 16.4%,  $Al_2O_3$  10.6%, small amounts of MgO,  $Na_2CO_3$ , S, etc., and carbonaceous matter 33.6%.

J. A. SUGDEN.

## IX.—BUILDING MATERIALS.

**False setting of cement.** F. WHITWORTH (Cement, 1931, 4, 759—762).—It is suggested that false setting may be caused by the dehydration of a portion of the gypsum during grinding. This crystallises in the mass with sufficient interlocking of crystals to produce an initial hardening without affecting the normal setting of the cement. It is unwise to attempt to eradicate the false set by either reducing or increasing the quantity of gypsum, which should be 1.5—2%  $SO_2$  equiv.

C. A. KING.

**Acid-proof cement mortars.** S. NAGAI (J. Soc. Chem. Ind. Japan, 1931, 34, 191—192 B; cf. B., 1929, 777, 980).—Of the mortars studied, those made with artificial silicious powders (clay extraction residues) have a better binding strength than those made with natural silicious powders. The former also show a smaller decrease in binding strength when acid-cured than the latter. The clay extraction residue should be finely divided, and the alkali silicate solution should have a high  $SiO_2$  content.

N. H. HARTSHORNE.

**Absorption of free lime by cement admixtures.** T. YOSHIOKA, K. KUMAGAE, and H. IJIMA (J. Soc. Chem. Ind. Japan, 1931, 34, 192—193 B).—To test the view that the effectiveness of diatomaceous earth, volcanic ash, blast-furnace slag, and other substances as cement admixtures is due to the free  $SiO_2$  which they contain combining with free CaO, measurements of their absorptive power for free CaO have been made. Wide variations in the amount and rate of absorption were observed. Water-granulated slag absorbed slowly, its initial rate of absorption being, however, unchanged at the end of 6 days. Diatomaceous earth of moderate purity absorbed rather rapidly for 3 to 4 days. The highest rate was shown by  $SiO_2$  gel.

N. H. HARTSHORNE.

**Volume changes in brick masonry materials.** L. A. PALMER (Bur. Stand. J. Res., 1931, 6, 1003—1026).—The variations of vol. with changes of  $H_2O$  content and of temp. of a variety of cement and lime mortars and of bricks have been determined, and the changes of vol. of the mortars during hardening and during alternate wetting and drying have been measured. The difference between the expansion of brick and of mortar is greater when caused by change of  $H_2O$  content than when resulting from change of temp.; plain lime-sand mortars produce the smallest difference, although their initial shrinkage is large. Underburned brick exhibits greater vol. variations than does well-burned brick. The vol. changes in hardened 1:2:9



cement-lime-sand mortar are less than those in a 1:3 cement-sand mortar.

H. F. GILLBE.

**Dimensional changes in clay wares during firing.** T. YOSHIOKA (J. Soc. Chem. Ind. Japan, 1931, 34, 208 B).—The author's apparatus enables clay ware to be heated at definite rates up to 1500°, and the progressive shrinkage to be measured, so that the effects due to varying the composition of the clay and other conditions may be studied.

N. H. HARTSHORNE.

**Aluminous insulating materials at high temperatures.** J. B. BARNITT and R. H. HEILMAN (Chem. Met. Eng., 1931, 38, 390—393, and Proc. Eng. Soc. W. Pa., 1931, 47, 349—361).—The thermal conductivity of monohydrated bauxite and monohydrated  $\text{Al}_2\text{O}_3$  bricks is 0.91 and 2.5 B.Th.U./sq. ft./hr./°F./in. respectively at 870°. The former may be used up to 1040° and the latter to 1370°. The residue from the extraction of aluminous material from bauxite has superior insulating properties, but the shrinkage on drying, when making into bricks, precludes its use.

D. K. MOORE.

**Measuring particles.**—See I.

See also A., Aug., 921, Di- and tri-Ca silicate and Ca aluminate.

#### PATENTS.

**Rotary kilns for burning cement, ore, and similar materials.** VICKERS-ARMSTRONGS, LTD., and L. D. PARKER (B.P. 351,283, 19.7.30).—In a kiln having a number of subsidiary cylinders surrounding the burner end, the inlet passages to the former are not quite radial so that the material falls through without touching the walls, and the inlet ends of the subsidiary cylinders are conical in shape, having those portions which receive the shock of the entering hot material easily renewable.

B. M. VENABLES.

**High-temperature cement.** A. HUTCHINSON (U.S.P. 1,787,625, 6.1.31. Appl., 8.4.30).—A mixture of finely ground  $\text{SiO}_2$  sand (15 pts. by wt.),  $\text{SiO}_2$  brick (3 pts.), Portland cement (2 pts.), and soda ash ( $1\frac{1}{2}$  pts.) is made plastic with water.

J. A. SUGDEN.

**[Clay] emulsion composition.** R. CROSS, Assr. to SILICA PRODUCTS CO. (U.S.P. 1,788,706, 13.1.31. Appl., 9.4.28).—The addition of 20%  $\text{CaSiO}_3$  (especially in the form of Portland cement) greatly enhances the swelling properties of gelatinising clays such as bentonite. Moreover, after the gel has been dried out, the mixture will not hydrate again. This irreversible reaction is utilised in the prep. of paints, waterproofing materials, asphalts, etc.

J. A. SUGDEN.

**Manufacture of artificial travertine or the like.** J. A. RICE, Assr. to the BUBBLESTONE CO. (U.S.P. 1,788,592, 13.1.31. Appl., 26.10.26).—Artificial voids are produced in imitation stone etc. by the random dispersion of masses of foam throughout the cementitious plastic material. A suitable foam is prepared by saponifying an aq. mixture of casein, rosin, and  $\text{NH}_3$ .

J. A. SUGDEN.

**Artificially coloured granule [for roofing etc.].** H. L. LEVIN, Assr. to PATENT AND LICENSING CORP. (U.S.P. 1,788,625, 13.1.31. Appl., 20.2.28).—A porous material (e.g., blast-furnace slag) is crushed to 8—35-mesh and impregnated with a colouring solution (con-

taining salts giving coloured oxides, e.g.,  $\text{FeSO}_4$ ,  $\text{Na}_2\text{CrO}_4$ , etc.) under reduced (and if necessary increased) pressure. The material is dried and roasted at a temp. (approx. 1000°) sufficient to form the colouring agent and to attach it firmly to the surface by incipient fusion.

J. A. SUGDEN.

**Preservative for wood.** GRUBENHOLZIMPRÄGNIERUNG G.M.B.H. (B.P. 352,272, 21.7.30. Ger., 10.8.29).—Solutions are used containing a mixture of a sol. F compound ( $\text{NaF}$ ,  $\text{Na}_2\text{SiF}_6$ , etc.) with more than 20% of a sol. Cr compound (e.g.,  $\text{K}_2\text{Cr}_2\text{O}_7$ ); other preservatives, e.g., nitrophenols, may also be added.

L. A. COLES.

**Glueing wood with starch or starch-containing substances.** E. ABRAMOVITSCH (U.S.P. 1,788,608, 13.1.31. Appl., 6.10.27).—A paste of starch-containing material (e.g., potato-, rice-, or corn-flour) and tragacanth with  $\text{MgCl}_2$  or dextrin and trioxymethylene is applied to the wood plys, which are then pressed at 90°/2—8 atm.

D. WOODROFFE.

**[Composite] materials particularly for wearing surfaces of floor and stair treads etc.** J. H. BENNETT, JUN. (B.P. 352,555, 10.7.30).

**Composite plates, boards, etc. [from wood sheets coated with artificial resin].** H. PIORNIK (B.P. 353,094, 28.4.30).

**Drying sludge.**—See I.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Properties of austenitic grey cast irons.** M. BALLAY (Compt. rend., 1931, 193, 47—49).—All samples were from cylindrical castings 25 mm. diam. Addition of from 5 to 12% of Cu to a cast iron containing C 2.71, Si 1.45, Mn 1.30, Ni 14.82, Cr 1.40, Cu 0.30%, diminished by about 65% corrosion by 10%  $\text{H}_2\text{SO}_4$  or HCl. In such a metal about 10% Cu is sol. Addition of Cr or Al somewhat diminishes corrosion by 5%  $\text{HNO}_3$  and also oxidation on heating; 8% Cr renders the metal almost unoxidisable. An austenitic cast iron, C 3.01, Mn 1.48, Si 2.48, Ni 14.09, Cr 3.59, Cu 5.95%, though softer than an ordinary pearlitic sample at low temp., is substantially harder at 800°. Increasing the Si to 4.06% increases resistivity by 27%.

C. A. SILBERRAD.

**Cold-working of iron.** W. BRONIEWSKI and J. KRÓL (Compt. rend., 1931, 193, 38—40).—Curves show for French Armco iron (less than 0.15% impurities) the relations (a) between degree of cold-working and tensile strength, elastic limit, Brinell hardness, elongation, and striction (cf. B., 1913, 605); (b) for cold-worked samples annealed for 1.5 hr., between temp. of annealing (0—1000°) and hardness and average size of crystals; and (c) between hardness determined by Hanriot's method (cf. B., 1913, 606) of a sample annealed at 650° and the number of times annealed. Series (b) shows that annealing below 520° has little effect (cf. B., 1913, 701); (c) that hardness decreases, reaching a limit after about 20 times annealing.

C. A. SILBERRAD.

**Tempering and ageing of extra-soft steels.** A. BATES (Compt. rend., 1931, 193, 35—38).—Extra-soft Thomas steel (0.026% C) tempered at 600—1000°

shows, after 30 days' ageing at 12°, marked increases in tensile strength, elastic limit, and hardness, and decrease in ductility. The increase in tensile strength is a max. for tempering at 700° and 950°, a min. at 850°. These effects are due to the varying solubility of cementite in  $\alpha$ -ferrite, or (above 900°) of C in  $\gamma$ -ferrite, as shown on a revised equilibrium diagram.

C. A. SILBERRAD.

**Influence of chemical composition and heat-treatment of steel forgings on machinability with shallow cuts.** T. G. DIGGES (Bur. Stand. J. Res., 1931, 6, 977—992).—The life of cutting tools operating at a const. feed and depth of cut, but variable cutting speed, on Cr-V, Ni-Cr, Cr-Mo, 3.5% Ni, and 0.4% C steels, heat-treated to possess tensile strengths from 75,000 to 220,000 lb./sq. in., has been determined. With the exception of the annealed Ni-Cr steel, the plain C steel is the most difficult to machine, and yields the poorest surface finish. The efficacy of elements added to improve machinability at shallow cuts depends on the tensile strength of the steel; for steels having a strength of about 90,000 lb./sq. in. Cr and Mo are the most effective, but for steels of considerably higher strengths Ni-Cr or Cr-V are more satisfactory. For a given tensile strength the permissible cutting speed is independent of the method of heat-treatment. The performance of the 18% W type of high-speed tool steel is improved by adding 3.5—5% Co and increasing the hardening temp.

H. F. GILLBE.

**Graphitisation of steel at low temperature.** A. PORTEVIN and P. CHEVENARD (Compt. rend., 1931, 193, 169—171; cf. B., 1927, 110).—Contrary to previous conclusions, graphitisation can be effected in a steel containing 1.6% C, 0.28% Si, and 0.16% Mn on annealing below Al. A sample tempered at 1180° in  $H_2O$ , and consequently consisting about equally of martensite and austenite, was heated at the rate of 250° per hr. to 602°, kept there for 7 hr., and cooled slowly. Dilatometric observations showed decarburisation of the martensite and austenite, the transformation  $\gamma \rightarrow \alpha$ , and return to Fe- $\alpha$ +Fe<sub>3</sub>C equilibrium, followed by an expansion indicating the graphitisation of the cementite. The result is probably due to the extreme fineness of the cementite grains (cf. B., 1929, 1045).

C. A. SILBERRAD.

**Grain size and fatigue-resistance in soft steel: effects of cold-working, tempering, and overheating.** CAZAUD (Compt. rend., 1931, 192, 1558—1560).—An extra soft steel (a) containing C 0.10, Mn 0.45, Si 0.03, S 0.08, P 0.03%, and the same after (b) tempering at 925° and slow cooling, (c) tempering at 925° and rapid cooling, and (d) tempering at 1125° and slow cooling (i.e., overheating) gave (when drawn into bars 20 mm. diam.) the following results (in this order), breaking load, elastic limit, elongation (%), hardness (Ménager), fatigue limit (kg./sq. mm.) for rotatory flexion, and number of crystals per sq. mm.: (a) 56.8, 53.5, 15.5, 2, 29.7, —; (b) 39.5, 26.7, 35, 8.6, 25.6, 400; (c) 41.2, 28.1, 31, 30.2, 26, 900; (d) 42, 26.5, 34, 0.60, 20.8, 150.

C. A. SILBERRAD.

**Determination of small quantities of hydrogen sulphide. Determination of sulphur in small**

**samples of steel.** S. G. CLARKE (Analyst, 1931, 56, 436—444).—The S in steels containing less than 0.1% of S may be determined by dissolving 0.1 g. in 5 c.c. of  $H_2SO_4$  (1 : 3 by vol.) in an atm. of  $H_2$  and absorbing the  $H_2S$  formed in 2 c.c. of 10% NaOH. After acidification with dil.  $H_2SO_4$ , treatment with I in  $CCl_4$  gives a quant. yield of HI and S. The diminution of colour of the I solution may be measured colorimetrically, the results being comparable with those of the gravimetric process. Black rubber must be used for all connexions.

T. McLACHLAN.

**Magnetic properties of permivar.** H. KUHLEWEIN (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 22—88).—The characteristic magnetic properties of permivar are attributed to the effects of internal stress distribution probably caused by the presence of impurities. An increase in the permivar characteristics of alloys in the Elmen range of composition can be obtained by increasing the Co content or by annealing at higher temps. followed by very slow cooling. The Fe-Co-Ni system has a second field nearer the Fe-Ni side of the ternary diagram in which the alloys also display permivar characteristics.

A. R. POWELL.

**Attack of metals by phosphoric acid.** A. PORTEVIN and A. SANFOURCHE (Compt. rend., 1931, 192, 1563—1565).—The action of (a) pure  $H_3PO_4$ ,  $d$  1.581, (b) the same,  $d$  1.262, and (c) industrial acid,  $d$  1.262, prepared from superphosphate, on pure commercial metals is shown by curves. Mg and Zn are attacked with great rapidity, Bi and Ag not at all. The (decreasing) order of attack of the remainder is: for (a) Al, Pb, Cd, Fe, Sn, Sb, Ni, Cu; for (b) Al, Fe, Pb, Cd, Sn, Cu, Sb, Ni; and for (c) Cd, Al, Sn, Fe, Cu, Ni, Sb, Pb. As a rule attack is more violent with the more conc., and still more so with the commercial, acid, save in the case of Pb, which is practically unattacked by this latter, due to formation of  $PbSO_4$ . The resistance of Bi is due to formation of phosphate. In presence of 10% HCl Ag alone is unattacked.

C. A. SILBERRAD.

**Attack of alloys by phosphoric acid.** A. SANFOURCHE and A. PORTEVIN (Compt. rend., 1931, 193, 53—55; cf. preceding abstract).—Attack by the acids previously studied on alpac, duralumin, brass, bronze, cupronickel, German silver, antimonial Pb, and 10 steels was examined. All alloys containing Al or Cu were severely attacked, as also was antimonial Pb. Of the steels examined only that with 21% Cr and 8% Ni withstood all three acids; 13% Cr steel was not attacked by (a) or (b); that with 10% Cr and 23% Ni by (b) and but slightly by (a); those with 12% Cr and 32% Ni and with 11% Cr and 52% Ni slightly by (a). All other steels were severely attacked. In the cupronickels and brasses both metals were equally attacked; in bronze the Sn, and in attacked Cr steels the Cr was most and Ni least. Of Fe alloys containing 65% Cr, 38% Mo, 30% Si, 39% Ti, 71% W, 64% Zr, 58% Ta + 14% Nb, 35% U + 7% V (all in powder), only Fe-Cr and Fe-Si were resistant. HCl ( $d$  1.161),  $H_2SO_4$  ( $d$  1.831), and  $HNO_3$  ( $d$  1.384) attacked all these Fe alloys except ferrosilicon, whilst Fe-Cr rapidly and Fe-Zr slowly attained passivity in  $HNO_3$ .

C. A. SILBERRAD.

**Determination of phosphorus in steel, alloy steels, and cast iron.** N. D. RIDSDALE (Analyst, 1931, 56, 452—454).—The work of Etheridge (B., 1931, 297) is criticised.  $\text{KMnO}_4$  is necessary to oxidise P to  $\text{H}_3\text{PO}_4$ ;  $\text{HNO}_3$  is insufficient. 30 c.c. of  $\text{HNO}_3$  (d 1.20) are enough to dissolve 2 g. of drillings. Pig iron contains 0.08—0.42% (av. 0.15%) Ti. T. McLACHLAN.

**Ballistic measurements on materials [iron-nickel alloys] of high permeability.** H. NEUMANN (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 55—71).—Two ballistic methods for measuring the magnetic properties of alloys of high permeability are described and the effect of additions of 1—30% Co to a 1:1 Fe-Ni alloy has been determined. The initial and max. permeability reach max. with 1% Co and min. with 10% Co and the remanance and coercivity have min. values with 1% Co. A. R. POWELL.

**Influence of arsenic on the dezincification of brass.** G. MASING (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 35—54).—The inhibiting effect of As on the dezincification of brass occurs only with  $\alpha$ -brass, increases with the As content from 0.01 to 0.08% As, and decreases slightly with decreasing Cu content. The presence of As in ( $\alpha + \beta$ )-brass has no effect on its resistance to corrosion. In corrosive media arsenical  $\alpha$ -brass becomes coated with a thin film of As which has a smaller cathodic overpotential than has the brass, and the protective effect of As is ascribed to this fact, which results in the more ready reduction of the dissolved  $\text{O}_2$  in the electrolyte. Various theories on the electrochemical mechanism of corrosion are discussed. A. R. POWELL.

**Aluminium alloys containing beryllium.** G. MASING and L. KOCH (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 15—24).—Addition of up to 8% Be to sand- and chill-cast duralumin,  $\gamma$  alloy, or Cu-Al alloys has no beneficial effect on the tensile properties, but 4—8% Be increases the resistance to wear of 12% Cu-Al alloys. Addition of <1% Be to silumin modified with Na improves the elongation. Be and Si form a ternary eutectic with Al containing 13% Si and 1—1.5% Be. A. R. POWELL.

**Electroplating aluminium with copper.** R. WEINER (Z. Elektrochem., 1931, 37, 349—356).—The Al is plated in a cyanide bath after the oxide film has been removed and the surface roughened by treatment with 10% NaOH and 2% HCl followed by anodic attack in  $N\text{-CH}_2\text{Cl}\cdot\text{CO}_2\text{H}$  and treatment with aq.  $\text{Na}_2\text{CO}_3 + \text{NaHCO}_3$ . An excellent deposit is obtained in this way. Plating in a  $\text{CuSO}_4$  bath is less satisfactory, owing to poor throwing power. R. CUTHILL.

**Improvement of the durability of chromium plate.** R. J. PIERSON (Chem. Met. Eng., 1931, 38, 386—389).—Commercial Cr-plate is <0.00001 in. thick. In plating there is a tendency to deposit more Cr on projections than in recesses. The range of current density for the production of a bright deposit is small. Cr-plating on porous surfaces, cast Fe high in free C, and on brass because of zincification of Cr, is not satisfactory. The article should be given an intermediate coat of Ni, or, if porous, Ni followed by Cu the surface of which is flowed on a polishing wheel, and then Ni.

Const. voltage and thermostatic temp. control of the plating bath should be used and the plating solution continuously circulated and filtered. Replating of damaged Cr plate is not satisfactory. D. K. MOORE.

**Electrodeposition of lead dioxide on metals and protection against corrosion.** N. ISGARISHEV and A. KUSNEZOVA (Z. Elektrochem., 1931, 37, 359—362).—By electrolysis of a solution of  $\text{Pb(OH)}_2$  in NaOH containing certain reducing substances, such as resorcinol, tannin, or glucose, a firm and elastic coating of  $\text{PbO}_2$  may be deposited on Cu or brass. Similar deposits may be obtained on Fe, but if resistance to bending is required a coating of Cu should be deposited first. The  $\text{PbO}_2$  prevents corrosion by the air, by 3% NaCl solution, and by 5%  $\text{H}_2\text{SO}_4$ . R. CUTHILL.

**Electrodeposition of silver from argentocyanide solutions.** II. S. GLASSTONE and E. B. SANIGAR (Trans. Faraday Soc., 1931, 27, 309—312; cf. B., 1929, 1018).—If KCNO at concentrations exceeding 0.1N is added to a  $\text{KAg(CN)}_2$  solution 1.5N in respect of  $\text{Na}_2\text{CO}_3$  the deposit of Ag obtained on electrolysis is microcryst. and exceptionally uniform. Since the KCNO and also  $\text{CS}_2$  and similar "brightening agents" cause a decrease in the cathodic polarisation, it seems probable that high cathodic polarisation tends to lead to matt deposits, whereas low polarisation favours "bright" deposits. Deposition potential is not, however, the only factor which affects the nature of the deposit. R. CUTHILL.

**Alloyability of beryllium with calcium and magnesium.** W. KROLL and E. JESS (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 29—32).—Be shows no sign of alloying with Mg in an A atm. at the b.p. of Mg. In boiling Ca the Be lumps become covered with a thin shell of an alloy of 71% Be and 29% Ca. Alloys of Be with Cu and Mn can be dissolved in boiling Mg, but at 20° the resulting ternary alloys all show numerous segregations of Be and their mechanical properties are poor. A. R. POWELL.

**Alloys of beryllium with iron.** W. KROLL (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 33—34).—Fe alloys with 12% Cr, 1% Be, and >8% Ni have a Brinell hardness of 150—180 after quenching from 1200°; this hardness increases with ageing temperature to a max. of 400 at 700—750°. The aged alloys are suitable for the manufacture of non-rusting springs which retain their strength up to a red heat. A. R. POWELL.

**Analytical chemistry of beryllium.** II. Gravimetric determination of beryllium in minerals, rocks, and steel. II. FISCHER and G. LEOPOLDI (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 1—14).—The rock is fused with  $\text{Na}_2\text{CO}_3$  and the  $\text{SiO}_2$  separated by evaporation with  $\text{H}_2\text{SO}_4$ . The filtrate is treated with a slight excess of aq.  $\text{NH}_3$  and the ppt. of  $\text{Fe(OH)}_3$ ,  $\text{Al(OH)}_3$ , and  $\text{Be(OH)}_2$  collected, washed with dil. aq.  $\text{NH}_4\text{NO}_3$ , redissolved in HCl, and reprecipitated as before to remove Mg. If much Al is present it is then separated by the  $\text{Et}_2\text{O-HCl}$  method, the filtrate evaporated to dryness, and the residue dissolved in very dil. HCl. The solution or the main solution, if little Al is present, is neutralised with aq.  $\text{NH}_3$ , made just acid with AcOH, and treated with a 5% solution of 8-hydroxyquinoline in 2N-AcOH to remove Al and Fe.

The filtrate is treated with a slight excess of aq.  $\text{NH}_3$  and the  $\text{Be}(\text{OH})_2$  collected, ignited, and weighed; it should be tested for  $\text{SiO}_2$  by evaporation with  $\text{HF}$  and  $\text{H}_2\text{SO}_4$ , followed by ignition at  $1200^\circ$ . For the determination of Be in special Ni-Cr steels the alloy is dissolved in  $\text{HCl}$ , the Fe oxidised with  $\text{HNO}_3$  and removed by the  $\text{Et}_2\text{O}$  method, the Cr oxidised with  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  and  $\text{AgNO}_3$ , and the Be precipitated with aq.  $\text{NH}_3$ . The ppt. is purified by repeating the last two operations, igniting, evaporating with  $\text{HF}$  and  $\text{H}_2\text{SO}_4$ , and igniting at  $1200^\circ$  to  $\text{BeO}$ . A. R. POWELL.

**Production of metallic antimony from its ores by electrolysis.** N. A. ISGARISCHEV and S. A. PLETENEV (*Z. Elektrochem.*, 1931, 37, 363—365).—Massive deposits of Sb may be obtained by electrolysis of a solution of  $\text{Sb}_2\text{S}_3$  in 10%  $\text{NaOH}$  using an Fe cathode, and if the cathode deposit is protected from polysulphides formed at the anode by enclosing the latter in a porous membrane and adding KCN the current yield may average 75%. Small amounts of As in the electrolyte do not separate with the Sb. Sb may therefore be manufactured from its ores by extracting with  $\text{NaOH}$  and electrolysing. R. CUTHILL.

**Hard and soft rubber for pickling tanks.** H. E. FRITZ (*Chem. Met. Eng.*, 1931, 38, 396—397).—A suitable lining for pickling tanks consists of a combination of hard and soft rubber plies arranged so that the hard plies overlap each other and form expansion joints, with a dovetailed wood sheathing. This construction may be used for dil.  $\text{H}_2\text{SO}_4$  up to temps. of  $65^\circ$ . With a sheath of 4-in. acid-proof brick and free circulation of air round the outside of the tank, acid up to temps. of  $100^\circ$  may be used. D. K. MOORE.

**Dilatation in electrolytically deposited metals.** C. MARIE and N. THON (*Compt. rend.*, 1931, 192, 31—32; cf. A., 1922, ii, 648).—Using as cathode a thin sheet of Pt, Au, Ag, or Cu, covered on one side with insulating material, dilatation or contraction of the deposit is determined by its change of shape. With a current of 1.1 amp./sq. dm. and 200 g.  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  per litre dilatation occurred with matt-surface Pt only; with the same solution with addition of 1%  $\text{H}_2\text{SO}_4$  it sometimes occurred with Ag and Au, the dilatation occasionally changing to contraction (attributed to formation of  $\text{Cu}^+$  salt). With more acid contraction always occurred, but dilatation invariably with a solution  $M$  in  $\text{CuCl}$  and 4.5*M* in  $\text{NaCl}$  containing  $\text{HCl}$ , and also with solutions of  $\text{ZnCl}_2$ ,  $\text{ZnSO}_4$ , or  $\text{Zn}(\text{CN})_2$ . The effects are not due to H. C. A. SILBERRAD.

**Measurement of tension in electrolytic metallic deposits.** C. MARIE and N. THON (*Compt. rend.*, 1931, 193, 233—236; cf. preceding abstract).—The pressure,  $p$ , in dynes per sq. cm. of the electrolytic deposit is approx.  $f/3$ , where  $f$  is the force, in dynes, which, applied at the edge of the plate on which the deposit is made at a distance  $l$  from the supported end, produces the same deflexion,  $\lambda$ , as that caused by the contraction of the deposit. Values of  $\lambda$ ,  $p$ , and  $f$  for deposits of Ni on plates of Pt, Ag, Cu, Ni, Fe, Al, and Ta are given. The surface tension of the deposit is  $pl^2/2\lambda$ . C. A. SILBERRAD.

**Influence of electromagnetic waves on resistivity and hardness of metals and alloys.** G. MABOUX

(*Compt. rend.*, 1931, 193, 27—29; cf. B., 1931, 206).—The resistivity of cast iron, special steel, a Ni-Cr-Mn steel, and an Al alloy subjected to electromagnetic waves diminished. The bars after exposure showed alternate rings, light and dark, differing in hardness. The hardness of all but the cast Fe was increased.

C. A. SILBERRAD.

**Use of special non-ferrous metals and alloys in marine engineering and shipbuilding.** J. W. DONALDSON (*Metallurgia*, 1931, 4, 77—80).

**Electric enamelling furnace.** Zinc sulphate electrolysis.—See XI. Tanning substances and metals.—See XV. Corrosive soil areas.—See XVI.

See also A., Aug., 888, Semi-silvering of interferometer plates. 900, Cu-Sn, Ca-Na, and Pb alloys. 906, Au sols. Colloidal Au. 914, Electrochemical behaviour of Pt in  $\text{HCl}$  solution. Electrolytic reduction of  $\text{H}_2\text{PtCl}_6$  in  $\text{HCl}$  solution. 917, Influence of stress on corrosion. 919, Ni catalysts for hydrogenation. 924, Action of  $\text{H}_2\text{SO}_4$  on Ni-Cu matte.

#### PATENTS.

**Smelting furnace.** J. T. McCOURT (U.S.P. 1,789,531, 20.1.31. Appl., 10.6.26).—Preheated ore from a rotary kiln is fed into an inclined shaft which terminates at the lower end in a hearth. The hearth and shaft are heated by a number of fuel burners, the ore being reduced directly by CO. A hump at the lower end of the shaft holds the charge at the point of max. temp. of the furnace. C. A. KING.

**Alloy for manufacture of cast iron.** A. F. MEEHAN, ASSR. to MEEHANITE CORP. (U.S.P. 1,790,552, 27.1.31. Appl., 29.8.28).—Graphitisation of cast Fe is effected by treatment with an "alloy" containing Ca and Si, with or without Mg, Ni, Cr, etc.; e.g., Ca 35, Si 51, Ni 5, Mg 9%. E. H. BUCKNALL.

**Annealing grey iron.** R. J. COWAN, ASSR. to SURFACE COMBUSTION CO., INC. (U.S.P. 1,789,136, 13.1.31. Appl., 10.7.28).—Grey cast Fe is heated at  $780$ — $900^\circ$  for 5 min.—1 hr. to initiate graphitisation and then cooled. C. A. KING.

**Process of heat-treating steel.** F. A. FAHRENWALD (U.S.P. 1,787,977, 6.1.31. Appl., 25.6.28).—Steel is heated rapidly in continuous annealing furnaces to a temp. above the crit. point and cooled below that temp. in a moderately oxidising atm. containing  $\text{H}_2\text{O}$ ,  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{N}_2$  produced by incomplete combustion of town gas. An adherent coat of oxide is formed in the early stages and further oxidation is slight. E. H. BUCKNALL.

**Magnetic material.** J. H. WHITE and V. LEGG, ASSRS. to BELL TELEPHONE LABS., INC. (U.S.P. 1,787,606, 6.1.31. Appl., 30.11.29).—Magnetic materials containing Ni and Fe, with or without other elements, are rendered less hot-short in the early stages of rolling by alloying with up to 4% Cu, which facilitates the production of finely disseminated materials by comminution. E. H. BUCKNALL.

**Heat-treatment of magnetic material.** G. W. ELMEN, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,788,017, 6.1.31. Appl., 6.7.25).—The permeability

at low field strength ( $<0.2$  gauss) of alloys of the "permalloy" type, containing  $>45\%$  (usually 78–80%) Ni, remainder Fe, is improved by heating to  $1100^\circ$ , slow cooling, reheating above the magnetic change point, say  $600^\circ$ , and cooling fairly rapidly, care being taken not to set up strains in the material.

E. H. BUCKNALL.

**Tough stable-surface alloy steel.** S. M. STOODY, Assr. to STOODY Co. (U.S.P. 1,790,177, 27.1.31. Appl., 14.11.28).—Steels containing about 2% C, 31.75% Cr, 10% Ni, and 4.75% Si have high hardness values, good resistance to acids, heat, and abrasion, and are especially useful as welding rods, as, *e.g.*, in mounting sintered WC cutting tools.

E. H. BUCKNALL.

**Stable-surface alloy steel.** R. P. DE VRIES, Assr. to LUDLUM STEEL Co. (U.S.P. 1,788,281, 6.1.31. Appl., 20.1.26).—An Fe alloy for general engineering and structural purposes contains approx. 1% Cr, 2% Si, 1% Cu, and  $<1\%$  C.

C. A. KING.

**Case-hardening metal.** P. W. and E. B. SHIMER, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,789,257, 13.1.31. Appl., 22.6.27).—Small quantities of Ca cyanamide or other cyanide-forming compound are added regularly to a bath of fused salts, *e.g.*, a mixture of  $\text{CaCl}_2$  and NaCl, to maintain the efficiency.

C. A. KING.

**Treatment [rust-proofing] of metals.** F. D. BURKE, Assr. to PASSIVATION PRODUCTS Co. (U.S.P. 1,789,805, 20.1.31. Appl., 16.2.29).—The attack of acid cleaning baths on metals is inhibited by the addition of a small quantity of  $\alpha$ -amino-acids, *e.g.*, hydrolysed animal proteins including keratin.

C. A. KING.

**Casting copper or equivalent copper alloys.** AMER. METAL Co., LTD., and W. F. EPPENSTEINER (B.P. 353,205, 24.6.30. Addn. to B.P. 305,998).—The moulds are cooled with water at  $<100^\circ$  ( $88$ – $93^\circ$ ).

**Cold brass rolling.** J. C. SHARP, Assr. to STANDARD OIL Co. (U.S.P. 1,789,054, 13.1.31. Appl., 30.9.29).—An alkaline compound of a petroleum sulphonic acid is applied to the wetted surface of a soft metal before entering the rolls.

C. A. KING.

**Zinc alloy.** L. E. WEMPLE and F. A. WARREN, Assrs. to ILLINOIS ZINC Co. (U.S.P. 1,789,854, 20.1.31. Appl., 18.10.29).—An alloy of substantially greater hardness than Zn contains Zn 100 pts., Cu 0.25–2 pts., and Ag 0.1–20% of the Cu.

C. A. KING.

**Preparation of a metallic colloid [lead].** F. E. BISCHOFF, Assr. to H. J. ULLMANN (U.S.P. 1,787,659, 6.1.31. Appl., 12.10.26).—The metal is disseminated at temps. below  $30^\circ$  by striking an arc between Pb electrodes under an aq. solution ( $p_H$  7–8) of gelatin, or other dispersing agent, and an aliphatic polyhydroxy-compound, usually a saccharide, which protects the colloid against oxidation.

E. H. BUCKNALL.

**Process for purifying [bearing] metals.** W. J. MERTEN, Assr. to WESTINGHOUSE ELECTRIC AND MANUFACTURING Co. (U.S.P. 1,790,164, 27.1.31. Appl., 4.6.27).—An addition of 1 lb. of  $\text{CaC}_2$  is made to 50 lb. melts of Pb-bronze bearing metals with the object of improving the distribution of the Pb, removing the oxide from the melt, and strengthening the Pb by the presence of Ca.

E. H. BUCKNALL.

**Amalgamator [for gold ores].** C. S. WHITE, Assr. to J. E. OGDEN and W. J. SCHEUSS (U.S.P. 1,787,923, 6.1.31. Appl., 26.9.28).—Removal of Au from previously ground sands is effected in two slightly sloping troughs, placed one above the other and rocked by mechanical means. Screens in the bottom of the upper trough retain coarse particles of Au while fine particles ("flour" Au) are caught in Hg cups in the lower trough.

E. H. BUCKNALL.

**Chromium plating.** J. A. HANLEY and W. L. PINNER, Assrs. to GEN. SPRING BUMPER CORP. (U.S.P. 1,787,477, 6.1.31. Appl., 24.1.27).—A Ni-plated article is cleaned firstly as a cathode in an alkaline bath, then in an acid bath, and dipped while wet into a Cr-plating bath for about 5 sec. before the current is turned on.

C. A. KING.

**Manufacture of light metal [magnesium] alloy.** E. C. BURDICK, Assr. to DOW CHEM. Co. (U.S.P. 1,788,616, 13.1.31. Appl., 11.12.24).—An alloy contains  $>80\%$  (93%) Mg, not more than 2% (0.7%) Cu or 0.5% (0.25%) Mn, and  $>2\%$  (6%) Al. It may be prepared by adding a Cu-Mn alloy to a molten rich Mg-Al alloy.

C. A. KING.

**Removal of arsenic from [tungsten] ores.** J. H. BRENNAN, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,790,088, 27.1.31. Appl., 8.12.26).—As contents up to 2% are almost entirely eliminated by treating the finely ground ore with S vapour, usually produced from S mixed with the ore, at about  $800^\circ$ .

E. H. BUCKNALL.

**Treatment [preventing corrosion] of thorium.** E. S. DAVENPORT, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,787,672, 6.1.31. Appl., 4.6.28).—Th is rendered resistant to atm. corrosion by anodic treatment in a bath consisting of glacial AcOH together with up to 30% of  $\text{HNO}_3$ .

E. H. BUCKNALL.

**Production of beryllium alloys.** L. J. KEELER, Assr. to BERYLLIUM CORP. OF AMER. (U.S.P. 1,790,155, 27.1.31. Appl., 14.11.25).—Be alloys, *e.g.*, Be-Cu, may be formed by electrolysis of a fused salt bath, containing a Be fluoride, to which additions of oxides or other compounds of Be and of the alloying metal or metals are made either simultaneously or alternately.

E. H. BUCKNALL.

**Flux for welding copper and steel.** E. J. and N. E. SCHAFFNER, and K. E. FIELD (U.S.P. 1,787,553, 6.1.31. Appl., 12.4.30).—A flux for joining Cu to Fe consists of a mixture of 2 pts.  $\text{Na}_2\text{B}_4\text{O}_7$ , 1 pt.  $\text{CuSO}_4$ , and 2 pts. of a pulverised alloy containing 10 pts. Cu, 25 pts. Zn, and 1.25 pts. Pb.

C. A. KING.

**Extraction of metal values from ores.** A. E. SMALL (U.S.P. 1,789,932, 20.1.31. Appl., 3.1.28).—Sulphide ores are roasted under conditions which result in max. solubility of non-ferrous metals, *e.g.*, Ni, Cu, etc., with the exception of precious metals, which remain with insol.  $\text{Fe}_2\text{O}_3$  in the residue after leaching the roasted product. The residue is separated by selective gravitation.

C. A. KING.

**Roasting and/or sintering fine ores etc.** A. D. H. L. FASSOTTE (U.S.P. 1,789,895, 20.1.31. Appl., 17.11.27).—Threads of small diam. are expressed from a die almost vertically against a vibrationless conveyor belt

so that the ore is broken into short lengths, which are allowed to dry before the introduction into the blast roasting operation. C. A. KING.

**Treatment of complex ores by flotation.** W. SCHAFER (U.S.P. 1,788,332, 6.1.31. Appl., 18.9.29).—The use of simple or complex metallic salts of  $H_2S_2O_4$  as flotation agents activates the separation of  $CuS$  and  $PbS$  in the froth and depresses  $ZnS$ .

C. A. KING.

**Foundry facings.** B. F. WALLACE (U.S.P. 1,787,964, 6.1.31. Appl., 18.12.26).—A facing composition for sand moulds contains carbonaceous dust made from retort coke, the particles of which are prevented from agglomerating by coating with a  $H_2O$ -sol. deflocculating agent. The dust is mixed with a highly colloidal clay such as bentonite.

E. H. BUCKNALL.

**Acetylene.**—See II. **Waste gases from contact plants.**—See VII. **Kilns.**—See IX. **Face plate.**—See XI.

## XI.—ELECTROTECHNICS.

**Theory of coreless induction furnaces.** W. ES-MARCH (Wiss. Veröff. Siemens-Konz., 1931, 10, [2], 172—196).—Equations are derived for calculating the efficiency of high-frequency induction furnaces and the most suitable furnace and crucible dimensions for a given current frequency.

A. R. POWELL.

**Power cost on an electric enamelling furnace.** M. E. MANSON (J. Amer. Ceram. Soc., 1931, 14, 499—501).—Operating 12 hr. per day the consumption varied from 7 lb. Fe per kw.-hr. for light loads to 13 lb. per kw.-hr. for heavy loads.

J. A. SUGDEN.

**Theory of the lead storage battery.** C. LIAGRE (Compt. rend., 1931, 193, 236—238).—Experiments are described showing that the brown material from the positive plate of a Pb storage battery differs chemically in no way from ordinary  $PbO_2$ , and that both positive and negative plates are sulphated and desulphated in conformity with Faraday's law.

C. A. SILBERRAD.

**Rapid determination of current efficiency in the electrolysis of a zinc sulphate electrolyte.** CONSOL. MIN. & SMELTING Co. (Trans. Amer. Electrochem. Soc., 1931, 60, 1—7).—The apparatus described gives a reliable indication of the electrolyzing quality of a  $ZnSO_4$  solution before it is discharged from the leaching plant to the cell rooms, the time required being only 45 min. The principle of the procedure is that if Zn is deposited from an electrolyte free from more electropositive impurities, using a  $Pb/PbO_2$  anode and a Zn cathode, there will be a min. evolution of  $H_2$  from the cathode, and the current efficiency of Zn deposition will be nearly 100%. On the other hand, if impurities such as As, Sb, etc. are present, they will tend to lower the  $H_2$  overvoltage at the cathode and  $H_2$  will be liberated in preference to Zn. Measurement of the amount of  $H_2$  liberated in a definite interval of time gives, therefore, a measure of the purity of the solution and the current efficiency that can be expected at any given current density.

E. S. HEDGES.

**Nature of the dielectric loss in oils.** E. BORMANN and A. GEMANT (Wiss. Veröff. Siemens-Konz., 1931,

10, [2], 119—128).—The effect of additions to transformer and cylinder oils on their dielectric loss at temp. down to  $-40^\circ$  has been investigated. No direct action in increasing the losses was found to be produced by dipolar substances.

A. R. POWELL.

**Electrical purification of gases.** W. DEUTSCH (Ann. Physik, 1931, [v], 9, 249—264).—It is shown that the suspended particles carried by the electric wind in an electric gas-cleaning plant require just as much time for their separation out of the wind as those in a gas at rest.

W. GOOD.

**Cr plate. Plating Al. Depositing  $PbO_2$ . Sb. Ag.**—See X.

See also A., Aug., 914, **Behaviour of Pt in HCl solution. Reduction of  $H_2PtCl_6$  in HCl solution.** 926, **Determination of small quantities of alkalis in  $H_2O$ -insol. acids ( $H_2WO_4$ ).** 928, **High-temp. furnace.** 929, **New cell for electrodialysis.**

## PATENTS.

**Electrodes for electrolytic cells.** K. ROTH, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,790,248—9, 27.1.31. Appl., [A] 21.1.26, [B] 7.2.28. Renewed 15.7.30. Ger., [A, B] 22.1.25).—An electrode comprising a framework divided by partitions into superposed pockets having permeable metal (A) front walls, (n) front and back walls, and connected by pipes for leading evolved gases upwards, is claimed.

J. S. G. THOMAS.

**[Production of metallic salts and alkalis by] electrolysis.** P. FIREMAN, Assr. to MAGNETIC PIGMENT Co. (U.S.P. 1,788,512, 13.1.31. Appl., 9.5.27).—An electrolytic cell comprising a sol. anode, a perforated cathode, and closely adjacent porous diaphragm at one side of the cell, spaced porous separators dividing the cell into anode and cathode compartments, and means for supplying electrolyte to and withdrawing products from the separate compartments, is claimed.

J. S. G. THOMAS.

**Dry cell.** G. W. HEISE, Assr. to NAT. CARBON Co., INC. (U.S.P. 1,788,870, 13.1.31. Appl., 2.12.24).—A dry cell of the bag type comprising a sheet of cellulosic material, e.g., paper, treated with  $ZnCl_2$ , and cemented to the cathode by the gelatinised cellulose so formed, is claimed.

J. S. G. THOMAS.

**Active material for storage batteries.** W. GARDINER and H. D. STALEY, Assrs. to VULCANITE, INC. (U.S.P. 1,788,571, 13.1.31. Appl., 2.2.27).—Active material containing  $PbO$ ,  $PbO_2$ , and a Ta compound for absorbing gases is claimed. Thus a thick paste composed of 100 pts.  $PbO_2$ , 100 pts.  $PbO$ , 4 pts.  $TaO_2$ , and  $H_2SO_4$  (d 1.050) may be used.

J. S. G. THOMAS.

**Method of activating filaments.** L. THOMPSON, Assr. to VICTOR TALKING MACHINE Co. (U.S.P. 1,790,449, 27.1.31. Appl., 1.4.25).—Filaments, e.g., of Pt, treated with Hg or an amalgam, e.g., of Hg and Zn, and one or more activating substances, e.g., Ba, Sr, are heated to drive off Hg.

J. S. G. THOMAS.

**Electrical resistance element.** A. H. HEYROTH, Assr. to GLOBAR CORP. (U.S.P. 1,787,749, 6.1.31. Appl., 4.10.27).—An extruded mixture of  $SiC_2$ , bentonite, C,

and  $\text{H}_2\text{O}$  is dried in air at about  $100^\circ$ , heated at above  $815^\circ$ , and finally fired at above  $1149^\circ$  for a much shorter period to glaze the outer surface. J. S. G. THOMAS.

**Electron-emission material.** J. W. MARDEN and H. C. RENTSCHLER, ASSRS. to WESTINGHOUSE LAMP Co. (U.S.P. 1,787,694, 6.1.31. Appl., 8.3.26).—A cathode composed of Mo and about 1% V is claimed.

J. S. G. THOMAS.

**Face plate for electrical contact.** C. A. LAISE, ASSR. to EISLER ELECTRIC CORP. (U.S.P. 1,788,243, 6.1.31. Appl., 25.8.27).—An alloy containing at least 75% W and more V than Si, and if desired Th, is claimed.

J. S. G. THOMAS.

**Electric-discharge devices.** GEN. ELECTRIC CO., LTD., J. W. RYDE, and H. G. JENKINS (B.P. 351,579, 2.4.30 and 1.1.31).—An electric discharge device, particularly an electric discharge lamp, is replenished continuously during operation with a permanent gas, e.g.,  $\text{H}_2$  or oxides of C, by connecting it with a supply of a substance, e.g.,  $\text{C}_{10}\text{H}_8$  or camphor respectively, capable of evolving at a temp. not far above atm., a vapour which under the action of the discharge yields the required gas.

D. F. TWISS.

**Photoelectric apparatus.** P. M. G. TOULON (B.P. 352,388, 27.2.30. Fr., 15.3.29).—A photoelectric cell provided with means for producing and utilising a secondary emission of electrons from the anode is claimed.

J. S. G. THOMAS.

**Electrical precipitator.** R. HEINRICH, ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,790,147, 27.1.31. Appl., 23.7.30).—A device is described for freeing the electrodes of an electrical precipitator from the collected dust by vibrating the whole device (elastically mounted) with a magnetically operated hammer. The dust is removed from the bottom of the device in the usual bin.

E. B. HUGHES.

**Liquid for electric accumulators.** G. SCHMAUS (B.P. 352,768, 6.8.30).—See U.S.P. 1,722,343; B., 1929, 824.

**[Grid structures for] galvanic batteries.** OLDHAM & SON, LTD., and T. G. MAIR (B.P. 353,378, 6.2.31).

**[Construction of] electric incandescence lamps.** FALK, STADELMANN & CO., LTD. From W. BERGER (B.P. 352,716, 30.6.30).

**Electron-discharge apparatus.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 352,617, 9.5.30. Holl., 19.11.29).

**Electric-discharge tubes.** S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 352,945, 10.4.30. Addn. to B.P. 340,049).

**[Starting of] luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., ASSEES. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 353,372, 30.12.30. Ger., 4.1.30).

**Electric gas-discharge rectifiers with thermionic cathodes.** GEN. ELECTRIC CO., LTD., F. CLARK, W. G. FROGLEY, and D. I. WATSON (B.P. 353,203, 23.6.30).

**Indicating changes in gases.**—See I. Carbon.—See II. Drying cellulose etc.—See V. Magnetic materials. Be alloys.—See X.

## XII.—FATS; OILS; WAXES.

**Optical activity of natural fats and oils.** B. SUZUKI, Y. INOUE, and R. HATA (Proc. Imp. Acad. Tokyo, 1931, 7, 222—225).—Various oils, rapidly extracted (cf. A., 1930, 736) from living seeds, showed definite optical activity (e.g.,  $[\alpha]_D +0.5^\circ$  to  $+0.35^\circ$ ) which decreased by racemisation of the glycerides to  $0^\circ$  in 70—200 hr. The fall was smooth in the case of soya-bean oil, but irregular for other seed oils. Similarly linoleo-dilinolenin bromides precipitated from fresh linseed oil at  $-10^\circ$  to  $-15^\circ$  had  $[\alpha]_D -0.9^\circ$  falling to  $0^\circ$  in 5 hr. Oils from seeds killed by  $\text{Et}_2\text{O}$  vapour were inactive. Oil extracted from fresh tissues of living carp and eel had  $[\alpha]_D +0.38^\circ$  and  $+0.15^\circ$  respectively, falling to  $0^\circ$  and  $+0.14^\circ$  (16 hr.). E. LEWKOWITSCH.

**Potentiometric titration in non-aqueous solutions. I. Differential method for determining oil acidity.** B. L. CLARKE, L. A. WOOTEN, and K. G. COMPTON (Ind. Eng. Chem. [Anal.], 1931, 3, 321—323).—A refined method is detailed; a dil. solution of  $\text{LiCl}$  in  $\text{BuOH}$  is used as fat solvent (cf. B. 1930, 248) with a quinhydrone electrode, and the thermionic titrometer (A., 1930, 1545) is adapted so that direct readings of the differential of the titration curves can be taken in systems having a high resistance.  $\text{KOH}$  in  $\text{BuOH}$  is used as reagent. The procedure is suitable for dark oils, asphalts, etc., and yields reproducible results which agree well with those obtained by the A.S.T.M. colorimetric method in cases where the latter is applicable (e.g., transformer oils). E. LEWKOWITSCH.

**Fractional saponification of fatty substances. II. Detection of the commoner adulterants of olive oil.** E. DE'CONNO and L. FINELLI (Annali Chim. Appl., 1931, 21, 203—210).—Partial saponification of olive oil (cf. B., 1930, 466), followed by determinations of the thermal value for the unsaponified residue and of the refractive index of the insol. non-volatile acids of the saponified portion, furnishes a means of detecting adulteration with arachis, colza, or sesame oil.

T. H. POPE.

**Hardening of linseed oil at very high hydrogen pressure.** H. I. WATERMAN and J. A. VAN DIJK (Rec. trav. chim., 1931, 50, 679—680).—Further experiments (cf. B., 1931, 499) show that hardening under high pressure of  $\text{H}_2$  produces fats containing more saturated acids than oils hardened to the same I val. at  $180^\circ$  at ordinary pressure.

J. D. A. JOHNSON.

**Influence of pressure and temperature on the hardening of soya-bean oil.** H. I. WATERMAN, M. J. VAN TUSSENBROEK, and J. A. VAN DIJK (Rec. trav. chim., 1931, 50, 793—795).—The products formed when soya-bean oil is hardened under high pressure and low temp. contain more saturated fatty acids than those produced by Wilbuschewitsch's (cf. B., 1929, 608) or Normann's methods, even when the I vals. are identical.

H. BURTON.

**"Kernel," the seed of the fruit of *Anacardium occidentale*.** L. GOBERT (Ann. Falsif., 1931, 24, 260—268).—A description of the cashew apple and nut with microscopical character of the seed and analyses of the seed and the fat.

E. B. HUGHES.



**Occurrence of arachidic acid in cacao butter.** O. LÜNING and W. DRUDE (Z. Unters. Lebensm., 1931, 61, 491—494).—Arachidic acid does not normally occur in appreciable quantities in cacao butter, which contains no fatty acids higher than stearic acid. Graf's original method of fractional precipitation of the Mg salts (cf. A., 1889, 35) gives positive results unless the Mg salt is dried at 95° till const. in wt. instead of in a vac. desiccator, and the m.p. of the fatty acids should also be checked. A positive result obtained from a questionable milk chocolate (0.97 g. of arachidic acid from 20 g. of fat) indicated adulteration with hardened arachis oil (cf. A., 1929, 294; B., 1929, 785).

J. GRANT.

**Analysis of fatty modifications obtained by action of sulphuric acid.** M. DEGROOTE, B. KEISER, A. F. WIRTEL, and L. T. MONSON (Ind. Eng. Chem. [Anal.], 1931, 3, 243—252).—Cold sulphonation of oleic acid produces principally the *cis*-sulphate,  $\text{Me} \cdot [\text{CH}_2]_7 \cdot \text{CH}(\text{SO}_3\text{H}) \cdot [\text{CH}_2]_8 \cdot \text{CO}_2\text{H}$ ; with ricinoleic acid addition of  $\text{H}_2\text{SO}_4$  at the double linking also occurs to some extent (cf. A., 1895, 82) but no compound with sulphate combined both at the double linking and at the OH group could be found. Use of the Parr peroxide bomb indicated a total combined S content of sulphonated castor oil considerably greater (up to 50%) than that obtained by the HCl-hydrolysis method (hydrolysable sulphate); the discrepancy (non-acidic, non-hydrolysable organically combined S) was not so great with oleic acid products, and both methods gave identical results with Na sulphoricinoleate and sulpholeate. The volumetric (double-titration) method tends to give low results, as boiling with  $\text{H}_2\text{SO}_4$  may convert part of the fatty acid sulphates into a non-hydrolysable derivative. The Hübl-Waller (24 hr.) I val. determination gave concordant results (about half the Wijs val.) and is the most reliable for research work; the Margosches method (3—5 min.) may be conveniently employed in certain cases (e.g., in absence of free  $\text{H}_2\text{SO}_4$ ) for plant control work. Determination of acetyl val., fatty matter,  $\text{NH}_3$ , etc. is discussed. The characteristics of a recovered oil vary with the mode of prep. of the original sulphonated product. The determination of lactones is complicated by the fact that treatment with mineral acid favours the production of ester-acids from hydroxylated acids. A method for the determination of lactones, such as are regenerated after alkaline saponification and re-acidification, is described (28.5—29.5% of stearylactone was found in the product from an oleic acid sulphonation). A stable hydroxystearic acid (m.p. 76°; cf. A., 1925, i, 5) may be prepared only from a pure  $\gamma$ -stearylactone or one containing oleic and/or *cis*-hydroxystearic acid. The action of  $\text{H}_2\text{SO}_4$  on oleic acid at about 100° yields a true sulphonic acid, probably  $\text{C}_{17}\text{H}_{33}(\text{OH})(\text{SO}_3\text{H}) \cdot \text{CO}_2\text{H}$ , non-hydrolysable by dil. acids at the b.p. Free  $\text{H}_2\text{SO}_4$ ,  $\text{SO}_2$ , hydrolysable acid sulphate,  $\text{SO}_3\text{H}$ , and total organically-combined S (Parr bomb) should be determined for such products.  $\text{Et}_2\text{O}$  may be conveniently replaced by  $\text{Pr}^2\text{O}$  in many determinations E. LEWKOWITSCH.

**Determination of glycerol in greases.** M. H. PRAMME (Ind. Eng. Chem. [Anal.], 1931, 3, 232—233).—The glycerol in the aq. layer separated after treatment of the grease with  $\text{H}_2\text{SO}_4$  is oxidised with excess of

$\text{K}_2\text{Cr}_2\text{O}_7$  and  $\text{H}_2\text{SO}_4$ , and the  $\text{CO}_2$  liberated is absorbed and weighed. The method is quick and suitable for normal greases etc. unless they contain  $\text{H}_2\text{O}$ -sol. org. matter (other than glycerol) which cannot be precipitated by inorg. salts. E. LEWKOWITSCH.

**Soap boiling. I. Distribution of glycerin in curd soap and lye.** Y. KAWAKAMI (J. Soc. Chem. Ind. Japan, 1931, 34, 186—188 B).—The distribution of glycerin between curd soap and lye is in agreement with the Freundlich adsorption isotherm. A method for calculating the number of times salting-out may be usefully employed in glycerin recovery is given.

A. A. LEVI.

Marzipan.—See XVII.

See also A., Aug., 1932, Preparation of hydrocarbons etc. by reduction of fats. 935, Determination of solid unsaturated fatty acids.  $\text{Et}_2\text{O}$ -sol. Pb salts of lumbang oil. 975, Fatty acids of egg-yolk lecithin. Wax of wool louse. 987, Influence of soaps on germicidal properties of certain Hg compounds. 988, Vitamins in liver- and butter-fat. 989, Fat-sol. vitamins.

## PATENTS.

**Method of making linseed oil.** R. H. ADAMS and B. H. THURMAN, Assrs. to GOLD DUST CORP. (U.S.P. 1,790,494, 27.1.31. Appl., 3.7.25).—The seed is cleaned from dockage to a max. of 1—1.5% and cracked, and the oil is (expeller) expressed below 43°, and cleared by treatment with fuller's earth. The product can be used for varnishes etc. without further treatment.

E. LEWKOWITSCH.

**Bleaching of [linseed] oil.** B. H. THURMAN, Assr. to GOLD DUST CORP. (U.S.P. 1,790,514, 27.1.31., Appl., 18.3.26).—A good quality cold-pressed (cf. U.S.P. 1,790,494, preceding) or refined linseed oil, free from mucilage, is heated at 260—282°/1 in. until sufficiently bleached, and is chilled rapidly to 93°.

E. LEWKOWITSCH.

**[Continuous dry] rendering process.** S. HILLER (U.S.P. 1,789,751, 20.1.31. Appl., 26.5.25).—Animal waste etc. is continuously passed through a digester where it is sterilised and disintegrated under steam pressure (conveniently developed from the material itself), through a vac. dryer until the moisture content is reduced to 20%, and is then drained and pressed.

E. LEWKOWITSCH.

**Bleaching of soap.** C. F. SCHUMAKER, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,788,848, 13.1.31. Appl., 26.6.28).—Stable bleaching is effected by mixing the soap (chips) with  $\text{NaOCl}$  (2%) and  $\text{CH}_2\text{O}$  (1%) before milling.

E. LEWKOWITSCH.

Preserving soaps.—See XIV.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Diagnosis of oil paint failures.** B. SCHEIFELE (Farben-Ztg., 1931, 36, 1767—1769).—The causes of oil paint failure are classified under: unsuitable quality of paint, faulty application, insufficient prep. of the surface to be painted, incorrect system of undercoats, and exposure to extreme conditions. Methods of diagnosis of failures are described. The details of

investigation of paint failure on the metal portions of an aq.  $\text{NH}_3$  tank-car illustrate the foregoing. S. S. WOOLF.

**Drying process of [artificial] iron oxide-boiled linseed oil paints.** H. SALVATERRA and F. ZEPPEL-ZAUER (Farben-Ztg., 1931, 36, 1771—1773, 1812—1814, 1851—1853).—Dried films of paints containing artificial Fe oxides retain a stickiness for considerable periods. The O-absorption and drying time of films of Pb- and Co-boiled oils were found to be but little affected by the incorporation of synthetic Fe oxide "B 20," which is relatively inert, no Fe soaps being formed by combination with the acid decomp. products of the oil. The stickiness of such films is due to a tendency of the artificial pigment to increase the extent of acid formation over that of films of the oil alone, natural Fe oxide pigments not producing this increase. The addition of 10% of  $\text{Pb}_3\text{O}_4$  cures this defect. A new apparatus for measuring the stickiness of paint films is described.

S. S. WOOLF.

**Hardness of films.** W. TOELDT (Farben-Ztg., 1931, 36, 1769—1771, 1810—1811).—The significance of "hardening through" of a film is discussed and existing methods of measuring film hardness are criticised. A new method is described involving the measurement in  $\mu$  of the extent of penetration of a loaded gramophone needle (selected microscopically). Complete penetration is indicated by the needle closing an electric circuit with the metal base and thus lighting a small lamp; the operation is then repeated with a smaller load until progressive penetration for 5 min. is recorded. Hardness at any given load is expressed as the percentage thickness of film penetrated after 1 min. Typical results are tabulated, graphed, and discussed.

S. S. WOOLF.

**Loss of weight of paint films.** H. WOLFF and G. ZEIDLER (Farben-Ztg., 1931, 36, 1850—1851).—The loss of wt. in 13½ months of a paint film comprising priming ( $\text{Pb}_3\text{O}_4$ ) and 6 subsequent thin coats exceeded the wt. of the first coat, the loss being shown by analysis to be loss of vehicle, that of pigment being relatively small. No obvious disintegration of the final coat had occurred and the thickness of the composite film was not noticeably diminished. Weathering has thus taken place by formation of gaseous decomp. products throughout the system. The bearing of these results on previous work on the relation of "chalking" to loss of wt. is discussed.

S. S. WOOLF.

**Identification of old paint and varnish films.** A. NAUROY (Peint., Fig., Ver., 1931, 8, 1538—1542).—The effects of ageing and weathering influences on the various constituents of paint and varnish films are discussed. Information as to the nature of old films can be obtained by visual and microscopical inspection, rubbing, gentle heating, determination of hardness and elasticity by the lead-pencil method, action of  $\text{EtOH}$ ,  $\text{CCl}_4$ ,  $\text{PhMe}$ ,  $\text{BuOAc}$ , etc.

S. S. WOOLF.

**Measurement of effect of carbon black on fluidity of printing ink.** E. P. W. KEARSLEY and G. L. ROBERTS (Ind. Eng. Chem. [Anal.], 1931, 3, 310—311).—The sample of C black to be tested (0.25 g.), ground into paste with lithographic varnish (2.5 g.) by standard procedure, is drawn into a 10-c.c. pipette which is

inserted in the top of a box maintained at standard temp. ( $45^\circ$ ), and after 5 min. preheating, 10 drops of the sample are allowed to drop on a horizontal glass plate inside the box. After 1 min. the plate is swivelled down to an angle of  $30^\circ$  with the horizontal and the extent of flow of the sample down the plate in 7 min. is measured. Standard samples may be run alongside and the results may readily be converted into poises. S. S. WOOLF.

**Camphor white oil as a paint and varnish thinner.** S. HIROSE (Proc. World Eng. Congr., 1929, 31, 489—496).—Two samples of camphor white oil (a low-boiling distillation fraction of crude camphor oil, mainly comprising pinene and camphene) were found to be comparable with 3 representative samples of American turpentine in solvent power, rate of volatilisation, influence on drying times of varnishes, etc. Camphor white oil shows advantages in higher flash point ( $49.5^\circ$ ) and in cost.

S. S. WOOLF.

**Testing of coloured pigments. I. Staining power.** H. WAGNER and M. ZIFFEL (Farben-Ztg., 1931, 36, 1889—1892).—Staining power is expressed as the difference in white content of  $\text{ZnO}$  and a 1 : 20 mixture of coloured pigment and  $\text{ZnO}$  as measured by the Ostwald half-shadow photometer. The usual method of determining staining power is outlined and the accuracy of the determination is discussed from the following aspects: type and concentration of oil, degree of mixing, weighing procedure, method of painting out, test surface. From these considerations a detailed standard method is developed.

S. S. WOOLF.

**Analysis of chrome-green and similarly composed pigments.** C. P. A. KAPPELMEIER (Rec. trav. chim., 1931, 50, 711—718).—The method, which is suitable for chrome-green and many other pigments containing Prussian-blue, is based on the decomp. of Fe complex cyanides by yellow  $\text{HgO}$  in boiling  $\text{H}_2\text{O}$ . This treatment, followed by the addition of  $\text{Na}_2\text{O}_2$ , permits the determination of Fe as  $\text{Fe}_2\text{O}_3$ , Cr as  $\text{Na}_2\text{CrO}_4$ , and Pb as  $\text{PbSO}_4$  by the usual methods.  $\text{SO}_4^{--}$  is determined as  $\text{BaSO}_4$  in a separate portion.

E. S. HEDGES.

**New methods of surface protection [by poly-vinyl esters].** A. EIBNER (Farben-Ztg., 1931, 36, 1849—1850, 1892—1894).—The inherent disadvantages of combinations of drying oils with natural and synthetic resins are discussed. The desirable properties of poly-vinyl resins (derived from chlorinated and non-chlorinated vinyl acyl esters), e.g., freedom from sensitiveness to light, hardness, elasticity, high pigment-binding capacity, etc., are put forward.

S. S. WOOLF.

**Varnish from xylose and aniline.** J. P. MONTGOMERY (Ind. Eng. Chem., 1931, 23, 761—762).—Equimol. proportions of xylose (obtained from cottonseed hulls) and  $\text{NH}_2\text{Ph}$  are heated for 10 min. at  $75$ – $80^\circ$ , and the product is dissolved in  $\text{COMe}_2$ . The varnish dries in ½ hr., giving, according to concentration of solution, a dark oak- to cherry-coloured film, resistant to  $\text{H}_2\text{O}$ , alkalis, and acids.

S. S. WOOLF.

**Viscosity increase and gelation in phenolic resin varnish cooking.** V. H. TURKINGTON, R. C. SHUEY, and W. H. BUTLER (Ind. Eng. Chem., 1931, 23, 791—797).—The time of gelation of tung oil with various

additions of Bakelite resin XR 254, ester gum, rosin, etc. was determined in a modified Brown-Worstell test which is fully detailed. The rate of increase in viscosity of such mixtures was also determined under varnish-cooking conditions, viscosity being measured by a De Vilbiss viscosimeter inserted periodically in the kettle. Resin XR 254 accelerated the gelation of tung oil in the first series of experiments, but in the viscosity measurements this is shown to be a nett acceleration arising from a decrease in "thickening time" and a slight restraining effect during subsequent gelation (all the viscosity-time curves showing such a division into at least two sections). S. S. WOOLF.

**Detection and identification of synthetic resins.** T. F. BRADLEY (Ind. Eng. Chem. [Anal.], 1931, 3, 304—309).—The types of synthetic resins most commonly found in protective coatings are summarised, constituents, general characteristics, and usual occurrence being detailed. A scheme for isolation of the synthetic resin component involves removal of pigment by repeated filtrations after addition of kieselsguhr and  $\text{CO}_2$ , precipitation of cellulose esters by dilution with  $\text{C}_6\text{H}_6$ , PhMe, etc., steam-distillation, and extraction with light petroleum. Densities and refractive indices of resins are tabulated, the figures for the synthetic products being in general higher than those for the natural resins. These are considered as characteristic criteria which, in conjunction with other indicated physical and chemical tests, form a basis for the identification of synthetic resins. Qual. tests for halogens, S, N, phthalates, phenols, vinyl esters, etc. are described. S. S. WOOLF.

**Phenol resin as moulding composition.** T. HIRAOKA (Proc. World Eng. Congr., 1929, 31, 481—488).—The solubility in NaOH solution of the initial condensation products of PhOH and  $\text{CH}_2\text{O}$  in the presence of alkaline catalysts was examined. Powdered resins, softening at 80—90° and suitable for direct conversion into moulding powder by incorporation with wood flour etc. on the hot roller, were obtained by adding excess of  $\text{H}_2\text{O}$  to NaOH solutions of the initial condensation products or by neutralisation of such solutions with acid, e.g., HCl. S. S. WOOLF.

**Macromolecules and micelles in organic polymers.** S. E. SHEPPARD (Ind. Eng. Chem., 1931, 23, 781—784).—The methods used for examining the structure of the high-mol. aggregates found in rubber hydrocarbons, cellulose derivatives, and synthetic resins, and the reaction mechanism of polymerisation are discussed. The formation of films of paint, varnish, and lacquer depends greatly on the group behaviour of such macromols. Evidence for the individual existence of the latter is summarised. S. S. WOOLF.

**Resin of *Ipomœa*. Balsams.**—See XX.

See also A., Aug., 937, **Intermediate stage of formation of bakelite from PhOH and  $\text{CH}_2\text{O}$ .** 960, **Separation of aleuritic and shellolic acids [from shellac]. Resin of *Garcinia Mangostana*.** Elemic acid from Manila elemi resin. 961, **New resin from Manila elemi resin.** 967, **Bilirubinoid pigments.**

## PATENTS.

**Duplicating [inks].** L. SILBERSTROM (U.S.P. 1,789,783, 20.1.31. Appl., 5.6.26).—The use of hexamethylenetetramine or its salts in duplicating inks, for processes involving neutral or acid plates, is claimed. S. S. WOOLF.

**Writing fluid.** R. A. BELMONT (B.P. 351,611, 9.4.30).—A solution formed of  $\text{H}_2\text{O}$ -sol. colour ingredients and an aq. carrier solution comprising as penetrating agent a deliquescent salt of an alkaline-earth metal, an alkali hydroxide or sulphide, etc. is claimed as a quick-drying, non-smearing, and highly absorbent writing fluid. S. S. WOOLF.

**Manufacture of printing colours.** E. FRENKEL (B.P. 351,824, 5.9.30. Ger., 10.9.29).—Printer's ink which dries very rapidly is prepared with a blown linseed oil having Ac val. not less than 100. A. J. HALL.

**Treatment of rosin.** W. B. LOGAN (B.P. 351,583, 2.4.30).—A bleached grade of rosin is heated for 10 min.—8 hr. below decomp. temp., e.g., at 260—325°, the content of potential colour body (i.e., that producing a yellow colour on dissolution in alkali and subsequent treatment with Al salts) and/or the tendency of the rosin to crystallise being thereby reduced considerably. The optical rotation is increased to at least +20°. S. S. WOOLF.

**Treatment of low-grade resins.** M. T. LACRUE (B.P. 351,065, 22.3.30. Sp., 22.3.29).—A low-grade resin containing about 15% of turpentine, 50% of rosin, 25% of  $\text{H}_2\text{O}$ , and 10% of impurities is heated with 15—75 wt.-% of turpentine, and after decantation and filtration, the solution is steam-distilled, all the turpentine being recovered, and light-coloured rosins obtained. S. S. WOOLF.

**Synthetic resins and methods of manufacturing them.** BRIT. THOMSON-HOUSTON CO., LTD. Assees. of R. H. KIENLE and W. J. SCHEIBER (B.P. 352,140, 30.4.30. U.S., 30.4.29).—An aromatic amine, e.g.,  $\text{NH}_2\text{Ph}$ , and an aldehyde, e.g.,  $\text{CH}_2\text{O}$ , are caused to react in acid, e.g., HCl, solution at temps. rising to 40°, a fibrous pulp with or without fibrous and/or non-fibrous filling materials, e.g., mica, is added to the resulting clear acid liquor, and the mixture is stirred for 1 hr. until the fibrous pulp is saturated. After dilution, alkali, e.g., CaO, is added to neutralise the acid and ppt. the resin, which is then flocculated by raising the temp. to 60°, and fixed on and/or in the fibres by continued stirring. The resulting product is moulded at about 150° under pressure of >1000 lb./sq. in. The flocculated product may be diluted to about 1% consistency and formed into sheets, which are dried, calendered, and heated under pressure. S. S. WOOLF.

**Manufacture of artificial masses and articles therefrom.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,508, 24.2.30).—Halides or nitriles of monomeric unsaturated aliphatic carboxylic acids, preferably of the acrylic acid series, are polymerised in known manner and heated to 35—150° under pressure with agents containing a reactive H atom, e.g.,  $\text{H}_2\text{O}$ , inorg. bases, alcohols,  $\text{H}_2\text{S}$ ,  $\text{NH}_3$ , amines, etc., in the presence, if desired, of inert diluents, e.g., aliphatic esters (when

working with halides) and  $\text{H}_2\text{SO}_4$  or  $\text{H}_3\text{PO}_4$  (when working with nitriles). Org. film-forming substances, *e.g.*, nitrocellulose, may be incorporated. S. S. WOOLF.

**Artificial [resinous] masses.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 352,042, 3.3.30).—Film-forming substances (*e.g.*, natural or synthetic resins, cellulose derivatives) are incorporated with the viscous or solid products of the polymerisation of alkylene oxides (*cf.* B.P. 346,550; B., 1931, 666) with or without addition of solvents *etc.* E. LEWKOWITSCH.

**Resinous compositions particularly for use in coating compositions.** H. A. GARDNER (B.P. 351,637, 22.4.30. U.S., 12.2.30).—Chlorinated diphenyls are heated with natural and/or synthetic resins, *e.g.*, rosin, ester gum, toluenesulphonamide- $\text{CH}_2\text{O}$ , the resinous composition being used as such or incorporated with nitrocellulose or drying oil components as lacquers. S. S. WOOLF.

**Resinous materials.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 351,945, 16.12.29).—Alcohols containing more than three OH groups (excluding carbohydrates), *e.g.*, pentaerythritol, are esterified with higher cyclic or open-chain aliphatic acids, *e.g.*, fatty acids (or glycerides), naphthenic acids, and simultaneously with resin acids. The products may be blown with air or vulcanised. E. LEWKOWITSCH.

**Resin or resinous product and process of making same.** C. ELLIS (U.S.P. 1,787,618, 6.1.31. Appl., 2.7.21. Renewed 1.3.30).— $\text{S}_2\text{Cl}_2$ , a phenolic substance, and  $\text{CH}_2\text{O}$  or a derivative, *e.g.*, hexamethylenetetramine, are heated together, giving a fusible resinous material containing 20–60% of combined S. S. S. WOOLF.

**Preparation of condensation products from urea and formaldehyde.** G. C. A. MANESSE and J. SECHAYE (B.P. 352,288, 6.8.30. Fr., 7.8.29).—Urea and  $\text{CH}_2\text{O}$  are condensed at  $80^\circ$ , in presence of glucose or lactose and a metallic oxide, preferably  $\text{ZnO}$ . The plastic products are known as "Veryx" and "Cristalyx." S. S. WOOLF.

**Condensation product of urea and formaldehyde.** O. A. CHERRY, Assr. to ECONOMY FUSE & MANUFACTURING CO. (U.S.P. 1,790,461, 27.1.31. Appl., 3.6.27).—Urea (1 pt.) is condensed in small quantities at a time with  $\text{CH}_2\text{O}$  (1–5 pts.) in solution of  $p_{\text{H}}$  about 3, and the  $\text{H}_2\text{O}$ -sol. condensation product is hardened by heat, after elimination of  $\text{H}_2\text{O}$ . S. S. WOOLF.

**Production of vinyl ester resins.** CANADIAN ELECTRO PRODUCTS CO., LTD., Asses. of F. W. SKIRROW, G. O. MORRISON, and K. G. BLAICKIE (B.P. 351,082, 25.3.30. U.S., 25.3.29).—A vinyl ester polymeride or the reaction product of an aliphatic vinyl ester, *e.g.*, the acetate, with a saturated aliphatic aldehyde, *e.g.*,  $\text{MeCHO}$ , is heated with mineral acid, *e.g.*,  $\text{HCl}$ , and  $\text{H}_2\text{O}$ , and the hydrolysed product is further condensed with an aldehyde, *e.g.*,  $\text{CH}_2\text{O}$ ,  $\text{MeCHO}$ . S. S. WOOLF.

**Production of artificial resin from polyhydric phenols and aldehydes.** INTERNAT. GEN. ELECTRIC CO., INC., Asses. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 350,934, 11.3.30. Ger., 11.3.29).—Polyhydric phenols, *e.g.*, resorcinol, and carbonyl compounds, *e.g.*,  $\text{CH}_2\text{O}$ , are caused to interact in stages, part only of the

carbonyl compound being added initially to form a "semi-condensate," an acid resin with free phenolic OH groups, the reaction being subsequently completed by the addition of the rest of the carbonyl compound. Fillers and colouring matter and natural and artificial resins up to 250% of the "semi-condensate" may be added. S. S. WOOLF.

**Manufacture of synthetic resins and moulding powders obtained therefrom.** BRIT. CYANIDES CO., and E. C. ROSSITER (B.P. 351,093—4, 25.3.30).—(A) A salt of hexamethylenetetramine, *e.g.*, the thiocyanate, with or without urea or thiourea, or (B) free thiourea, is added to synthetic resins or moulding powders made substantially from  $\text{CH}_2\text{O}$  and urea, as accelerators of "curing." S. S. WOOLF.

**Thermoplastic materials.** BRIT. THOMSON-HOUSTON CO., LTD., Asses. of B. W. NORDLANDER (B.P. 351,188, 14.5.30. U.S., 14.5.29).— $\text{SeS}_2$ , prepared by direct union, is fused with a filler, *e.g.*,  $\text{Fe}_2\text{O}_3$ , asbestine, wood flour, *etc.*, at about  $125^\circ$ , the mass is cooled and pressed, and finally "cured" for about  $\frac{1}{2}$  hr. at  $80$ – $90^\circ$ , giving a hard, dense product. S. S. WOOLF.

**Manufacture of mouldable materials.** R. E. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 351,390, 20.1.30).—A cellulose ester, *e.g.*, the acetate, is stirred vigorously with plasticisers, *e.g.*, camphor, Et phthalate, in suspension in  $\text{H}_2\text{O}$  (>twice the wt. of cellulose derivative), fillers and colouring matter are added if desired, and the suspended mixture is mechanically separated, giving homogeneous easily-moulded mixtures. The cellulose derivative may be precipitated from solution in the presence of fillers, to ensure uniform distribution. S. S. WOOLF.

**Shellac composition.** A. P. THURSTON. From CALCO CHEM. CO., INC. (B.P. 352,720, 2.7.30).—See U.S.P. 1,781,711; B., 1931, 643.

**Coating compositions.**—See V. Emulsion.—See IX.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Natural and synthetic rubber.** VII. Fractional precipitation of natural rubber. T. MIDGLEY, JUN., A. L. HENNE, and M. W. RENOLL (J. Amer. Chem. Soc., 1931, 53, 2733–2737).—A method of separation of N-free rubber hydrocarbon (resembling the " $\alpha$ -rubber" of Pummerer), based on the progressive precipitation of natural rubber from a mixture with EtOH and  $\text{C}_6\text{H}_6$  by slow cooling, is given. Temp.-concentration diagrams of the systems natural rubber-EtOH- $\text{C}_6\text{H}_6$ , pure rubber-EtOH- $\text{C}_6\text{H}_6$ , and synthetic rubber-EtOH- $\text{C}_6\text{H}_6$  are also given. H. BURTON.

**Permeability of rubber to air.** II. Effect of stretch, thickness, milling, compounding ingredients, kind of crude rubber, and temperature of vulcanisation. V. N. MORRIS (Ind. Eng. Chem., 1931, 23, 837–843; *cf.* B., 1930, 112).—The permeability of rubber membranes is approx. inversely proportional to their thickness; the increase in permeability of stretched rubber is mainly attributable to the accompanying decrease in thickness. The degree of milling, variation in the temp. of vulcanisation, and alteration in the type of plantation rubber used have little

influence, but caucho ball rubber gave a mixture of somewhat lower permeability. None of the various softeners or other org. materials was found to have appreciable beneficial effect. Permeability appears to be roughly proportional to the vol. of rubber hydrocarbon present in the mixture, but the nature of the principal compounding ingredients has a slight influence, clay, channel black, and blanc fixe being the most beneficial.

D. F. TWISS.

**Value of softeners in [rubber tyre] tread stock.** M. J. DE FRANCE and W. J. KRANTZ (Ind. Eng. Chem., 1931, 23, 824—827).—If max. abrasion-resistance is to be economically obtained, it is desirable to use the min. proportion of pine tar or mineral oil commensurate with good factory processing. The proportion of stearic acid in a tyre-tread mixing accelerated with mercapto-benzthiazole should be maintained at about 4% on the rubber content. The results of laboratory abrasion tests with 30% slippage do not coincide exactly with road tests, but give indications in the same direction.

D. F. TWISS.

**Effect of scrap-drying temperature on quality of reclaimed rubber.** H. F. PALMER, G. W. MILLER, and J. E. BROTHERS (Ind. Eng. Chem., 1931, 23, 821—827).—Experiment shows that a max. ultimate drying temp. of 121.1° to a min. moisture content of 3% may be used for all standard types of "devulcanised" rubber scrap. It is recommended, however, that a lower ultimate temp. be used and that more rapid drying be promoted by the use of higher temps. in the wet end of the dryer. Increase in drying temp. does not affect the proportion of acetone-sol. matter, but raises the  $\text{CHCl}_3$  extract and increases the softness of the material. Vulcanised rubber mixtures containing 35% of reclaimed rubber do not exhibit undesirably abnormal stress-strain characteristics either before or after ageing.

D. F. TWISS.

**$\gamma$ -Methyl- $\alpha$ -pentadiene.**—See III. Rubber for pickling baths.—See X. Macromols. and micelles.—See XIII.

## PATENTS.

**Preservation of oxidisable hydrocarbons [including rubber, also soaps].** A. M. CLIFFORD (B.P. 351,645, 29.4.30).—Naphthylformamidines, of the general formula  $\text{R}\cdot\text{N}\cdot\text{CH}\cdot\text{NHR}'$ , where R and R' represent  $\text{C}_{10}\text{H}_7$  groups, are efficient preservatives for rubber, transformer oils, soaps, etc. Di- $\alpha$ - or di- $\beta$ -naphthylformamidine can be obtained by heating the naphthylamine and formic acid in  $\text{NH}_2\text{Ph}$  for 6 hr. under conditions permitting the escape of  $\text{H}_2\text{O}$  vapour formed in the reaction.

D. F. TWISS.

**Preservation of rubber.** GOODYEAR TIRE & RUBBER Co. (B.P. 351,844, 26.9.30. U.S., 6.11.29).—Aminohydroxy diphenyl, prepared by successive nitration and reduction of *p*-hydroxydiphenyl (B.P. 302,147; B., 1929, 990), is a more powerful antioxidant than the parent compound; it is also non-poisonous and inodorous.

D. F. TWISS.

**Retardation of deterioration of rubber.** W. S. CALCOTT and W. A. DOUGLASS, ASSRS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,789,696, 20.1.31. Appl., 17.9.29).—The reaction product of  $\text{C}_2\text{H}_2$  and an  $\text{NH}_2$  compound, e.g.,  $\text{NH}_2\text{Ph}$ , produced preferably with the

aid of a catalyst such as  $\text{Cu}_2\text{Cl}_2$ , is used as an antioxidant for rubber. The product from  $\text{C}_2\text{H}_2$  (2 mols.) and  $\text{NH}_2\text{Ph}$  (1 mol.) contains 9.82% N and has m.p. 92.4—93.7°.

D. F. TWISS.

**Manufacture of rubber articles.** PIRELLI, LTD. From Soc. Ital. PIRELLI (B.P. 351,012, 17.3.30).—Articles are manufactured from vulcanised substantially uncoagulated aq. dispersions of rubber and the like by local heating in the presence of a destabilising agent (cf. B.P. 284,608; B., 1929, 612).

D. F. TWISS.

**Treatment of rubber. [Vulcanisation accelerators.]** DEUTS. HYDRIERWERKE A.-G. (B.P. 351,356, 3.10.30. Ger., 22.10.29).—Alcohols of high mol. wt., particularly fatty or wax alcohols or cyclic alcohols, are used as softeners, vulcanisation accelerators, and anti-agers for rubber; e.g., incorporation of 5—10% of dodecyl or octadecyl alcohol gives an excellent finished product.

D. F. TWISS.

**[Accelerator for] manufacture of rubber.** GOOD-YEAR TIRE & RUBBER Co. (B.P. 351,832, 18.9.30. U.S., 5.10.29).—Vulcanisation is accelerated by the condensation product of cyclohexylamine with an open-chain aldehyde such as aldol,  $\text{CH}_2\text{O}$ , crotonaldehyde, or heptaldehyde; the products have high tensile strength and good resistance to ageing.

D. F. TWISS.

**Vulcanisation of rubber articles [inner tubes etc.].** M. A. MARQUETTE, ASSR. to FISK RUBBER Co. (U.S.P. 1,790,011, 27.1.31. Appl., 22.7.26).—In the vulcanisation of articles on mandrels with the outer surface of the rubber exposed to the steam, air is introduced together with the steam into the vulcanising chamber; the resulting excess pressure increases the moulding effect and reduces the risk of formation of air pockets. Uniformity of temp. is maintained by subsequently opening a vent to permit slight discharge of the gaseous mixture, the supply of steam being maintained so as to ensure a const. vulcanising temp.

D. F. TWISS.

**Rubber process and product.** E. E. AYRES, JUN., ASSR. to B.A.S. Co. (U.S.P. 1,789,062, 13.1.31. Appl., 21.2.23).—Rubber latex is mixed with oils, fats, waxes, resins, or liquid hydrocarbons either in aq. dispersion or under such conditions that they readily become emulsified; the emulsified mixture of added ingredient and rubber is then separated from the water by evaporation or coagulation.

D. F. TWISS.

## XV.—LEATHER; GLUE.

**Tanning with nepheline.** N. V. BELOV (Bull. Acad. Sci. U.S.S.R., 1931, 315—318).—Kay's experiments with silicic acid as a tanning material (Le Cuir, 1919, 209, 257, 314) yielded unsatisfactory products. Good results are, however, obtainable with a solution of nepheline in acid, diluted and stabilised with the salts commonly used in tanning, such as  $\text{NaCl}$ , from 5 to 6% of nepheline (on the dry skin) being absorbed.

T. H. POPE.

**Influence of tanning substances on certain metals.** A. GANSSER (Boll. Off. Staz. Sperim. Ind. Pelli, 1931, 9, 2—5, 90).—The use of metal wire in baling salted hides is inadvisable, Fe being especially

harmful. The resistance to corrosion of various metals used in tanning is considered. Monel metal resists superheated steam and is particularly useful, being more easily worked than Ni-Cr steels of the V2A and V4A types. Monel metal and V2A steel resist cellulose sulphite liquor satisfactorily. Al is unsuitable, as it is attacked by tanning extracts producing yellow lakes, and by various materials commonly used in tanning. The use of standardised dishes of monel metal and Ni-Cr steel V2A, in place of Ag basins for evaporation in the analysis of tanning substances, is being tried.

T. H. POPE.

See also A., Aug., 929, Cell for electrodialysis [of gelatin]. 971. Amorphous and cryst. gelatin.

#### PATENTS.

**Tannage of sole leather.** C. KANNEL (U.S.P. 1,789,629, 20.1.31. Appl., 4.4.28).—The limed hides are treated first with a mixture of  $K_2CrO_4$ ,  $H_3BO_3$ , glycerin,  $H_2O$ , and  $H_2SO_4$ , formic or butyric acid for 16–24 hr. and subsequently drummed for 24 hr. in a tanning solution of  $d$  1.080–1.092, in a rotating drum mounted on a longitudinally perforated shaft, one end of which is continuously evacuated during the tannage.

D. WOODROFFE.

**Culture of micro-organisms for unhairing [hides].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,600, 4.4.30).—Proteolytic bacteria, e.g., *B. vulgatus*, *B. megatherium*, are cultivated in nutrient media containing degraded keratin, e.g., meat extract, peptone, agar, 1–2% keratose, and 0.1%  $K_2HPO_4$ , the bacterial films isolated, killed with  $COMe_2$ , dried, and powdered.

D. WOODROFFE.

**Imparting lustre to lambskins and sheepskins.** M. LASKIN (U.S.P. 1,788,026, 6.1.31. Appl., 19.8.29).—The dyed skins are immersed in a 1% emulsion of sulphonated neatsfoot or other oil, centrifuged, dried, milled in sawdust mixed with a small amount of oil emulsion, combed, beaten, and ironed hot after spraying the wool with white paraffin oil.

D. WOODROFFE.

**Leather dye [for shoes].** L. A. WATT. ASSR. to MONSANTO CHEM. WORKS (U.S.P. 1,789,375, 20.1.31. Appl., 7.5.27).—A mixture of  $o$ - $C_6H_4Cl_2$ , spirit-sol. nigrosine, oleic acid, and denatured  $EtOH$ .

D. WOODROFFE.

**Vegetable glue.** E. H. HARVEY, ASSR. to PERKINS GLUE Co. (U.S.P. 1,790,346, 27.1.31. Appl., 19.9.23).—Cassava starch (1 pt.) of sp. viscosity < 60,000,  $H_2O$  (2–3 pts.),  $NaOH$  (3%), and  $FeSO_4$  (0.2%) are heated together at 77–5°.

D. WOODROFFE.

**Adhesive.** L. BRADSHAW, ASSR. to CASEIN MANUFG. Co. (U.S.P. 1,787,611, 6.1.31. Appl., 8.6.27).—A mixture of an alkali-sol. protein, a commercial oil-seed cake, "starch-viscose" (made by treating starch with  $CS_2$  and  $NaOH$ ),  $Ca(OH)_2$ , and  $H_2O$  alone or with one or more of the following:  $Na_2B_4O_7$ ,  $NaF$ ,  $Na_2CO_3$ ,  $Na_3PO_4$ ,  $Na_2SO_3$ .

D. WOODROFFE.

**Production of articles [buttons etc.] from water-insoluble albuminous substances of casein-like character.** J. H. SMIDT (B.P. 352,100, 9.4.30. Ger., 2.8.29).—Articles cut or stamped from sheets or rods of unhardened casein, preferably of animal origin, are

partly hardened by treatment with  $CH_2O$  for, e.g., 1 week, dried superficially in the air, softened at about 100°, and pressed hot to their final form. A uniform  $H_2O$  content is maintained at each stage of the process.

L. A. COLES.

Colloidal solutions.—See XIX.

#### XVI.—AGRICULTURE.

**Mechanical analysis of soils by the pipette method.** M. KÖHN (Z. Pflanz. Düng., 1931, 21A, 211–222).—Criticisms of the author's method by Kötting and Heuser (B., 1931, 453) are unjustifiable. In viscous liquids the hydrodynamic behaviour of an ordinary vertical pipette and a horizontal pipette with a lateral jet is similar. In  $H_2O$  and liquids of low viscosity all pipettes induce a simple streaming motion, whereas in viscous liquids a swirling or turbulent streaming occurs. Glycerin is a very unsuitable medium for pipette analysis owing to the high temp. coeff. of viscosity and the wide variations in viscosity brought about by small additions of  $H_2O$ .

A. G. POLLARD.

**Use of sodium hypobromite for the oxidation of organic matter in the mechanical analysis of soils.** E. TROELL (J. Agric. Sci., 1931, 21, 476–483).—A modified method of analysis is based on the substitution of  $H_2O_2$  by  $NaOBr$  in the preliminary oxidation process, excess of the reagent being removed by the addition of aq.  $NH_3$ . Large amounts of org. matter and  $MnO_2$  in soils may thus be decomposed in the cold and the decomp. of clay minimised. The customary preliminary acid treatment is unnecessary, no further addition of deflocculant is needed, the shaking period is reduced, and the separation of the coarser particles is more precise. In soils having a high ratio of org. : inorg. colloids, oxidation is necessary for complete dispersion.

A. G. POLLARD.

**Soil sampling tube.** H. J. HARPER (Soil Sci., 1931, 32, 65–69).—The sampling tube has two opposite longitudinal ribs. By rotation of the tube after insertion, the hole is enlarged to permit easy withdrawal.

A. G. POLLARD.

**Soil reaction. VII. Electrodialysis apparatus for the determination of replaceable bases in soils.** J. K. BASU (J. Agric. Sci., 1931, 21, 484–492).—A battery of two-compartment electrodialysis cells is described. A modified technique permits the elimination of  $H_2O$ -sol. cations from the analytical values.

A. G. POLLARD.

**Effect of dilution on the  $p_H$  of soils treated with various cations.** A. T. PERKINS and H. H. KING (Soil Sci., 1931, 32, 1–8).—In determinations of the  $p_H$  of soil suspensions by the quinhydrone electrode, values obtained increased with the ratio  $H_2O$  : soil used. The dilution effect was not materially influenced by treatment of the soil with commonly occurring cations.

A. G. POLLARD.

**Pipe-line currents and soil resistivity as indicators of local corrosive soil areas.** E. R. SHEPARD (Bur. Stand. J. Res., 1931, 6, 683–708).—Corrosion is most severe at points of abrupt discharge and abnormal resistivity. Soil resistivities of about 500 ohms per cm. usually indicate severe corrosion; above 1000 ohms

per cm. there is little connexion between resistivity and corrosion.

C. W. GIBBY.

**Effects of various sources of organic matter on the properties of soils as determined by physical measurements and plant growth.** H. B. SPRAGUE and J. F. MARRERO (Soil Sci., 1931, 32, 35—49).—The effect on the physical properties of various types of soils of well rotted manure, spent mushroom soil, raw and cultivated peat, and peat moss are recorded. A sandy soil and a loam were improved by all the materials in the order named. On a clay-loam the more fibrous materials were superior. Peat produced too open a structure. Rates of decomp. of the org. matter were in the order manure > peat moss > raw peat > mushroom soil > cultivated peat.

A. G. POLLARD.

**Carbon-organic matter factor in forest soil humus.** H. A. LUNT (Soil Sci., 1931, 32, 27—33).—The org. matter of forest soils and peat contains <58% C. Numerous analyses show that the customary factor (1.724) for calculating amounts of org. matter from C determinations should be increased to 1.89 for freshly fallen leaves, 1.85 for the decomp. or "duff" layers and 1.80 for structureless humus in forest soils.

A. G. POLLARD.

**Electrokinetic determination of colloids [in soils].** A. REIFENBERG (Z. Pflanz. Düng., 1931, 21A, 138—160).—A modification of Michaelis' cataphoresis apparatus is utilised to determine the colloidal content of soil suspensions. Results agree closely with those of sedimentation methods. The method is rapid and the disturbing influence of perikinetic coagulation is avoided. Applications of the method for determining adsorbed cations of the soil colloids, the fineness of division of ground rock phosphate, etc. are discussed.

A. G. POLLARD.

**Lime studies. Relationships between various methods for determining the lime and acid condition of soils and a method for determining the base required to neutralise exchange acidity.** S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1931, 21A, 160—187; cf. B., 1931, 556).—The rapid electrometric titration of acid soils (1 min. intervals) requires a smaller proportion of base than for the electrometric neutralisation after 24 hr. The difference is ascribed to the presence of bases not easily adsorbed by the soil. Only insignificant amounts of base are adsorbed after 24 hr. Rapid titration of soils to  $p_H$  5.5 (except in the case of very highly buffered soils) indicates approx. the same amount of acid as the exchange acidity recorded by Daikuhara's method. A method for determining the base required to neutralise exchange acidity is described.

A. G. POLLARD.

**Fixation of phosphoric acid [in soils] by titanium dioxide.** E. UNGERER (Z. Pflanz. Düng., 1931, 21A, 129—138).—Artificial and naturally occurring  $TiO_2$  adsorbed  $H_3PO_4$  from alkali phosphate solutions. Such adsorbed  $PO_4$  was practically insol. in  $H_2O$  and dil. acids and was not assimilable by seedlings. It is suggested that a portion of the soil phosphate may be rendered unavailable by this means. The presence of  $TiO_2$  in soil may introduce error into determinations of total P through the occlusion of P by titanic acid

formed during the evaporation of the acid extract of soil and again in the subsequent dissolution of the phosphomolybdate prior to precipitation with magnesia mixture.

A. G. POLLARD.

**Transformations of phosphoric acid in soil and its determination.** W. SAUERLANDT (Z. Pflanz. Düng., 1931, 21A, 187—210).—The effects of fertilisers of  $CaO$ , S, and rock phosphate on the available P content of soils are examined by Neubauer tests and by chemical extraction methods. A modification of Wrangell's formula for determining the total P content of soils from that of successive soil extracts is suggested. Results of Nemec's extraction method for determining soil P are dependent on the total surface area of the soil particles as measured by means of the heat of wetting.

A. G. POLLARD.

**Colorimetric determination of the citric-soluble phosphoric acid of soils.** W. LESCH (Z. Pflanz. Düng., 1931, 21A, 222—231).—The phosphate in citric acid extracts of soils may be determined by the colorimetric phosphomolybdate method provided the disturbing effects of citric acid and  $SiO_2$  are avoided. Oxidation of citric acid by  $KMnO_4$  is satisfactory and the effect of  $SiO_2$  is obviated by the use of  $SnCl_2$  as the reducing agent.

A. G. POLLARD.

**Comparison of the colorimetric determination of the citric-soluble phosphoric acid in soil by the permanganate method and that of Arrhenius.** A. NĚMEC [with J. LANIK and A. KOPPOVA] (Z. Pflanz. Düng., 1931, 21A, 231—233).—For soils of low P content results of Arrhenius' method (B., 1929, 31) do not agree with those of the author's (B., 1930, 207). By the latter method, differences between the values obtained by use of 5 c.c. of extract (= 0.5 g. of soil) and 10 c.c. of extract (= 1.0 g. of soil) are much smaller than by Arrhenius' method.

A. G. POLLARD.

**Mobility of phosphoric acid [in soils].** M. ESCHENHAGEN (Z. Pflanz. Düng., 1931, 10B, 313—332).—Significant variations in the solubility of soil phosphate during a 6 months' period could not be detected by either of the several methods employed. Discrepancies between laboratory determinations of assimilable  $PO_4'''$  and the results of field trials cannot be explained on this basis. The true value of laboratory tests of  $PO_4'''$  availability and the significance of "limiting values" are probably much less than those usually attached to them.

A. G. POLLARD.

**Exchangeable cations of the soil and the plant. I. Relation of plant to certain cations fully saturating the soil exchange capacity.** K. K. GEDROIZ (Soil Sci., 1931, 32, 51—63).—Growth of oats was compared in a chernozem soil after saturation with various bases. Saturation with Ca produced the same yield as the natural soil. Soil saturated with Sr produced nearly as great a yield and, moreover, needed no fertilisation with Ca. Plants did not grow at all in soils saturated with H,  $NH_4$ , Na, K, Mg, Cd, Ba, Mn,  $Fe^{++}$ ,  $Fe^{+++}$ , Co, Ni, Cu, or Al, whether unfertilised or receiving N and P. Addition of  $CaCO_3$  with N and P to the H-saturated soil produced a crop equal to that from the original soil. Smaller crops were similarly obtained from samples saturated with Mg, Mn,  $Fe^{++}$ ,  $Fe^{+++}$ , and Al, but soil



saturated with any of the remaining bases was unproductive. Plants can utilise the unexchangeable Mg and K but not the Ca in soils. Removal of the exchangeable Mg and K still permitted the growth of a normal crop, but no growth whatever occurred in soils devoid of exchangeable Ca.

A. G. POLLARD.

**Method for the study of *Azotobacter* and its application to fertility plot soils.** I. H. CURIE (Soil Sci., 1931, 32, 9–25).—*Azotobacter* were more numerous in limed soils than in those receiving CaO and fertilisers.  $\text{NaNO}_3$  promoted the growth of *Azotobacter* to a greater extent than did superphosphate or KCl. Manure reduced the *Azotobacter* population. The organism was not found in soils more acid than  $p_{\text{H}}$  6.0. A new agar plate method for enumerating *Azotobacter* colonies is described.

A. G. POLLARD.

**Causes of the beneficial effect of brown coal on the growth of crops.** III. A. KISSEL (Brennstoff-Chem., 1931, 12, 245–251; cf. B., 1931, 507).—Field experiments with humin preps. from brown coal show these to be efficient fertilisers. The theory of their effect is discussed in detail. It is attributed not only to the improved physical nature of the soil but to a stimulation of the physiological processes in the plant, in particular the  $\text{CO}_2$  assimilation, by certain of the org. constituents of the humin prep. The use of such preps. as fertilisers constitutes a more efficient method of utilising the energy of low-value carbonaceous materials than either applying the materials themselves directly as fertilisers, or by burning them by any of the technical processes at present available.

A. B. MANNING.

**Effect of summer green manures on the ammonia and nitrate content of soils cropped for winter wheat; examination of the Woburn green manure plots.** T. J. MIRCHANDANI (J. Agric. Sci., 1931, 21, 458–468).—Poor crops of wheat following green manuring with tares and with mustard are ascribed to the heavy leaching of nitrate and a consequent deficiency at the crit. growth period. During the winter months  $\text{NH}_3\text{-N}$  exceeded the  $\text{NO}_3\text{-N}$  in the soil.

A. G. POLLARD.

**Winter leaching and the manurial value of green manures and crop residues for winter wheat.** E. M. CROWTHER and T. J. MIRCHANDANI (J. Agric. Sci., 1931, 21, 493–525).—The nitrification in soil of org. matter with C:N ratio 13:1 was more rapid than that with C:N = 26:1. In corresponding pot cultures wheat yields were higher in untreated soil and in soil receiving the 13:1 than the 26:1 material. After systematic winter leaching equal yields resulted from the use of the two materials. Crop reduction due to leaching was closely correlated with but not proportional to the amount of  $\text{NO}_3$  leached. Early nitrification reduces crop yields not only through the leaching of nitrate but by increasing the amount of N converted by micro-organisms into relatively unavailable forms. The failure of wheat after summer crops of tares and of mustard (cf. preceding abstract) is explained on the above basis. The C:N ratio of tares permits rapid nitrification, and early leaching losses are heavy. Mustard reduces the winter loss, but the N compounds present and those produced during the microbiological

decomp. of the excess C compounds become available too slowly for their adequate utilisation by the crop.

A. G. POLLARD.

**Action of frost on various arable soils and the resulting effects on the growth of oats and barley.** E. GÜNTHER (Landw. Jahrb., 1931, 73, 893–922).—Freezing increased the pore space, hygroscopicity, and  $\text{H}_2\text{O}$  capacity of acid soils but was without effect on soils containing chalk. The extent of the frost action was largely controlled by the  $\text{H}_2\text{O}$  content of the soil when frozen. Although the adsorptive power of soils for acid was increased by freezing, there was no change in reaction. Freezing did not increase the solubility of plant nutrients, but in acid soils the resulting improvement in physical condition permitted an increased intake of K and P by plants.

A. G. POLLARD.

**Value of lucerne.** F. MÜNTER (Z. Pflanz. Düng., 1931, 10B, 332–344).—Field trials are recorded establishing the value of lucerne as a fodder crop and as a soil improver for subsequent crops.

A. G. POLLARD.

**Nutritional physiology of buckwheat.** UMBERG (Z. Pflanz. Düng., 1931, 10B, 344–347).—In field trials with buckwheat, K fertilisers increased the hectolitre wt. of the grain and reduced frost injury. The efficiencies of the fertilisers used were in the order  $\text{K}_2\text{SO}_4 > 40\%$  potash salts  $>$  kainite.

A. G. POLLARD.

**Availability of phosphoric acid in ammoniated superphosphates.** K. KITSUTA and R. M. SALTER (Ind. Eng. Chem. [Anal.], 1931, 3, 331–333).—Max. solubility of  $\text{H}_3\text{PO}_4$  was obtained from the  $\text{H}_2\text{O}$ -insol. residues of the ammoniated superphosphates when 0.1 g. of the sample was used in 100 c.c. of citrate solution for 30 min. at  $65^\circ$ . Similar results were obtained using a 2-g. sample in 100 c.c. of citrate solution containing 4 g. of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  and digesting for 2–4 hr. The time of digestion may be shortened to 30 min. by addition of larger quantities of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  in the case of highly ammoniated superphosphates, but not when the content of  $\text{NH}_3$  is less than 2.5%. There was no noticeable decrease in the availability of the  $\text{P}_2\text{O}_5$  of ammoniated superphosphates to buckwheat and barley when the material contained as much as 6.2%  $\text{NH}_3$ .

E. S. HEDGES.

**Chemical treatments to shorten the rest period of tree seeds.** C. G. DEUBER (Science, 1931, 73, 320–321).—Dormant seeds of sugar maple, Norway maple, and acorns of black oak and red oak can be stimulated into germination by treatments with solutions of thiocarbamide and ethylene chlorohydrin and the vapour of the latter.

L. S. THEOBALD.

**Effect of ammonium sulphate on the field germination of rape and turnips.** J. W. WOODCOCK (N. Zealand J. Agric., 1931, 42, 89–90).—Sown with the seed of rape and turnips  $(\text{NH}_4)_2\text{SO}_4$  caused a heavy reduction in germination. The effect was similar when superphosphate, with or without lime, was applied simultaneously.

A. G. POLLARD.

**Yield in cereals.** Victoria. I. H. C. FORSTER and A. J. VASEY (J. Agric. Sci., 1931, 21, 391–409).—Factors affecting grain yields in Australia are examined. Superphosphate increased the number of plants per unit

area, number of ears per plant, and the size of the heads. Top-dressing with N produced a type of growth of lowered drought-resistance.

A. G. POLLARD.

**Influence of sulphur compounds in breaking the dormancy of potato tubers.** L. P. MILLER (Contr. Boyce Thompson Inst., 1931, 3, 309—312).—Among S compounds stimulating the sprouting of potatoes were thiosemicarbazide, thioglycolic acid,  $\text{AcSH}$ ,  $\text{H}_2\text{S}$ ,  $\text{Me}$  disulphide,  $\text{EtSH}$ , thioglycol, and various derivatives of dithiocarbamic acid. The  $\text{NH}_4$ -salt of the last-named was particularly effective.

A. G. POLLARD.

**Fumigation of seed maize.** W. H. DARRAGH (Agric. Gaz. N.S. Wales, 1931, 42, 377—378).—Fumigation of maize of high moisture content with  $\text{CS}_2$  leads to lowered germinative vitality if the treatment is prolonged at summer temp. A safe dosage should not exceed 5 lb. of  $\text{CS}_2$  per 1000 cu. ft., for not more than 48 hr. Thorough aeration should follow immediately.

A. G. POLLARD.

**Wheat.**—See XIX. **Tobacco.**—See XX.

See also A., Aug., 1930, Soils of bed of Lake Albert, S. Australia. 986, Nitrite formation by soil bacteria. 990, Effects of X-rays on growth of wheat seedlings.

## PATENTS.

**Manufacture of an ammonium potassium phosphatic fertiliser and of a utilisable slag.** E. URBAIN (B.P. 351,749, 4.7.30. Fr., 29.8.29).—A mixture of crude  $\text{Ca}_3(\text{PO}_4)_2$  rock containing Si, Al, and K (1 mol.  $\text{K}_2\text{O}$  per 2 mols.  $\text{P}_2\text{O}_5$ ), coal, and sufficient  $\text{CaO}$  to give a slag suitable for use as cement, is agglomerated by heating in steam at  $180^\circ$  in an autoclave and then heated in an electric or shaft furnace. The gases evolved are treated with atomised liquor containing the sol. constituents maintained at  $d$  1.5 by the addition of  $\text{H}_2\text{O}$ , and the excess liquor is treated with crude  $\text{Ca}_3(\text{PO}_4)_2$ ,  $(\text{NH}_4)_2\text{SO}_4$ , and  $\text{NH}_3$  to yield on drying at  $100^\circ$  a fertiliser containing 30—34%  $\text{P}_2\text{O}_5$ , 8—11%  $\text{K}_2\text{O}$ , and 10—11% N.

L. A. COLES.

**Production [ageing] of mixed fertilisers.** A. C. FINCH, K. W. YOUNG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,609, 8.4.30).—Mechanical mixtures comprising, e.g.,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{H}_2\text{PO}_4$  and/or  $(\text{NH}_4)_2\text{HPO}_4$ , and  $\text{K}_2\text{SO}_4$  are rendered non-caking by spraying with water or an aq. solution of one of the salts with agitation and heating.

L. A. COLES.

**Production of fertilisers.** R. GOLDBERG and K. O. SCHMITT, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,788,828, 13.1.31. Appl., 28.2.28. Ger., 16.12.26).—Crude  $\text{Ca}_3(\text{PO}_4)_2$  is treated with a wt. of  $\text{HNO}_3$  at least 10% in excess of that required for decomp., the product is treated with  $\text{K}_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$ , gypsum is removed, and the liquor is neutralised with  $\text{NH}_3$  conc., and dried by spraying.

L. A. COLES.

**Manufacture of products for use as manures and weed killers.** CHINOIN GYÖGYSZER ÉS VEGYÉSZETI TERMÉKEK GYÁRA R.T. (KERESZTY & WOLF) (B.P. 351,515, 20.3.30. Hung., 21.3.29).—Bauxite etc. is treated cold with conc.  $\text{H}_2\text{SO}_4$  and a  $\text{H}_2\text{O}$ -binding agent, e.g., calcined  $\text{CaSO}_4$ , or with oleum, in proportions such as

to yield a dry, easily strewn product; S or its ores, fertilisers, Ca ores, etc. may also be added.

L. A. COLES.

**Insecticides and fungicides.** I. G. McBETH and J. R. ALLISON, ASSRS. to LEFFINGWELL RANCHO CO. (U.S.P. 1,787,585—6, 6.1.31. Appl., [A] 25.2.27, [B] 20.8.27).—(A) An insecticide with an oil and water emulsion basis contains as stabilising and spreading material a protein, e.g., dried blood dissolved in dil.  $\text{NH}_3$ . (B) An emulsion of slow-drying oil containing a toxic compound sol. in the oil in preference to  $\text{H}_2\text{O}$  is intimately mixed with an emulsion of a penetrating oil, or the latter emulsion containing an alkaloid combined with a fatty acid and saturated with  $\text{HCN}$  is mixed with an emulsion of a slow-drying oil.

W. G. CAREY.

**Insecticides.** STANDARD OIL DEVELOPMENT CO., ASSCES. of N. A. SANKOWSKY (B.P. 352,062, 2.4.30. U.S., 29.4.29).—The use is claimed of  $\text{H}_2\text{O}$ -white petroleum naphtha, b.p. about  $205$ — $283^\circ$ , containing the extracts from  $\frac{1}{2}$ —3 lb. of dry pyrethrum flowers per gal.

L. A. COLES.

**Germicides, fungicides, etc. for soil sterilisation.** M. S. KHARASCH, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,787,581, 6.1.31. Appl., 10.11.26).—A fertiliser is mixed with a small quantity of a mercuri-org. compound, e.g., a mercurised phenol or chlorophenol.

W. G. CAREY.

## XVII.—SUGARS; STARCHES; GUMS.

**Photomicrographic studies of sucrose crystals.** S. WOODRUFF and H. VAN GILDER (J. Physical Chem., 1931, 35, 1355—1367).—Photomicrographs of sucrose crystals in fondants containing dextrose, laevulose, and different amounts of invert sugar are recorded. Dextrose, laevulose, and invert sugar regulate the size of crystals equally well. No relation could be found between  $p_H$  of the syrup and amount of inversion using different acids. Syrups containing more than 43.43% of invert sugar do not crystallise and those containing 16.3—23.55% give a semi-fluid cryst. mass. A difference of 6—10  $\mu$  represents that between a smooth and a gritty texture as judged by the sense of touch in the mouth.

L. S. THEOBALD.

**Value of the diastase test for honey in artificial honey.** J. PRESCHER and E. BOHM (Z. Unters. Lebensm., 1931, 61, 500—508).—Auzinger's test (B., 1910, 291) gives reliable results for artificial honey containing >10% of bee honey, but is doubtful for <10% or if the latter has been heated, when pollen analysis should be used. The test, which was carried out at  $40^\circ$  for 1 hr., was used for starch solutions of various concentrations with various sugar-honey mixtures. The results show that 5 c.c. of 20% pure honey will readily hydrolyse 1 c.c. of a 3—5% starch solution, but that addition of other substances, particularly sugars, inhibits enzyme action; e.g., in the presence of 90% of maltose and laevulose respectively, the hydrolysis is nil and 60% of that for pure honey. Allowance should be made for the extreme variations in enzyme activity shown by different honeys and for the colours developed by certain sugars on addition of I. The starch substrate should

be prepared by adding a suspension of 2.5 g. of sol. starch in 15 c.c. of cold  $H_2O$  to 50 c.c. of boiling  $H_2O$ .

J. GRANT.

**Determination of the fat content of marzipan and persipan products.** K. BRAUNSDORF (Z. Unters. Lebensm., 1931, 61, 494—500).—The fresh undried sample (10 g.) is ground and heated with 100 c.c. of  $H_2O$ , and 10 c.c. of Fehling's  $CuSO_4$  are then added followed by 10 c.c. of 0.25N-NaOH. The ppt. is washed with hot water, dried with the paper for 8 hr. at 100—110°, ground with 20 g. of sand, and extracted for 24 hr. (Soxhlet) with  $Et_2O$ , the residue after evaporation being dried for 1 hr. at 100—110° and weighed. Equally good results are given by  $C_2HCl_3$ , which, however, is more troublesome to use, whilst direct extraction, after grinding with sand, with  $Et_2O$  (B., 1925, 822) and especially with light petroleum may give results 3—5% low.

J. GRANT.

**Calculation of sucrose and dextrose contents of marzipan and persipan preparations from the polarisation before and after inversion.** J. GROSSFELD and M. SCHNETKA (Z. Unters. Lebensm., 1931, 61, 485—490).—Fincke's method (*ibid.*, 1926, 52, 423) is modified, a 10% solution of the sample being cleared with  $K_4Fe(CN)_6$  and  $Zn(OAc)_2$ , and 50 c.c. heated with 5 c.c. of 25% HCl at 67—70° for 5 min., cooled, and made up to 100 c.c. without neutralisation,  $[\alpha]$  being read before and after inversion in a 400-mm. tube. Clerget's formula is then used to calculate sucrose contents of about 80%, and a table of factors is given for other amounts (5.66 for 100% and 5.69 for 10%). Correction factors for the vol. of insol. matter are also tabulated. In certain cases (*e.g.*, in presence of invert sugars) it is preferable to determine the lævulose content by Kruisheer's method (B., 1929, 533) and thence the original sucrose, using the factor 1.90.

J. GRANT.

See also A., Aug., 939, Microchemical determination of dextrose in sugar solutions. 941, P and N in potato- and wheat-starch. 973, Determination of sugar and non-fermenting Cu-reducing substances.

#### PATENTS.

Gluing wood.—See IX. Adhesive.—See XV.

### XVIII.—FERMENTATION INDUSTRIES.

**Determination of alcohol in alcoholic beverages.** E. DE'CONNO and D. FUSCO (Annali Chim. Appl., 1931, 21, 288—295).—The sample (5—20 c.c. according to strength) is diluted with 30 c.c. of  $H_2O$  and distilled, 30 c.c. of the distillate being diluted to 100 c.c. 5 c.c. of this solution (containing less than 16 vol.-% EtOH) are added to a mixture of 20 c.c. of  $K_2Cr_2O_7$  solution (33.382 g. per litre) and 10 c.c. of  $H_2SO_4$  (d 1.84). After being kept in a stoppered vessel for 5 min. the excess of  $K_2Cr_2O_7$  is titrated with  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$  (135.31 g. per litre), using 0.1% aq.  $K_3Fe(CN)_6$  as indicator; 1 c.c.  $K_2Cr_2O_7$  solution = 7.943 mg. EtOH.

O. F. LUBATTI.

**Influence of bottle glass on the quality of wines.** R. MUMMENDEY (Z. Unters. Lebensm., 1931, 61, 514—519).—Bottle glasses are classified according to their respective contents of  $SiO_2$ ,  $(Al_2O_3 + Fe_2O_3)$ ,  $(MnO +$

$CaO + MgO)$ , and  $(K_2O + Na_2O)$ : (1) French champagne (oldest) type, 60.4, 7.4, 26, 6.2; (2) German mouth-blown, 62.6, 10, 19, 8.5; (3) German Owens glass, 64.0, 8.8, 15.5, 11.7; (4) Anglo-American feeder glass, 70.5, 2.1, 9.8, 17.6; (5) American carbon yellow glass, 73, 1, 9, 17.2%. The solubility of the glasses in water increases with the alkali-metal content, and is due mainly to  $Na_2O$ , which, however, is present in quantities too small to neutralise the acidity of the wine to an appreciable extent. A poor and a good-quality wine were stored in bottles of various colours, shapes, and compositions for 6 months, but the max. differences in  $p_H$  value between the contents of any pair of the bottles were then only 0.013 and 0.05, respectively. No marked differences in taste were detectable in these experiments or after addition of 24 mg. of  $Na_2CO_3$  per litre of wine.

J. GRANT.

**Qualitative evaluation of spirits, particularly rum, arrack, and kirsch.** H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1931, 61, 465—479).—Spirits may be assessed qualitatively by determination of the max. dilution at which the characteristic flavour is still detectable. These values are tabulated for 83 samples of rum, and are correlated with EtOH content (43.4—80.8 vol.-%; 71—81, mean 77, vol.-% for Jamaica rum), and ester val. (11—3081 g. of AcOEt per 100 litres of pure EtOH present). The best types contained about 75% of EtOH and had ester val. 600—700. Arrack (23 samples): 58—60 vol.-% of EtOH and ester val. 228—471. Kirsch (23): EtOH 31.0—54.5%, acid val. (as AcOH) 12—308, ester val. 56—684, and fusel oil 65—572 (all as mg./100 c.c. of pure EtOH). An approx. correlation exists between the ester and fusel oil contents. A special rectification apparatus is described.

J. GRANT.

See also A., Aug., 975, Sol. enzymes in the spleen. 984, Zookinase. 985, Co-zymase. Decomposition of malic acid by *Saccharomyces* in wines. 986, N fixation and  $NH_3$  production by *Azotobacter*. Nitrite formation by soil bacteria other than *Nitrosomonas*.

### XIX.—FOODS.

**Influence of manurial treatment on the baking quality of English wheat. I. Quality of Rothamsted Broadbalk wheats.** E. A. FISHER and C. R. JONES (J. Agric. Sci., 1931, 21, 574—594).—A consistent increase in N content of wheat was produced only by heavy dressings of N fertilisers. Improved baking quality was not a necessary outcome of an increased protein content. Poor quality of flour was not due to unsoundness of grain or to poor gas production during fermentation. Neither baking quality nor storage properties of wheat could be correlated with manurial treatment.

A. G. POLLARD.

**Lævulosans in cereals. [Detection of rye flour.]** A. GIOVANARDI (Annali Chim. Appl., 1931, 21, 296—302).—The presence of 0.4 to 0.8% of a lævulosan in rye flour is confirmed. This carbohydrate has properties similar to those of secalose ( $\beta$ -lævulin; B., 1895, 377) and trifructose (B., 1929, 70). Traces of this lævulosan were found in wheat but could not be isolated from barley and oats. On this property is based a test for the detection of rye flour

in mixtures. 10 g. of flour are shaken for 15 min. with 40 c.c. of 70% EtOH; after cooling for 5 min. at  $-3^{\circ}$  the mixture is centrifuged and 10 c.c. of supernatant liquid are treated with 0.5 c.c. of *N*-NaOH in 70% EtOH. A product containing more than 10% of rye flour will show a strong turbidity followed by formation of a ppt. The test is applicable to bread after drying for 3 days in the air at room temp. O. F. LUBATTI.

**Normal variation in the f.p. of milk and in the "simplified molecular constant."** J. STRAUB (Chem. Weekblad, 1931, 28, 427—430).—The normal f.p. is for America  $0.54(7)^{\circ}$  or  $0.54(3)^{\circ}$ ; Germany  $0.54(2)^{\circ}$ ; Holland  $0.54^{\circ}$ ; Dutch E. Indies  $0.53(2)^{\circ}$  with a total normal variation (min. to max.) of  $0.010^{\circ}$ . Occasional abnormal variations are noted and discussed. The low f.p. for Dutch E. Indies confirms Donath's results. S. I. LEVY.

**Analysis of butter.** A. VERCILLO (Annali Chim. Appl., 1931, 21, 229—238).—For 10 genuine Italian butters, determinations have been made of the Reichert-Meissl and Polenske vals., the insol. Ag value (cf. B., 1928, 208), Savini's acetic index (Ann. Ist. Sperim. Caseif. Lodi, 1, 199), Kuhlmann and Grossfeld's butyric acid val. (B., 1926, 447), and the *A* and *B* vals. of Bertram *et al.* (B., 1926, 140). All these vals. give useful information on the composition of butter, but vary over such wide ranges that none of them allows a definite judgment to be made in the majority of doubtful cases. T. H. POPE.

**Fat and phosphatide contents of cacao beans.** B. REWALD and H. CHRISTLIEB (Chem.-Ztg., 1931, 55, 393—394).—The average water and fat contents of cacao beans are 5.2% and 53.4% respectively. The fat content is normally never below 50%, irrespective of the origin of the beans. The phosphatide content varies from 0.02 to 0.27% (av. 0.1%). In determining the phosphatide content the ground beans are exhaustively extracted with a 4:1 mixture of  $C_6H_6$  and EtOH; the solvent is distilled off, the fat and phosphatide are taken up with  $Et_2O$ , and after removal of the latter are filtered. The P content of the fat is determined and the phosphatide content is calc. on the assumption that lecithin contains 3.94% P. Analytical data for beans of various origins are given. W. J. BOYD.

**Natural phosphatide content of cacao beans and its change during manufacture.** B. REWALD and H. CHRISTLIEB (Z. Unters. Lebensm., 1931, 61, 520—523; cf. preceding abstract).—The phosphatide content of cacao beans is not affected by roasting and/or treatment with alkali. Phosphatide occurs only in small amounts in cacao butter separated by pressure and, as in other foods (cf. A., 1930, 261, 642), appears to be closely related to the protein constituents. J. GRANT.

**Determination of caffeine in coffee infusions and coffee extract.** (MME.) S. GOBERT (Ann. Falsif., 1931, 24, 288—290).—The sample is treated with a little  $NH_3$  after dilution with water and the mixture extracted with  $CHCl_3$  in the automatic apparatus of Jalade, for 5 hr. in the case of infusions and 10 hr. for extracts. The caffeine so extracted is purified with  $KMnO_4$  and weighed after re-extraction with  $CHCl_3$ . E. B. HUGHES.

**Evaluation of coffee [labelled] "non-injurious to health."** H. SCHLOSSMANN (Z. Unters. Lebensm., 1931, 61, 509—511).—A prep. sold for addition to coffee consisted of packets containing 4.5 g. of ground active charcoal, which removed 31—32% of the total caffeine and 4—6.5% of other N compounds from 20 g. of ground coffee in 400 c.c. of hot  $H_2O$ . Merck's carbo medicinalis removed 97.5 and 43.5%, respectively, but impaired the flavour and inhibited filtration; kieselguhr was also unsuitable (cf. Rinck and Kaempf, B., 1930, 790). J. GRANT.

**Test for predetermining the culinary quality of potatoes as affected by the accumulation of soluble sugars.** W. M. PEACOCK and B. C. BRUNSTETTER (U.S. Dept. Agric. Circ., 1931, No. 158, 4 pp.).—Potatoes for "chips" should have a min. sol. sugar content. The picric acid test (heat 1 c.c. of saturated picric acid solution + 1 c.c. of 20% aq.  $Na_2CO_3$  +  $1 \times \frac{1}{8}$ -in. potato core for 1 min. and allow to cool) should give a light yellow colour only. A. G. POLLARD.

**Bleaching almonds.** A. A. RAMSAY and G. W. NORRIS (Agric. Gaz. N.S. Wales, 1931, 42, 153—154).—Lightly weather-stained almonds are bleached by immersion in 1% aq.  $H_2SO_3$  for 2—5 min. and spread out to dry. For black-stained nuts this process should be preceded or followed by dipping in aq. bleaching powder (1 oz. per gal.) acidified with AcOH for 20 min. with an intervening drying period of 12—24 hr. A. G. POLLARD.

**Possible effect of sulphur dioxide when used as a preservative for dried fruits etc.** C. E. SAGE (Analyst, 1931, 56, 451—452).—"Sulphured" fruit and "sulphited" pulps when moistened or diluted with syrup develop an objectionable amount of  $H_2S$  if fermented by yeast. In the prep. of soft drinks etc. sterilisation is essential. T. McLACHLAN.

**Pectin from *Citrus limetta*, Risso.** A. ROMEO (Annali Chim. Appl., 1931, 21, 268—287).—This fruit, rich in linalyl acetate (26—30% of epicarp) and containing 8.5% of sugar, may be utilised for the extraction of pectin. The rind treated by G. Romeo's method yielded 3% of pectin. Analytical data show that the protopectin of *Citrus limetta* is hydrolysed to an araban identical with Ehrlich's tetra-araban and a sol. pectin (Ca and Mg pectate) the mol. of which contains 3 OMe groups and has  $[\alpha]_D^{25} +177.77^{\circ}$ , jelly grade 150. O. F. LUBATTI.

**Chemical changes in fish flesh and [brine] pickle produced by different pickling processes.** W. UGLOW and O. DONETZKY (Z. Unters. Lebensm., 1931, 61, 479—485).—Fish dry-salted and stored for 7 months increased in dry solids 18.8, ash 12.2, NaCl 10.1, protein 2.3, and fat 4.3%. The  $pH$  val. of an extract of salted fish is normally 6.6—7.2, but may be higher for non-eviscerated samples, in which cases the  $NH_4$  salts are also increased.  $HNO_3$  and  $H_2SO_4$  were not detectable in the extract. The ratio  $NH_3$ : total N of the fish varies according to the conditions, e.g., 1:640 for fresh fish, 1:232 after evisceration, storage 3 days on ice, and pickling in fresh brine, 1:118 for fresh fish and 1:108 for foul fish in used brine, and 1:40 for unsalted fish kept for 3 days at room temp.

The ratios found for the pickle itself were 1:15 with salted, frozen eviscerated fish, 1:9, 1:5, and 1:3.6, respectively, if drawing, freezing, and both were omitted from the process, and 1:5.5 for foul eviscerated fish.

J. GRANT.

**Injury of foodstuffs by "menstrual poison."** T. SÜTTERLIN and B. SZELINSKI (Z. Unters. Lebensm., 1931, 61, 511—514).—Evidence from medical literature indicates that excessive sweat from the hands of menstruating women may contain substances such as choline (from lecithin),  $\beta$ -hydroxybutyric acid, and "menotoxin" (cf. Böhmer, A., 1928, 912) which inhibit fermentation by yeast. Spoilage of handled food in this way is, however, improbable.

J. GRANT.

**Digestibility and utilisation of hay and straw in the feeding of working horses.** K. HÖTZEL (Landw. Jahrb., 1931, 73, 937—998; cf. B., 1931, 741).—Chopped rye straw is used to replace an equal wt. of hay in rations for working horses. Experimental results indicate that the standards of Kellner and of Nils-Hansson for the starch val. and digestible protein in straw rations should be lowered.

**Cacao butter.**—See XII. **Honey.** **Marzipan.**—See XVII. **Waste-water fungi.**—See XXIII.

See also A., Aug., 1926, **Determination of Na + K in milk etc.** 975, **Egg-yolk lecithin.** 978, **Mn in foodstuffs.** 988—9, **Vitamins, various.** **Vitamins in liver- and butter-fat, and in cereals.**

#### PATENTS.

**Extraction of albumin and sugar from milk whey.** (Miss) E. M. MEYER (U.S.P. 1,787,754, 6.1.31. Appl., 16.4.29).—Whey is treated with up to 4% of slaked lime and then water, up to 100%, added slowly with gentle stirring. After keeping, the upper layer containing albumin and some sugar is removed. Addition of 1—2%  $\text{KNO}_3$  prior to the  $\text{CaO}$  may be advantageous.

E. B. HUGHES.

**Treating or packaging ripe or green olives.** A. B. STEWART (B.P. 351,813, 27.8.30).—On the top of the brine in which olives are preserved is placed a layer of edible oil. Flavouring materials such as garlic may also be added to the brine.

E. B. HUGHES.

**Concentrated fruit juice.** P. GARRETT (U.S.P. 1,790,140, 27.1.31. Appl., 14.2.27).—Fruit juices are conc. in vac. at low temp. ( $38^\circ$ ) to such concentration as will prevent fermentation. Normal bacteria are inhibited, but become active on dilution of the concentrate so that natural fermented beverages can be thus prepared. Apparatus is claimed.

E. B. HUGHES.

**Meat treating method and product.** SWIFT & Co. (B.P. 351,700, 28.5.30. U.S., 6.6.29).—Fresh meat is cooled to a uniform temp. of  $-8^\circ$  to  $-2^\circ$  and cured meat to  $-10^\circ$  to  $-7^\circ$ .

E. B. HUGHES.

**Decolorising and deflavouring colloidal solutions.** W. A. BENDER, R. DOUGLAS, and L. H. CUTHBERT, Assrs. to GEN. FOODS CORP. (U.S.P. 1,787,467, 6.1.31. Appl., 19.8.25).—Colloidal solutions, e.g., pectin, gelatin, may be clarified by passing through layers composed of powdered charcoal mixed with fibrous

material such as sulphited paper pulp, giving rapid percolation and min. C turbidity of the filtrate.

E. B. HUGHES.

**Process of and apparatus for the production of a chocolate mass ready for rolling.** HERMANN BAUERMEISTER MASCHINENFABR. & MÜHLENBAUANSTALT G.M.B.H. (B.P. 352,852, 22.10.30. Ger., 22.10.29).

**Rendering process.**—See XII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Assay of the cinchona galenicals.** P. A. W. SELF and C. E. CORFIELD (Pharm. J., 1931, 127, 74).—2 g. of Extractum cinchonæ are washed into a separator with 10 c.c. of a mixture of equal vols. of EtOH and  $\text{H}_2\text{O}$ , 1 c.c. of  $\text{N-HCl}$  and 20 c.c. of  $\text{CHCl}_3$  are added, and after shaking the  $\text{CHCl}_3$  layer is run into a second separator and washed with 5 c.c. of  $\text{N-H}_2\text{SO}_4$ . The first extraction is repeated, using two further quantities of  $\text{CHCl}_3$ , and washing the  $\text{CHCl}_3$  layers with the same acid liquid as before. To each separator 2.5 c.c. of NaOH solution are added and the contents of the first are extracted with successive portions of 20 c.c. of  $\text{CHCl}_3$ , transferring the extracts in turn to the second separator until the alkaloids are completely removed. The mixed extracts are washed with  $\text{H}_2\text{O}$ , the  $\text{CHCl}_3$  is distilled off, 5 c.c. of EtOH are added, and the evaporated product is dried at  $100^\circ$  and weighed. The method, which is but slightly varied for other cinchona preps., is claimed to overcome emulsification difficulties.

E. HOPKINS.

**Determination of morphine.** II. L. DÁVID (Pharm. Ztg., 1931, 76, 748—749; cf. A., 1931, 972).—The method is applied to the determination of morphine in Domopon (opium concentratum), Capita Papaveris, and Syrupus Diacodii.

E. H. SHARPLES.

**Determination of strychnine in Easton's syrup.** I. A. HADDOCK and N. EVERS (Pharm. J., 1931, 127, 72).—Detailed procedure for extracting the HCl solutions of the alkaloids with  $\text{CHCl}_3$  and separating the strychnine is given.

E. HOPKINS.

**Biological and spectroscopic tests on ergot alkaloids: Maurice Smith colour test.** F. WOKES and H. CROCKER (Pharm. J., 1931, 127, 83—84).—Ergotoxine, ergotamine, ergotinine, and ergotamine all show in aq. or alcoholic solutions as dil. as 0.001% a characteristic absorption band with max. at 316  $\text{m}\mu$ , whilst ergotinine and ergotamine also give a smaller band with max. at 242  $\text{m}\mu$ , although this cannot be used for their quant. differentiation. The blue colour given by all four alkaloids with Maurice Smith's reagent has a max. at 580  $\text{m}\mu$  and a smaller band at 401  $\text{m}\mu$ , but the colour intensity as determined by tintometer and spectrophotometer is not a satisfactory measure of the physiological activity, values for ergotinine and ergotamine being much higher than their relative activity. Parallel tests by spectroscope and biological methods on fresh and deteriorated ergot preparations do not give concordant results.

E. HOPKINS.

**Variations in potency and total solids of tinctures of digitalis.** F. WOKES (Pharm. J., 1931, 127, 82—83).—No definite relation exists between the potency and total solids of the tinctures examined, even in those

prepared from the same batch of leaf. The total solids of 100 commercial tinctures vary from 1.8 to 5.98% wt./vol., whilst 40 tinctures prepared in the laboratory from 11 different samples of leaf show a small variation, 2.56 to 4.16%, with, however, the same average, 3.24%. Total solids of tinctures from the same batch of leaf may show differences of 20% from one another and 11% from the average. The potencies of the 100 commercial tinctures ranged from 49 to 100% of that of the standard tincture. 24 samples of leaf had potencies of from 63 to 156% of that of the international standard digitalis powder, with nearly the same average potency, 114%, as for the tinctures, 113%. Tinctures from the same batch of leaf varied in potency as much as 34% from the average. E. HOPKINS.

**Quantitative methylation of theobromine and theophylline, and determination of these substances in theobromine sodium salicylate and theophylline sodium acetate.** P. A. W. SELF and W. R. RANKIN (Pharm. J., 1931, 127, 75—76).—Compounds or mixtures containing theobromine or theophylline are methylated with  $\text{Me}_2\text{SO}_4$  and NaOH. A  $\text{CHCl}_3$  extract gives anhyd. caffeine on evaporation and drying at  $100^\circ$ , in yield of 99.5% from theobromine and 98% from theophylline. E. HOPKINS.

**Theophylline sodium acetate of commerce.** G. J. W. FERREY (Pharm. J., 1931, 127, 74—75).—Of 12 samples examined, 3 consisted of theobromine-NaOAc, whilst most of 8 others were deficient in theophylline, mainly due to excess of  $\text{H}_2\text{O}$ . A standard of not less than 60% of anhyd. theophylline and not more than 5%  $\text{H}_2\text{O}$  is suggested. E. HOPKINS.

**Assay of the official balsams.** T. T. COCKING (Pharm. J., 1931, 127, 73—74).—The balsamic acids are present as esters of aromatic and resinoid alcohols, and in the free state. Extraction with boiling  $\text{H}_2\text{O}$  is incomplete, but boiling with  $\text{H}_2\text{O}$  and MgO in the presence of xylene is satisfactory. The total acids from the saponified balsams are converted into Mg salts, and the balsamic acid salts separated from those of the resin acids by filtration. An  $\text{Et}_2\text{O}$  solution is then extracted with aq.  $\text{NaHCO}_3$  to remove aromatic alcohols, and the balsamic acids are finally extracted from an acidified solution with  $\text{Et}_2\text{O}$ , dried in vac. over  $\text{H}_2\text{SO}_4$ , and weighed. The method is adaptable to the determination of the free balsamic acids of benzoin and balsam of tolu. E. HOPKINS.

**Resin of *Ipomoea*: Mexican scammony root: its solubility in ether and the acid value as a test for rosin.** C. E. CORFIELD and W. R. RANKIN (Pharm. J., 1931, 127, 76).—Differences in the results of previous workers were caused by variations in the vol. and sp. gr. of the  $\text{Et}_2\text{O}$  used in extraction. 1 g. of resin, after drying at  $100^\circ$ , should yield less than 0.4 g. of  $\text{Et}_2\text{O}$ -insol. material on extraction with 50 c.c. of  $\text{Et}_2\text{O}$  of  $d$  0.720. Determination of the acid val. of the  $\text{Et}_2\text{O}$ -sol. portion is recommended as a test for the absence of colophony. E. HOPKINS.

**Potassium, chlorine, and sulphate content of Kentucky tobacco as related to grade.** O. M. SHREDD (Kentucky Agric. Exp. Sta. Res. Bull., 1930, No. 308, 447—471).—Wide variations in the mineral

contents of tobaccos are recorded (0.8—7.3% K, 0.02—1.50% Cl, 0.09—0.81%  $\text{SO}_4$ ). High proportions of Cl and  $\text{SO}_4$  were not usually accompanied by high K. Good quality tobaccos were usually rich in K, and in general the K, Cl, and  $\text{SO}_4$  contents were higher in good than in inferior grades. Burley tobaccos had higher K contents, much higher Cl, and slightly lower  $\text{SO}_4$  than dark grades. Variations in K contents were paralleled by the market values. In dark grades the leaves were richer in K than the trash. Organically combined K predominated in the better grades of smoking tobaccos. Leaf disease tended to cause a decrease in the proportion of K, Cl, and (to a small extent)  $\text{SO}_4$  in the leaves and is associated with nutritional disorders. A. G. POLLARD.

**Volumetric assay of lactic acid, B.P.** H. BRINDLE (Pharm. J., 1931, 127, 80).—Consistent results, within 1% of those given by the B.P. 1914 method, are obtained for total acidity by keeping the sample for 0.5 hr. with excess of NaOH, and back-titrating with standard acid. In all cases boiling with excess of alkali for 5 min. gives results identical with those obtained by longer boiling. A separate determination of lactide is difficult and unnecessary. E. HOPKINS.

**Hydrolysis of acetylsalicylic acid in alcohol, glycerol, and alcohol-glycerol solutions.** F. G. GERMUTH (J. Amer. Pharm. Assoc., 1931, 20, 568—572).—Acetylsalicylic acid is hydrolysed at slightly more rapid rate in aq. EtOH than in aq. glycerol of similar concentration, but in an aq. mixture of the two alcohols hydrolysis is not so great as in either alone containing the same amount of  $\text{H}_2\text{O}$ . No hydrolysis occurs with the pure alcohols. E. H. SHARPLES.

**Correlation of chemical and toxicity tests on mercurochrome.** H. A. MITCHELL (Pharm. J., 1931, 127, 72—73).—Of the 9 examined, 4 samples having relatively high toxicity contain much ionic Hg and give a large sediment. Maladjustment of  $p_H$  or use of an excess of Hg in manufacture might cause decomp. on dissolution to give an ionised Hg compound which later ppt. Toxicity on rabbits does not vary with Hg content. Total Hg is determined by oxidising the sample with alkaline  $\text{KMnO}_4$ , adding excess of oxalic acid and dil.  $\text{H}_2\text{SO}_4$ , and precipitating as  $\text{HgS}$ , while ionic Hg is similarly precipitated from the filtrate from an acidified solution of the drug. Standards suggested: moisture content of commercial samples to be stated (it may vary from 2.65 to 15.96%); content of Hg to be 26—28% of dry wt.;  $p_H$  to be 8.25—8.55; ionic Hg in 100 c.c. of a 2% solution and sediment from 100 c.c. of a 0.05% solution to be only a trace. E. HOPKINS.

**Solubility relationships of iodine and tannin in water. "Iodotannicum" produced in dry form.** A. MIHALOVICI and G. SPECH (Pharm. Zentr., 1931, 72, 419—421).—During the preparation of Sirupus iodotannicus according to the French Pharmacopoeia, free HI is formed first due to I substitution in the tannin and this with excess I forms  $\text{HI}_3$  which again reacts with the tannin and finally an I-free solution is obtained, but on keeping a cryst. I-free ppt. is formed by disruption of the tannin and free I is liberated. The solution probably consists of iodotannin and I-substitution products of gallic, ellagic, and luteolic acids together

with  $\text{HI}_3$ . Prep. in  $\text{H}_2\text{O}$ -free solvents forms no I-substitution products. Dry iodotannicum is prepared from a solution of powdered I (30 g.) and tannin (60 g.) in  $\text{H}_2\text{O}$  (410 g.) made by heating at  $45\text{--}50^\circ$ , by evaporation in vac. at  $30\text{--}33^\circ$  until of syrupy consistency; after filtering and drying over  $\text{H}_2\text{SO}_4$  it forms reddish-brown plates (23% I). E. H. SHARPLES.

**Characteristics and assay of colloidal silver prepared by the chemical method.** G. GAUME (J. Pharm. Chim., 1931, [viii], 13, 609—617).—Colloidal Ag prepared by Paal's method with protalbic acid for therapeutic use should be grey-blue and lustrous. It should contain 70—72% Ag and 1—1.5% alkali (as NaOH), and give a clear 0.1% solution in water, unaffected by mixing with an equal vol. of 5% NaCl (cf. D.A.B. VI). R. K. CALLOW.

**Assay of drugs yielding essential oils.** G. R. A. SHORT (Pharm. J., 1931, 127, 84—85).—A quantity of drug sufficient to produce 2—3 g. of volatile oil is distilled under reflux with 500 c.c. of  $\text{H}_2\text{O}$  into a receiver containing  $\text{H}_2\text{O}$  and 5 c.c. of light petroleum. After 6 hrs. distillation the petroleum extract is filtered, evaporated on the water-bath at  $80^\circ$  in a current of air, dried over  $\text{H}_2\text{SO}_4$ , and weighed. Results are in fair agreement with those obtained by larger-scale distillations. E. HOPKINS.

**Pharmacopœial constants of chenopodium oil.** J. C. MUNCH and W. F. REINDOLLAR (J. Amer. Pharm. Assoc., 1931, 20, 564—568).—The ascaridole content of normal wormseed oils varies directly with the density and inversely with the alcohol-solubility. Other variables show no relationship. Consts. of 39 oils are given.

E. H. SHARPLES.

**Essential oil of hyacinth flower.** I. L. HOEJENBOS and A. COPPENS (Rec. trav. chim., 1931, 50, 708—710).—The essential oil of the hyacinth flower extracted by light petroleum (0.01% from the calyx) had  $d_{40}^{20}$  1.0423,  $n_D^{20}$  1.4981,  $\alpha_D^{20}$   $-0^\circ 12'$ , acid val. 3, ester val. 143, do. after acetylation 272. It contained eugenol, BzOH (possibly formed by hydrolysis during steam-distillation), cinnamaldehyde, PhCHO, benzoic esters, cinnamic alcohol, higher aliphatic alcohols, and possibly cœnanthol, but no  $\text{CH}_2\text{Ph}\cdot\text{CHO}$ . J. D. A. JOHNSON.

**Fluorescence of orange-flower water.** F. GRÉGOIRE and J. RIPERT (Ann. Falsif., 1931, 24, 290—295).—The fluorescence in Wood's light is measured, giving an index of the quantity of Me anthranilate present (limit of visibility, 1 mg. per litre). The method offers a means of distinguishing between the flower water and leaf water. E. B. HUGHES.

**Extraction of lemon essence by Peratoner's process.** P. LEONE (Annali Chim. Appl., 1931, 21, 259—268).—Bonelli's apparatus provided with a plate fractionating column operates directly on the oil contained in the acid liquid from the lemon presses. This liquid contains about 15 g. of essential oils (90% of limonene) per litre together with other volatile and non-volatile substances and some vegetable debris. The process is continuous, enzymic and bacterial action has no time to develop, and less water needs to be distilled, with consequent great fuel economy.

O. F. LUBATTI.

**Cresylic acid.**—See III. **Hypochlorites.**—See VII.

See also A., Aug., 959, Separation of carotene into its constituents. 966, Diagnosis of "veronalides." 970, Prep. of 3-carbamido-4-hydroxyphenylarsinic acid. "Tryparsamide." 972, Determination of citronellol and rhodinol in presence of geraniol and nerol. Determination of morphine. 981, Biological assay of ergot preparations. 987, Influence of soap on germicidal properties of certain Hg compounds. Prep. of adrenal extract. 988—9, Vitamins, various.

#### PATENTS.

**Manufacture of hydroxyacylaminoarsenobenzenes.** I. G. FARBENIND. A.-G. (B.P. 348,480, 15.8.30. Ger., 27.8.29. Addn. to B.P. 318,491; B., 1931, 654).—The glycollamidoarsenobenzenes etc. of the prior patent are made by condensing an arsine with a suitable arsenoxide or dichloroarsine. C. HOLLINS.

**Manufacture of [formaldehyde-bisulphite] derivatives of arseno-compounds.** I. G. FARBENIND. A.-G. (B.P. 348,479, 13.8.30. Ger., 24.8.29).—Unsymmetrical arseno-compounds,  $\text{OH}\cdot\text{CH}_2\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{As}_2\cdot\text{C}_6\text{H}_3(\text{OH})\cdot\text{NH}_2$ , where X is H or a substituent, are converted into non-irritant,  $\text{H}_2\text{O}$ -sol.  $\text{CH}_2\text{O}$ -bisulphite derivatives. Compounds from 3'-amino-4-glycollamido-4'-hydroxyarsenobenzene and its 2-Me derivative are described. C. HOLLINS.

**Preparation of tobacco free from nicotine.** ZIGARETTENFABRIK R. GREILING A.-G. (B.P. 351,779, 25.7.30. Ger., 14.11.29).— $\text{H}_2\text{O}$  is added until the moisture content is 20—25% and the tobacco extracted with low-boiling light petroleum in a chamber through which air mixed with  $\text{NH}_3$  is passed. E. B. HUGHES.

**Freezing colloidal liquids.**—See I. Cotton etc. **pulps.**—See V.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See also A., Aug., 917, Reduction of Ag halides, and a cheap developer. 920, Action of low-speed electrons on photographic emulsions. 928, X-Ray fibre photography.

#### PATENTS.

**Treatment of photographic pictures and kinephonographic films.** SOC. INDÉPENDANTE DE TELEGRAPHIE SANS FIL (B.P. 351,956, 26.3.30. Fr., 27.3.29).—The whole width of the film can be used for the acoustic record when the Ag is replaced by  $\text{BaCl}_2$ , which is transparent to visible light and opaque to infra-red radiation. Methods effecting the replacement via  $\text{BaO}$ , or the double chloride, or  $\text{Ba}_2\text{Fe}(\text{CN})_6$ , are detailed.

J. LEWKOWITSCH.

**Production of corrected colour-selection positives for colour-photography and multi-colour printing.** L. O. VAN STRAATEN (B.P. 353,151, 22.5.30. Holl., 6.9.29).

**[Photomechanical] method of transferring the pattern of materials, fabrics, skins, furs, etc.** MASA G.M.B.H. ZUR HERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 352,498, 9.4.30. Ger., 10.4.29).



## XXII.—EXPLOSIVES; MATCHES.

**Determination of sulphur in black powder.** V. ÖHMAN (Chem.-Ztg., 1931, 55, 394—395).—The  $\text{H}_2\text{O}$ -insol. material from 0.5 g. of the sample is burned in the bomb calorimeter at an  $\text{O}_2$  pressure of 25–30 atm. To ensure complete combustion, the material (contained in the filter-paper) and the wall of the calorimeter must be perfectly dry. 10 c.c. of  $\text{H}_2\text{O}$  are placed in the bottom of the calorimeter beforehand. After the bomb has cooled for 5–10 min. the contained gas is passed through dil.  $\text{H}_2\text{O}_2$  (10 c.c. of a 3% solution diluted with 50 c.c.  $\text{H}_2\text{O}$ ). The  $\text{H}_2\text{O}$  in the bomb and the rinsings are united, boiled to remove  $\text{CO}_2$ , and titrated with 0.1N-NaOH using phenolphthalein as indicator. The  $\text{H}_2\text{O}_2$  solution is also titrated. The titration of the latter should not exceed by more than 0.2 c.c. that of a blank, otherwise the combustion has been incomplete. The amount of the titration in excess of the blank (being < 0.2 c.c.) is added to the amount of the titration of the solution from the bomb. The weight of S present is calc. from the acidity of the solution.

W. J. BOYD.

**Deflagration temperature of colloidal powders.** A. MICHEL-LEVY and H. MURAOUR (Compt. rend., 1931, 193, 40–42).—The most satisfactory deflagration temp. is that obtained by heating thereto very rapidly 1–2 mg. of the explosive, for which an arrangement is described. Const. results are obtained. Nitrocellulose powders and nitroglycerin powders and also similar powders containing centralite deflagrate at 197–203°, pure glyceryl trinitrate at 206°, pentaerythritol tetranitrate at 213°, and an unstabilised guncotton at 154°. There seems no connexion between these temps. and the results of the ordinary stability test at 110°.

C. A. SILBERRAD.

## PATENTS.

**Smokeless powder.** S. G. NORTON, ASSR. to HERCULES POWDER Co. (U.S.P. 1,788,438, 13.1.31. Appl., 28.5.28).—A homogeneous smokeless powder is made from nitrocellulose (60–90%), nitroglycerin (5–30%), and Et abietate (5–25%).

W. G. CAREY.

**Flashlight powders.** C. KREBS (B.P. 352,118, 16.4.30).—A high-combustion smokeless flashlight powder is made by mixing powdered Zr (3 pts.) with Zr nitrate (4 pts.) or  $\text{Ba}(\text{NO}_3)_2$  (3 pts.). Other O-containing substances, e.g., chlorates, permanganates, etc., may be used combined with alkaline earths or with  $\text{NH}_4$ . Carbohydrates increase the rapidity and Al or Mg alters the spectrum.

W. G. CAREY.

## XXIII.—SANITATION; WATER PURIFICATION.

**Processes for de-acidifying drinking water.** R. SCHMIDT (Gas- u. Wasserfach, 1931, 74, 635–637).—Water with a carbonate hardness of 6° or more can generally be sufficiently de-acidified by aeration. If the carbonate hardness after de-acidification amounts to 4–6° sufficient de-acidification to ensure the production of a protective layer on the walls of the conduit can be effected by treatment with marble. With very soft waters containing little free  $\text{CO}_2$  treatment with marble brings about only a diminution and not a complete removal of the corrosive action. With waters of more

than 6° carbonate hardness treatment with marble effects the desired de-acidification only within certain narrow limits of water composition. Where aeration and treatment with marble fail de-acidification can generally be effected by treatment with lime water; this, however, requires careful supervision.

A. B. MANNING.

**Damage caused by waste-water fungi. II.** AMELUNG (Chem.-Ztg., 1931, 55, 394).—Injurious effects of fungal growths in streams polluted by waste waters from factories producing foodstuffs are described.

W. J. BOYD.

Cresylic acid.—See III.

## PATENTS.

**Boiler feed-water treatment.** P. M. CONTANT, ASSR. to NECKAR WATER SOFTENING CORP. (U.S.P. 1,788,149, 6.1.31. Appl., 11.10.28).—Feed water containing  $\text{NaHCO}_3$  is heated in the boiler to form  $\text{Na}_2\text{CO}_3$  and NaOH, and this water is continuously blown off and mixed with additional feed water containing  $\text{NaHCO}_3$  and with  $\text{FeSO}_4$ , forming  $\text{Na}_2\text{SO}_4$  and  $\text{FeCO}_3$ .

W. G. CAREY.

**Apparatus for sterilising liquids.** G. LAKHOVSKY (B.P. 351,992, 28.3.30. Fr., 18.9.29).—Small chains are made of platonix (25% Zn, 75% Ni) or any highly conductive and rustless bactericidal metal and after use are cleaned from CaO and org. matter by agitation.

W. G. CAREY.

**Material for killing insects.** R. C. ROARK and R. T. COTTON, ASSRS. to U.S.A. (U.S.P. 1,789,322, 20.1.31. Appl., 23.11.27).—A mixture of 1–9 vols. of an ester of monochloroacetic acid or other halogeno-fatty acid and 9–1 vols. of  $\text{CCl}_4$  or other non-inflammable chlorohydrocarbon.

W. G. CAREY.

**Fumigant solution.** H. W. HOUGHTON, ASSR. to SAFETY FUMIGANT Co. (U.S.P. 1,790,095, 27.1.31. Appl., 1.7.25).— $\text{CNCl}$  or other CN halide and HCN gas are dissolved in  $\text{H}_2\text{O}$  containing about 25% of glycerol or sol. carbohydrate and 10% of  $\text{COMe}_2$ .

W. G. CAREY.

**Purification of air.** P. O. ROCKWELL (U.S.P. 1,789,194, 13.1.31. Appl., 20.3.25).—Air vitiated with toxic gases, particularly  $\text{CNCl}$  and  $\text{HCN}$ , is passed through granular alkaline material (e.g., pumice + aq. NaOH), and then through granular activated C impregnated with hexamethylenetetramine.

W. G. CAREY.

**Purification of air.** G. ST. J. PERROTT and M. YABLICK, ASSRS. to MINE SAFETY APPLIANCES Co. (U.S.P. 1,787,875, 6.1.31. Appl., 2.11.25).—Air vitiated with  $\text{NH}_3$  is rendered suitable for breathing by passing it through  $\text{SiO}_2$  gel.

W. G. CAREY.

**Preservation of bodies and anatomical specimens.** J. O. FENÉS (B.P. 352,001, 28.12.29).—A support carrying the specimen is saturated with a substance containing  $\text{CH}_2\text{O}$  which is brought into action in the presence of  $\text{CaCl}_2$ , glycerin, or other hygroscopic substance to absorb the heat of reaction and to dry the gaseous  $\text{CH}_2\text{O}$ .

W. G. CAREY.

[Valves for] gas masks etc. SOC. ITAL. PIRELLI (B.P. 353,380, 5.3.31. Ital., 6.3.30).

Germicide. Insecticides.—See XVI.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 25 and OCT 2, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Boiling, drying, and heating with high-pressure hot water.** H. STENGER (Chem. Fabr., 1931, 4, 309—310).—The advantages of circulating high-pressure hot  $H_2O$  (e.g.,  $190^\circ$ ) through a heating system instead of steam are discussed. Any ordinary type of boiler working up to 30 atm. may be used and circulation of the  $H_2O$  through the system is ensured by installing a small rotary pump in the return circuit just before the boiler. As the return  $H_2O$  is at a temp. of only  $15\text{--}35^\circ$  below that of the  $H_2O$  in the boiler there is a considerable fuel economy, and as no provision need be made for reducing valves, draw-off cocks for condensed steam, etc., the circulating system is considerably simplified and the labour required in supervision is much reduced. The maintenance of steady temps. is also facilitated and a higher heat efficiency obtained.

A. R. POWELL.

**Detection of phosphate in boiler feed-water.** P. KÖPPEL (Chem.-Ztg., 1931, 55, 539—540).—The presence of a sufficient excess of  $Na_3PO_4$  in  $H_2O$  softened with this salt may be detected by the usual phosphomolybdate test; addition of 10 c.c. of the reagent preheated to  $70^\circ$  to 1 c.c. of the  $H_2O$  gives an immediate yellow ppt. when the  $P_2O_5$  exceeds 30 mg. per litre and a ppt. after 2 min. when less than this quantity is present. Well-softened  $H_2O$  should give the ppt. only after 1—2 min.

A. R. POWELL.

### PATENTS.

**Furnaces.** R. S. COCHRAN (B.P. 351,676, 19.5.30. U.S., 18.5.29).—The bed of the furnace is composed of rails which alternately are stationary and given a reciprocating and rising and falling motion. The outside rails of the moving part form part of a rigid frame which is hung by a number of bifilar suspensions protected as far as possible from the heat. The intermediate rails are attached to only one cross-member of the frame and allowed to slide on the other cross-members; when expansion takes place, these rails are maintained under tension. The surface of both sets of rails is wavy, the pitch of the waves being equal to the stroke.

B. M. VENABLES.

**Pulverulent-fuel furnaces.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,768, 16.7.30).—With the object of shortening the flame, that part of the secondary air which is supplied in the neighbourhood of the fuel burner is preheated by burning part of the fuel before it reaches the furnace proper. The apparatus may comprise a hollow and perforated refractory cylinder within the air supply pipe. B. M. VENABLES.

**Removal of dust such as [ash] from plant fired by pulverulent fuel.** CLARKE, CHAPMAN & Co., LTD.,

and W. A. WOODSON (B.P. 351,968, 1.4.30).—The dust is removed by water-operated ejectors through open-ended collecting pipes. B. M. VENABLES.

**Rotary tubular furnace.** E. SCHULZE, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,787,841, 6.1.31. Appl., 9.10.28. Ger., 11.11.27).—The material (e.g., ore, cement) falling from a rotary kiln is received in a funnel-like structure having adjustable walls. The side walls swing about hinges at the top, and the wall upon which the material first impinges is movable bodily and is provided with cooling coils; the opposite wall has a flap door in the lower part for discharge of the material. B. M. VENABLES.

**Heating furnace.** A. W. SMITH and R. B. JONES (U.S.P. 1,790,172, 27.1.31. Appl., 13.5.22).—A regenerative open-hearth furnace is provided with auxiliary currents of preheated air led to points in and at the side of the main ports, which are inlets for the time being, to cause effective mixing of the air and gas.

B. M. VENABLES.

**Heat-treatment furnaces.** HEVI-DUTY ELECTRIC Co., Assees. of W. B. COOLEY (B.P. 352,174, 21.5.30. U.S., 22.5.29).—A furnace having a belt conveyor for the treatment of articles without containers has the belt entirely within the furnace and both runs are supported by rollers which are positively driven to agree with the speed of the belt.

B. M. VENABLES.

**Tunnel kilns.** WOODALL-DUCKHAM (1920), LTD., and A. MCD. DUCKHAM (B.P. 353,086, 24.4.30).—In a tunnel kiln in which the goods are not permitted to come in contact with the combustion gases, in addition to being heated by radiation the goods are further heated by contact with clean air which has been raised to a high temp. in passages that alternate with the combustion chambers in the roof and walls.

B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 352,206, 12.6.30. U.S., 13.6.29).—In a Hg boiler the downtake tubes are screened from the fire, but the uptake tubes are divided into a bundle of smaller tubes around and between which good conducting material, solid at the temp. of operation, is placed. Twisted strips may be placed inside the tubes. B. M. VENABLES.

**Heat accumulators.** STEIN- U. THON-INDUSTRIEGES. "BROHLTHAL," and E. and H. POHL (B.P. 352,955, 14.4.30).—The filling of a heat accumulator is arranged in zones having bricks or tiles of exactly similar shape, but the number of passages through the successive zones is in the ratio 1 : 4 : 16 : 64 and so on. The walls are of equal total thickness and the apertures of equal total

\* The remainder of this set of Abstracts will appear in next week's issue.

area. At the junctions of zones the bricks are in the form of De Laval nozzles, and intermediately there may be expansions and contractions producing change of velocity energy into pressure energy and back again.

B. M. VENABLES.

**Chemical heat-storage devices.** A. JUST and M. I. W. J. BYLEVELD (B.P. 351,951, 20.3.30. Holl., 20.8.29).—Such a device (*e.g.*, a foot warmer) is composed of a hermetically sealed rigid vessel containing a salt having negative heat of dissolution and  $H_2O$  (*e.g.*, a 92.5% solution of NaOAc), also at least one solid body (*e.g.*, a few pebbles), and a divided substance (*e.g.*, sand) in sufficient quantity to hold the solid bodies steady unless the vessel is violently shaken. Under these conditions the solution will remain supersaturated when cold under ordinary shocks of transport, and will not lose that property after repeated use.

B. M. VENABLES.

**Drying apparatus, particularly of the continuously operating type.** SOC. ANON. DES ETABL. NEU (B.P. 352,363, 13.11.30. Fr., 13.11.29).—The goods are conveyed through the dryer twice by the same conveyor, on the outside of the upper run and inside of the lower run in succession; they are contained in trucks with foraminous bottoms, and mechanism is described for transferring the trucks from one run to the other.

B. M. VENABLES.

**Apparatus for drying, heating, or cooling.** F. J. WARDEN-STEVENS (U.S.P. 1,787,878, 6.1.31. Appl., 6.9.28. U.K., 18.3.27).—Lump or broken material is allowed to pass downward in a narrow space between two walls which are composed of louvres through which a treating medium enters and leaves. To loosen the material and cause it to flow freely, the louvres are attached to a framework which, when reciprocated, alters the angle of the slats; in addition, cross-bars through the material may be reciprocated up and down.

B. M. VENABLES.

**Drying of materials [tea etc.].** L. N. LLOYD, and PHILLIPS ENG. CO., LTD. (B.P. 351,976, 24.12.29).—A drying chamber is provided with forced- and induced-draught fans and the flow is reversed by a single valve operated at intervals by a funicular arrangement. Means for admixture of fresh air are described.

B. M. VENABLES.

**Rotating drums or cylinders for drying or roasting malt, grain, etc.** PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 352,933, 14.3.30).—A rotary cylinder is heated internally by a number of tubular combustion chambers parallel to the axis, which communicate by radial tubes with an annular space in the lining of the kiln.

B. M. VENABLES.

**Drying plant [for road-making materials etc.].** C. G. HODGSON, and MILLARS' MACHINERY CO., LTD. (B.P. 353,458, 23.4.30).—A rotary drum is provided with a combustion chamber adjacent the inlet end, and air, in addition to the gases of combustion, is admitted through ports in the same end after traversing an annular space between the drum and a surrounding shell.

B. M. VENABLES.

**Radiant reading pyrometer.** J. SCHÜNEMANN, Assr. to HARTMANN & BRAUN A.-G. (U.S.P. 1,788,849,

13.1.31. Appl., 16.12.27. Ger., 24.12.26).—In a radiation pyrometer the blackened hot junction is provided with a screen which obstructs the radiation more or less according to the temp. of the surrounding pyrometer body, the change of position of the screen being effected by a bimetallic strip.

B. M. VENABLES.

**Temperature control in chemical reactions.** C. R. DOWNS, Assr. to BARRETT CO. (U.S.P. 1,789,809, 20.1.31. Appl., 11.6.19).—The temp. of an exothermic reaction is regulated by the vaporisation of Hg distributed throughout the zone of reaction, but out of contact with the reagents. The process is applicable to the oxidation of naphthalene to phthalic anhydride.

B. M. VENABLES.

**Binary mixture for absorption refrigerating machines.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 353,806, 13.11.30. Ger., 6.12.29).—The mixture consists of PhMe as the evaporating medium and paraffin oil as the solvent. Part of the difference between condensation pressure and evaporation pressure may be compensated for by the addition of a neutral gas, *e.g.*, A.

D. K. MOORE.

**Refrigerants and absorbents for absorption refrigerating machines.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 351,713, 4.6.30. Ger., 5.6.29).—A refrigerant is chosen having an atm. b.p. close to 100° and a f.p. below 0°, also so that the 50% solution in a suitable solvent has a b.p. at least 10° above that of the pure refrigerant. Examples claimed are: Et isoamyl ether dissolved in paraffin oil, PhMe dissolved in quinoline. The difference in pressure between the absorber and generator can be attained by a reasonable head of liquid and neither mixture attacks steel.

B. M. VENABLES.

**Manufacture of thermal insulating material.** J. A. ROBERTS, R. W. HARDY, and T. HAYMAN (B.P. 352,680, 12.6.30).—Mica is reduced to small flakes and mixed with a paste made by adding boiling  $H_2O$  to a cold solution of flour in such a way that it has become aerated. The mixture is then formed into insulating jackets or other articles and charred by heat.

B. M. VENABLES.

**Sieving or sifting of aggregate material.** LONDON BRICK CO. & FORDERS, LTD., and G. H. C. RACTLIFFE (B.P. 352,155, 10.5.30).—The apparatus comprises a fixed frame, an outer uniformly rotating ring, and an inner ring carrying the screen which rotates with the outer ring but at intervals is held stationary by a catch and then released so that it resumes its normal position with a shock, under the action of springs.

B. M. VENABLES.

**Sorting of coal and other minerals.** W. H. and W. N. BARKER (B.P. 352,796, 2.9.30).—An apparatus of the inclined-plane type is maintained in a humid atm. either by means of a humidifying apparatus within an enclosing casing or by means of currents of humidified air.

B. M. VENABLES.

**Pneumatic separation of materials of different densities.** P. SOULARY, and COMP. DES MINES DE BRUAY (B.P. 352,943, 10.4.30. Fr., 15.4.29. Addn. to B.P. 320,207; B., 1929, 1036).—In an apparatus as described in the prior patent the separating zones are provided

with longitudinal vertical partitions, means are provided for regulating the distribution of air, and the shaking tables are given an upward component of motion by supporting them on leaf springs which are inclined at an angle not exceeding  $55^\circ$  to the horizontal.

B. M. VENABLES.

**Mixing devices.** C. HILDEBRANDT-SØRENSEN (B.P. 353,240, 16.7.30).—A pair of stirrers, eccentric to the vessel, rotate about their own shafts and simultaneously about the axis of the vessel; the lower part of one is in the form of a triangle with twisted apex, which latter runs in an annular channel formed between the sloping wall of the vessel and a conical centre piece, in which channel the discharge ports are placed.

B. M. VENABLES.

**Apparatus for mixing materials.** F. C. OVERBURY, Assr. to FLINTKOTE Co. (U.S.P. 1,789,320, 20.1.31. Appl., 7.12.25. Renewed 16.1.30).—The apparatus comprises a tank in which are a number of vertical, jacketed, cylindrical partitions, spaced from the bottom of the tank except in the case of the last, which is provided with a valved outlet. Each partition is provided with propellers driven by vertical shafts, and some may have heating-cooling coils in addition to the jackets. The flow of liquid is under and over the edges of the partitions and constituents may be added at various points. The apparatus is stated to be suitable for pasty materials.

B. M. VENABLES.

**Mixing machines for plasticising phenol compounds and such like.** D. A. DUCHARME and F. H. BANBURY (B.P. 352,803, 9.9.30. U.S., 31.5.30).—A mixing machine of the Banbury or other type is rendered usable on very sticky compounds by cooling the supply neck and ram or other feeder so that the material is fed in non-sticky condition.

B. M. VENABLES.

**Mixing, dissolving, or dispersing apparatus.** G. C. BAKEWELL, Assr. to TURBO-MIXER CORP. (U.S.P. 1,790,257, 27.1.31. Appl., 20.3.29).—A turbine-like rotor runs near the bottom of a vessel and is surrounded by fixed guide blades, which are again surrounded by plates having a toothed surface, attached to the bottom of the vessel.

B. M. VENABLES.

**Safety mixers.** MIXING EQUIPMENT Co., INC. (B.P. 351,808, 20.8.30. U.S., 28.8.29).—A mixing device suitable for attachment to the rim of a tank of inflammable liquid and embodying an electric motor has the latter pipe-ventilated, the air being drawn from a safe place and delivered in the general direction of the propeller.

B. M. VENABLES.

**Apparatus for making emulsions.** M. G. GREGG, Assr. to FLINTKOTE Co. (U.S.P. 1,789,897, 20.1.31. Appl., 19.6.22. Renewed 27.7.29).—The apparatus comprises two straight pieces of pipe provided with agitators and connected by two return bends to form a closed circuit.

B. M. VENABLES.

**Lixiviation of vegetable, particularly beetroot, slices.** K. PHILIPP, Assee. of A. REIHER (B.P. 352,923, 2.1.30. Ger., 14.1.29).—The lixiviation takes place in a rotary cylinder which is provided with annular end-walls to retain liquid and with lifting vanes which are

inclined so as to work the solid matter countercurrent to the liquid. Washing is completed in an elevator. [Stat. ref.]

B. M. VENABLES.

**Filters.** T. F. MILLER (B.P. 352,038, 6.1.30 and 26.2.30).—A metallic (or other non-fibrous) filter is constructed so as to retain a considerable quantity of filter aid on the edges of the metal. A metallic strip is wound round a central inlet (or outlet) pipe, and the strip is formed with raised portions to act as distance pieces and with central elongated perforations which are longer than the lands left between them, so that some part of the holes will overlap right through all the turns except the outermost which is left unperforated to form the outer wall; the feed tube is correspondingly perforated. Alternate strips may be of different widths (or of the same width but slightly staggered) so that the uneven edges form pockets to retain filter aid, and annular screens may be juxtaposed to the edges for additional support.

B. M. VENABLES.

**Filtering device.** A. E. TOMLINSON (B.P. 353,106, 2.5.30).—The pulp is applied to the inside of an endless belt of filter medium, is squeezed while passing over the pulley, and removed by scrapers or a reverse curve. The cake may be removed from one longitudinal zone of the belt and applied to another for washing or other treatment.

B. M. VENABLES.

**Filtration.** C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,789,425, 20.1.31. Appl., 5.5.28).—The apparatus comprises an inclined pervious conveyor running within and without a tank.

B. M. VENABLES.

**Filter press.** P. L. DORNER (U.S.P. 1,788,086, 6.1.31. Appl., 11.5.29).—The press is suitable, e.g., for reconditioning dry-cleaning fluids. It is divided into sections each of which is charged with a different filter aid or absorbent in suspension in clean fluid, after which dirty fluid is passed through all sections in series. The claims mainly refer to an arrangement of pipe-work and valves by which one pump may effect all the operations.

B. M. VENABLES.

**Separation of liquids from fine-grained material.** L. ALTPETER, and GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 352,645, 20.5.30).—A centrifuge of the type comprising upper and lower bowl walls with a hydraulically closed gap between the two for outlet of solid material is provided with a feed-distributing plate and a conical baffle, both rotating with the bowls and with sprays for washing the material while it is on the baffle and bowl walls.

B. M. VENABLES.

**Manufacture of filter blocks of paper pulp or similar material.** F. G. RILEY (B.P. 352,379, 1.5.30).—A press is described for the manufacture of blocks suitable for use in the apparatus described in B.P. 349,262 (B., 1931, 703).

B. M. VENABLES.

**Apparatus for making precipitable the solids held in solution in liquids.** G. W. GANE (U.S.P. 1,789,738, 20.1.31. Appl., 14.6.28. N.Z., 19.7.27).—The liquid is allowed to flow to and fro through a number of horizontal, double-concentric vessels, and at the ends of the inner flows is subjected to jets of steam which either aid or retard the flow.

B. M. VENABLES.

**Recovery of crystallisable constituents from liquors.** R. W. MUMFORD, Assr. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,790,436, 27.1.31. Appl., 14.9.25. Renewed 4.10.30).—The process is described in connexion with the recovery of KCl from Searles Lake brine, which consists mainly of the sulphates, chlorides, carbonates, and borates of Na and K. By evaporation at a high temp. the liquor may be brought to a condition such that, on cooling, KCl and, later, borax will crystallise out, the latter having the greater tendency to remain supersaturated. The cooling is therefore effected first by evaporation under reduced pressure with considerable agitation, H<sub>2</sub>O or KCl wash-liquor being added to replace loss by evaporation; before precipitation of borax begins the liquor is transferred to an indirect cooler having less agitation, in which the remainder of the KCl will grow round the small crystals already formed, while the borax remains in supersaturated solution. B. M. VENABLES.

**Centrifuging salts [ammonium sulphate].** N. V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & Co. G.M.B.H. (B.P. 352,828, 29.9.30. Ger., 28.9.29).—The centrifuge is provided with a system of chains in contact with the walls and round the base, the latter being attached to a hood on the bottom of the centrifuge. At the conclusion of the process the raising of the hood and chains breaks up the mass and removes adherent material from the walls, thus facilitating emptying. W. J. WRIGHT.

**Column.** C. B. SCHNEIBLE, Assr. to C. B. SCHNEIBLE, K. F. SCHREIER, and B. B. SCHNEIDER (U.S.P. 1,790,596, 27.1.31. Appl., 16.5.27).—A column for dephlegmating etc. is filled with alternate annular and central plates which do not overlap to any extent under the plates; also not overlapping the edge are a number of vanes inclined to the radial direction. B. M. VENABLES.

**Pipe still.** G. W. WATTS, Assr. to STANDARD OIL Co. (U.S.P. 1,789,267, 13.1.31. Appl., 18.7.25. Renewed 3.10.27).—The heating means comprise a combustion chamber and long flue, preferably after a return bend, and across the latter are a number of banks of tubes. The flow of oil (or other material) is first through a number of tubes in series, and lastly through coils comprising several tubes in parallel; intermediately the oil flows through a smaller number of tubes exposed to direct radiation in the combustion chamber, and at this point a supply of steam may be added. The operations may be effected under pressure; straight tubes are used and cellular connecting headers are described, with means to facilitate cleaning and renewal, and to eliminate return bends and tube sheets. B. M. VENABLES.

**Distillation control.** W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,789,339, 20.1.31. Appl., 31.3.28).—In a line of stills operating on, e.g., petroleum, the bottoms flowing from one of the later stills, usually the one effecting the final removal of gasoline, are subjected to continuous sampling at a uniform rate and the sample is heated with a const. supply of heat. The vapours are rectified with a const. reflux and the temp. of the rectified vapour is caused to vary the conditions of the main distillation

so as to hold the temp. of the rectified sample vapour nearly const. In the control still the const. heat supply is preferably electrical, and the const. reflux may be produced by the evaporation of a const. drip of water. B. M. VENABLES.

**Apparatus for dephlegmating vapours.** W. M. CROSS, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,789,670, 20.1.31. Appl., 29.10.23).—A tower containing means for producing a reflux, bubbling trays, and a reboiler is described. B. M. VENABLES.

**Fractionation.** T. DE C. TIFFT and W. MENDIUS, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,789,059, 13.1.31. Appl., 23.1.28).—A form of tray for a fractionating column is described. No bubbling caps or other apertures are provided. The top of each diaphragm is provided with a single weir extending across a chord, and the edge of each diaphragm is bent down along a chord rather more distant from the axis of the tower and terminates in an adjustable straight edge, so that the depending part dips into the pool formed by the weir on the plate below; the flow of the rising vapour is thus in a zig-zag manner over the weirs and under the depending edges, spray being produced at the latter points. B. M. VENABLES.

**Tubular filters [for gases].** E. W. STRAUS-SCHARINA (B.P. 353,600, 28.5.30. Ger., 28.5.29).—The filter sleeves are subjected to longitudinal shocks during the whole of the time they are in operation. B. M. VENABLES.

**Centrifugal apparatus for separating dust, grit, etc. from flue or furnace gases.** O. B. JACOBSEN (B.P. 352,374, 6.6.30).—In an apparatus as described in B.P. 341,058 (B., 1931, 281), the turbo-blades are supported between conical walls, the angle of the outer one being greater than that of the inner. B. M. VENABLES.

**Apparatus for separating and recovering gases [by adsorption].** O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,787,714, 6.1.31. Appl., 29.7.22. Renewed 3.11.28).—A number of separated layers of adsorbent are supported on coarse and fine gauze, or other foraminous 2-ply material, and each layer is provided with embedded means (such as a pipe-coil) for indirect heating or cooling and also for adding steam directly to the adsorbent material. B. M. VENABLES.

**Separation of gaseous mixtures by liquefaction.** M. FRÄNKEL (B.P. 352,570, 16.4.30. Ger., 19.4.29. Addn. to B.P. 246,172. Cf. F.P. 613,755; B., 1927, 513).—The major part of a compressed gas mixture is subjected to cold exchange with a resolved product of low b.p. by means of reversing regenerative cold accumulators, as described in the prior patent, the cold-accumulating substance being metallic and having large surface. The minor part of the gas mixture exchanges cold with the high-b.p. product by means of tubular countercurrent devices which may also be subjected to reversal at intervals, but only for the sake of removing frost. The high-b.p. substance may be compressed to a high pressure while in the fluid state before evaporation in the cold exchanger. B. M. VENABLES.

**Liquefaction of gases.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. W. HARROLD (B.P. 353,618, 6.6.30).—Schemes are described for the recovery by condensation of a gas of high b.p. from an atm. of low b.p., *e.g.*, SO<sub>2</sub> from pyrites gas; the nett amount of cold is provided by the evaporation of a liquid of low b.p., *e.g.*, CO<sub>2</sub>. The simplest scheme comprises compression in one side of an engine, cooling in three heat exchangers the last two of which deliver liquefied desired constituent, expansion in the other side of the engine, and return passage through the third and first exchangers, the second being cooled by the independent fluid.

B. M. VENABLES.

**Liquefaction.** W. L. DE BAUFRE and J. W. DAVIS (U.S.P. 1,786,898, 30.12.30. Appl., 29.12.23).—A scheme for the purification of a gaseous mixture rich in a desired constituent of low b.p. (*e.g.*, He) which has been obtained as residual vapour from a previous liquefaction is described. The gaseous mixture is subjected to cold exchange with a bath of liquefied refrigerant (*e.g.*, N<sub>2</sub>) which has been obtained by compression, heat exchange with itself, partial expansion through an engine, and further cold exchange between the expanded and unexpanded portions; the undesired constituents of the mixture are liquefied out and returned to the original liquefier.

B. M. VENABLES.

**Means for producing and storing non-oxidising gases.** W. J. WILLENBORG (B.P. 352,006, 19.2.30. U.S., 19.2.29).—The gases comprise the exhaust from an internal-combustion engine and are pumped to a storage vessel by a pump cylinder combined with the engine. Electrical controls are provided so that when the gases contain an insufficient proportion of CO<sub>2</sub> they are pumped to waste.

B. M. VENABLES.

**Carrying out catalytic gas reactions.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 353,622, 6.6.30).—The gases prior to passage over the catalyst proper are caused to take up small quantities of the vapour of alkali or alkaline-earth metals by passing them at a moderate temp., *e.g.*, 400° in the case of K, over the heated metal. The process is applicable to the cracking of methane and the synthesis of NH<sub>3</sub>.

B. M. VENABLES.

**Machines for testing the hardness of materials.** W. & T. AVERY, LTD., and A. H. HANCOX (B.P. 353,729, 21.8.30).—The plunger of a machine of the penetration type is loaded by a lever and wts. A device is described by which the latter can be removed and replaced without shock.

B. M. VENABLES.

**Apparatus for measuring the hardness of materials.** A. F. SHORE (B.P. 353,549, 5.5.30).—In a machine of the penetration type, the pointer of the dial that measures the advance of the plunger is triple, the two branches of the tail making an angle equal to the desired penetration so that readings can be obtained with samples of varying height without resetting to zero.

B. M. VENABLES.

**Determination of the m.p. of materials.** G. A. DE GRAAF, ASSR. to EIMER & AMEND (U.S.P. 1,789,098, 13.1.31. Appl., 27.1.28).—The sample (*e.g.*, coal ash) is placed on an electrically-heated strip of Pt or other suitable metal and the temp. is slowly raised by a

motor-operated sliding rheostat. The sample is observed simultaneously by a visual telescope and a radiation pyrometer. Black-body conditions are maintained by an enclosing shell which may also be gastight so that a vac. or inert atm. may be maintained.

B. M. VENABLES.

**Determining the plasticity of [semi-elastic] substances.** A. E. WHITE. From B. F. GOODRICH Co. (B.P. 353,693, 24.7.30).—A motor running at const. speed closes and opens a set of toggles which at one end are loaded by a spring and at the other bear upon a sample of fixed dimensions, *e.g.*, for rubber, a cylinder of length and diam. 1 cm. each. By this means a rigid material will be subjected to a large load and have small deformation, and vice versa for a soft material, so that the range covered is very wide. One dial reads the max. deformation and hence also the load, another dial reads the recovery on removal of the load.

B. M. VENABLES.

**Apparatus for examining crystalline bodies with the aid of X-rays.** N. V. PHILIPS' GLOEILAMP-ENFABR. (B.P. 351,744, 1.7.30. Holl., 15.7.29).—An apparatus suitable, *e.g.*, for distinguishing between natural and cultivated pearls is described, comprising an X-ray tube, a box for the object, and means for observation both by visual fluorescence and photographically. The whole is covered with material impervious to the rays except through the necessary windows, and safety automatic shutters are provided for the latter.

B. M. VENABLES.

**Agitation and settling apparatus.** W. W. TRIGGS. From DORR Co. (B.P. 351,989, 28.3.30).—See U.S.P. 1,752,789 and 1,752,795; B., 1930, 886.

**[Boiler] furnaces.** A. W. BENNIS (B.P. 353,609, 31.5. and 2.10.30).

**Gas-heated apparatus for heating liquids.** H. JUNKERS (B.P. 353,814, 15.12.30. Switz., 17.4.30).

**Liquid fuel burning apparatus.** A. LANSER (B.P. 353,711, 31.7.30. Luxembourg, 4.7.30).

**Refrigerating apparatus.** FRIGIDAIRE CORP., Assees. of H. F. SMITH (B.P. 353,945, 29.4.30. U.S., 13.9.29).

**Intermittently working refrigerating machines of the absorption type.** AKTIEBOLAGET SVEAEXPORT (B.P. 353,963, 12.5.30. Swed., 12.4.30).

**Evaporator for absorption refrigerating machines.** DEUTS. GASGLÜHLICHT-AUER-G.M.B.H. (B.P. 353,746, 3.9.30. Ger., 3.9.29).

**Heating bituminous matter. Distilling apparatus.**—See II. **Pulp-filtering machine.**—See V. **Drying of clay ware etc.**—See VIII. **Granulation of slag.**—See X. **Precipitating particles from gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Spontaneous electrification in coal-dust clouds.** S. C. BLACKTIN and H. ROBINSON (Safety in Mines Res. Bd., 1931, Paper No. 71, 17 pp.).—Large-scale experiments conducted in the open air have shown that high voltages are produced on well-insulated metal tubes through which a stream of air carrying coal dust

in suspension is passed. The concentration of dust necessary to produce ignition of firedamp is extremely small, and the speed of the air current is well within the limits of mining practice. These effects are obtained only when electrical and atm. conditions are favourable for the accumulation of the electrostatic charges produced by friction, and ducts conveying dust-laden air must possess, therefore, a very high insulation resistance to earth if the electrostatic charge is to be retained on the system. Humidity of the atm. inhibits the production of high voltages on such ducts. A minute leakage to earth is sufficient to eliminate completely the electrostatic charge. Accidental contact of a mine ventilation duct with earth at a number of points, or a single deliberate earth connexion, provides a leakage which will effectively prevent the accumulation of an electrostatic charge on the duct, even though the conditions for electrification may be otherwise favourable.

E. S. HEDGES.

**Evaluation of blast-furnace coke.** A. KILLING (Stahl u. Eisen, 1931, 51, 901—905).—Coke prepared from finely-ground coal (90% through a 3-mm. screen) has a high porosity (over 50%) and a correspondingly low apparent sp. gr. (430 kg. per cu. m.); such coke has given excellent results in the production of Thomas pig iron over a period of several months with an appreciable economy in fuel consumption. The suitability of a coke for blast-furnace purposes does not depend on its combustibility, but is measured by its crushing strength, lightness, and reactivity, the last-named being dependent on its porosity. A. R. POWELL.

**Coke and charcoal. Effect of ash on the reactivity and combustibility.** Y. OSHIMA and Y. FUKUDA (J. Soc. Chem. Ind. Japan, 1931, 34, 238—240 B).—Graphite, coke, and charcoal when finely ground and extracted with HF and HCl show an increase of effective surface, as measured by adsorption of H<sub>2</sub>O vapour. Ash removal increases the reactivity of coke, but reduces that of wood charcoal, to the oxidation of which ash acts as a catalyst. Charcoal ash is much more finely dispersed than ash in coke. The combustibility in air of different carbonaceous materials when freed from ash is almost identical, the physical structure and state of the C having no apparent influence. C. IRWIN.

**Production of active carbons by impregnation methods.** J. MILBAUER (Z. Zuckerind. Czechoslov., 1931, 55, 435—439).—Org. materials impregnated with CaCl<sub>2</sub> solution were dried, powdered, and heated in graphite crucibles at const. temp.; the resulting carbons were boiled with HCl, washed, dried, powdered, and tested on molasses solution. No differences in decolorising power resulted from variations of 2:1 to 6:1 in the ratio of CaCl<sub>2</sub> to org. matter, on dry basis. With ratio 4:1 and temp. 500°, the optimum period of heating was in most cases 3 hr. With ratio 4:1 and period 3 hr., the optimum temp. for starch, peat, molasses, cacao shell, and nut shell was 500°, for sawdust 700°, and for horn meal and pure cellulose still higher. Under optimum conditions the most active carbons were obtained from pure cellulose and sawdust.

J. H. LANE.

**Two-step ash-pan for rotary gas producer.** PRADEL (Chem.-Ztg., 1931, 55, 581).—The usual type of ash-pan involves an accumulation of clinker around the edges and an unequal distribution of the fire within the generator. The pan described has sides inclined outwards with a ledge halfway up which delivers the ashes in two stages and obviates this. Works' trials have shown that generators fitted with it need cleaning much less frequently, less poking, and fewer attendants.

C. IRWIN.

**Dilution of straight coal [gas] by producer gas externally generated.** A. L. HOLTON (Gas J., 1931, 195, 213—215).—Data showing the effect of dilution on three qualities of straight coal gas with different diluents are tabulated. Trefois producer gas (B., 1931, 5) has proved a suitable diluent for reducing the calorific val. of horizontal-retort gas from about 600 to 450 B.Th.U. The inclusive cost of producing Trefois gas at the Partington works over a year is estimated at 1.35d. per 1000 cu. ft. of average calorific val. 133 B.Th.U.

A. B. MANNING.

**Dilution of straight coal gas by steaming in intermittent chamber ovens.** G. DIXON (Gas J., 1931, 195, 215).—The ovens were of the inclined type, with three steaming points in the floor of each. The charge was 4½ tons. On a 16-hr. schedule carbonisation was completed in 11½ hr. and steaming was then operated for 4½ hr. prior to discharge. Steam was admitted to the ovens at 26 lb./sq. in. pressure, superheated to 250°. The calorific val. of the straight coal gas was 550 B.Th.U., that of the diluted gas 496 B.Th.U. Steaming raised the yield from 12,727 to 16,178 cu. ft./ton. The net cost of the gas into the holder was 2.67d. per therm.

A. B. MANNING.

**Ignition and propagation velocity in acetylene-oxygen mixtures. Striking back of the welding torch.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 52—60; Chem. Zentr., 1931, i, 2418).—With a cooled jet the velocity of inflammation markedly increases with increase in O<sub>2</sub> content; with a jet temp. rising to 400° the velocity change is relatively small (3—10%). The higher is the thermal conductivity of the jet, the lower (40—50°) is the temp. of back-firing.

A. A. ELDRIDGE.

**Hydrogenation of bitumen.** E. H. BOOMER and A. W. SADDINGTON (Canad. J. Res., 1931, 4, 517—539).—Experiments on Alberta bitumen indicate that 75% of the latter may easily be converted into gasoline and oil. A cylindrical autoclave of 1.8 litres capacity and designed for close temp. control and efficient agitation is described.

W. GOOD.

**Hazards in catalytic hydrogenation at elevated temperatures and pressures.** H. ADKINS (J. Amer. Chem. Soc., 1931, 53, 2808—2809; cf. A., 1931, 919).—Commercial-scale hydrogenations require adequate preliminary investigations and properly designed bombs.

J. G. A. GRIFFITHS.

**Determination of olefine, aromatic, and paraffin hydrocarbons in neutral oil from coal tar.** E. B. KESTER and W. D. POHLE (Ind. Eng. Chem. [Anal.], 1931, 3, 294—297).—Methods previously available are



briefly discussed. In the suggested method 100 c.c. of neutral oil are shaken with 300 c.c. of 80%  $\text{H}_2\text{SO}_4$  for 5 min., set aside for 30 min., and the acid is drained off; after a further 30 min., any additional acid which separates is also drained off. The reduction in vol. is recorded ( $R_1$ ). The oil is then distilled to  $5^\circ$  higher than the end-point of the untreated oil. To the vol. of distillate found a small correction factor is added, computed in earlier runs from the total vol. distilled minus the total vol. of distillate and residuum. The sum of the vol. reduction on distillation, computed to the whole oil basis, and  $R_1$  gives % olefines. 20 c.c. of the distillate are shaken with 60 c.c. of 98%  $\text{H}_2\text{SO}_4$  for 5 min. and allowed to settle as in the 80%-acid treatment. From the contraction 0.19 c.c. is deducted to allow for the solubility of the paraffins in the acid, the corrected contraction, computed to the whole oil basis, then giving the aromatic content. By this procedure mixed-type compounds, e.g., styrene and indene, are included in the olefines. Satisfactory results are obtained with synthetic mixtures. The results of the analyses of a number of tar neutral oils are tabulated. A. B. MANNING.

**Analysis of gaseous hydrocarbons. A short-cut method.** R. ROSEN and A. E. ROBERTSON (Ind. Eng. Chem. [Anal.], 1931, 3, 284—289).—The method, which has been worked out primarily for mixtures of  $\text{C}_2\text{H}_6$ ,  $\text{C}_3\text{H}_8$ , and  $\text{C}_4\text{H}_{10}$ , consists in distilling the liquefied gas at const. pressure, and comparing the distillation curve, i.e., the curve in which vol. distilled is plotted against temp., with standard curves obtained from known mixtures. Standard curves covering a range corresponding with the composition of certain refinery gases are given. The influence of the presence of  $\text{C}_2\text{H}_4$ , propylene, and isobutane on the form of the standard curves has been studied, and a correction curve for use with mixtures containing those constituents is given. The apparatus and technique are simple; only 200—250 c.c. of liquid  $\text{N}_2$  are required for an analysis, which can be completed in 60—75 min. A. B. MANNING.

**Analysis of light oils from low- and intermediate-temperature carbonisation.** E. B. KESTER and C. R. HOLMES (Ind. Eng. Chem. [Anal.], 1931, 3, 292—294).—The crude oil is refined by shaking with  $\text{H}_2\text{SO}_4$  (d 1.84), 70 c.c. of acid per litre of oil, to remove olefines, and is then steam-distilled. The refined oil is fractionated under a column (cf. "Gas Chemists' Handbook," p. 402—3) and cuts are taken as follows: (1) to  $79^\circ$ , pre- $\text{C}_6\text{H}_6$ , (2)  $79$ — $81^\circ$ ,  $\text{C}_6\text{H}_6$ , (3)  $81$ — $109^\circ$ ,  $\text{C}_6\text{H}_6$ —PhMe, (4)  $109$ — $111.5^\circ$ , PhMe, (5)  $111.5$ — $137^\circ$ , PhMe—xylene, (6) above  $137^\circ$ , residuum. The aromatic content of each fraction is determined by sulphonation of a portion (20 c.c.) with 3 vols. of 98%  $\text{H}_2\text{SO}_4$ . To determine the vol. of each aromatic hydrocarbon in those fractions where two are present, 30 c.c. of the fraction are subjected to a distillation test (*loc. cit.*, p. 406), and the % PhMe is found directly from curves obtained with known mixtures. When paraffins are present the % read is modified by multiplying by the % of aromatics in the fraction, the assumption being made that the paraffins are equally distributed throughout the boiling range of the fraction. Satisfactory results are obtained with synthetic mixtures. The results of analyses of light oils from the carbonisa-

tion of two American coals over the temp. range  $500$ — $1100^\circ$  are tabulated.

A. B. MANNING.

**Determination of small amounts of benzene vapours in air.** H. F. SMYTH (J. Ind. Hygiene, 1931, 13, 227—230).—The method described previously (B., 1930, 265) is satisfactory for 30 p.p.m. An accuracy of over 85% is claimed for determinations down to this concentration in presence of not more than three times as much PhMe.

R. K. CALLOW.

**Determination of softening point as control method for evaluation of paraffin [waxes].** E. DAWIDSON and S. VON PILAT (Allgem. Oel- u. Fett-Ztg., 1931, 28, 261—263).—The range of the components of a paraffin wax may be gauged from the softening point; the smaller the difference between this and the setting point (Shukoff) the sharper has been the fractionation of the wax. Thus waxes of different composition, but with the same setting point, can be differentiated. A modification of the ring-and-ball method for determining softening point is detailed, by which reproducible results are readily obtained. For mixtures such as paraffin wax the softening point is always below the setting point; for pure substances it is slightly above the latter.

E. LEWKOWITSCH.

#### PATENTS.

**Manufacture of peat briquettes.** L. RUDEMAN (B.P. 352,542, 11.4.30).—The moisture in the peat is first reduced to 40—60%, then the peat is broken up, mixed with pitch in a cold state, impregnated with phenols, subjected to superheated steam while still being agitated, and finally pressed into briquettes which are waterproof.

B. M. VENABLES.

**Manufacture of fuel briquettes.** W. W. WHITE and H. D. PONTER (B.P. 352,444 and 352,556, [A] 3.1.30, [B] 17.3.30).—Carbonaceous briquettes are stoved in a current of non-oxidising gas so as to maintain a clear atm. to permit the transmission of radiant heat, the temp. in (A) being  $400$ — $600^\circ$  and in (B)  $500$ — $600^\circ$ . In (A) the stoved briquettes may be dipped in Na silicate and subsequently subjected to the direct action of a Bunsen flame. To produce a luminous flame, or a heavy ash, a Na salt or Fe (and/or Mn) oxide may be incorporated respectively. In (B) the atm. from the retort may be cooled to recover volatile matter and the inert gas re-used; coke breeze may be incorporated in order to prevent adjacent briquettes from sticking.

B. M. VENABLES.

**Coke ovens and the like.** C. STILL (B.P. 351,872, 27.10.30. Ger., 28.10.29).—A tubular apparatus for leading away the gases and vapours from the interior of the charge comprises an upper tube supported by the roof of the oven and a lower tube supported by the charge. The upper tube can be withdrawn from the oven, the lower tube being left in the charge and discharged with the coke. The upper tube opens into a box, for collecting the gas, vapours, and their condensates, which communicates by means of a common collecting pipe for the oven with the collecting main of the battery.

A. B. MANNING.

**Coking retort ovens.** KOPPERS Co., Assees. of J. BECKER (B.P. 353,473, 24.5.30. U.S., 28.1.30).—When producing coke requiring a long coking period, e.g.,

foundry coke requiring 24–30 hr., the decreased heating rate leads to unequal heat distribution. This is avoided by periodically and alternately supplying fuel gas and air at max. and min. rates which are equally removed from the average rate required for the coking time. The gas and air supplies are automatically controlled.

D. K. MOORE.

**Heating systems for coke ovens.** KOPPERS CO., Assees. of J. BECKER (B.P. 352,383, 3.1.30. U.S., 19.2.29).—A series of retort-type ovens is fired almost entirely with blast-furnace gas, but enough coke-oven gas is added to maintain the calorific val. of the mixture at the max. value of blast-furnace gas alone. The mixture is continuously tested and its thermal val. controls the admixture. [Stat. ref.] B. M. VENABLES.

**Coke-oven doors.** A. L. MOND. From VEREIN. STAHLWERKE A.-G. (B.P. 353,741, 29.8.30).—Heat radiation losses from coke-oven doors are reduced by covering the outside of the doors with a layer of insulating material, e.g., sterchamol brick (analysis given) about 80 mm. thick.

D. K. MOORE.

**Manufacture of coke.** R. A. MOTT, R. V. WHEELER, and NAT. FEDERATION OF IRON & STEEL MANUFRS., LTD. (B.P. 351,546, 27.3.30).—The coal to be carbonised is blended with up to 12% of powdered fusain, material rich in fusain, wood charcoal, low-temp. or high-temp. coke. The particles of the added material should be not more than  $\frac{1}{16}$  in., and preferably not more than  $\frac{1}{32}$  in., in size.

A. B. MANNING.

**Low-temperature carbonisation and distillation of coal, lignite, and oil shale.** "INTERTRUST," COMP. GÉN. DE DISTILLATION À BASSE TEMP. ET MINÈRE SOC. ANON., Assees. of P. BENU (B.P. 351,552, 27.3.30. Belg., 6.4.29).—The apparatus consists of a succession of distillation chambers, preferably rectangular, each containing a number of vertical retorts rigidly connected with each other and adapted to be lifted bodily with their charges out of the chamber and transferred to a cooling chamber before being discharged. The retorts are provided with central, perforated tubes for leading away the volatile distillation products. Between the distillation chambers are combustion chambers, the hot gases from which can pass into the former and circulate round the retorts therein. An assembling plate provided with holes into which the tops of the retorts fit closes the top of the distillation chamber in a gastight manner, and an outer cover is provided which forms with the assembling plate a collecting chamber for the distillation gases and vapours. This communicates through a seal and a valve with a collecting main. The chambers are directly heated in turn; the hot gases from a directly heated chamber are circulated through the subsequent chambers up to the next directly-heated chamber, so that the temp. of the charges is gradually raised until the corresponding chamber is in turn directly heated.

A. B. MANNING.

**Manufacture of water-gas of regulated nitrogen content.** POWER GAS CORP., LTD., N. E. RAMBUSH, and J. M. BALLENGALL (B.P. 353,522, 26.4.30).—The cycle of operations consists of two up-runs, one lasting for 2 min. and the other for 1 min., one down-run (2 min.), and one blow (1 min.). Precautions are taken to prevent

the admixture of air and gas. A flow-meter on the gas outlet pipe operates a valve controlling the air supply, thus regulating the  $N_2$  content of the gas. A servomotor is provided to operate the valves, some of which are interlocked, and also a delay-action device.

D. K. MOORE.

**Manufacture of producer gas.** K. KOLLER and Z. GALOCSY (B.P. 351,892, 26.11.30. Hung., 28.11.29).—The process is carried out continuously in two stages. In the first stage a solid, liquid, or gaseous fuel is burned with  $O_2$ , air, or mixture thereof, in a combustion chamber which is in direct communication with the gas producer proper. The combustion gases and excess  $O_2$  are mixed with saturated or superheated steam, and in the second stage of the process the mixed gases are passed through the incandescent fuel column of a producer operating with molten slag.

A. B. MANNING.

**Distillation of tar.** BARRETT CO., Assees. of S. P. MILLER (B.P. 351,388—9, 17.1.30. U.S., 26.1.29. Cf. B.P. 347,240; B., 1931, 663).—(A) The tar is heated in an externally-heated still, preferably a pipe-coil still, by indirect contact with hot waste gases, to a temp. above the b.p. of part of the oils contained therein, and the preheated tar is then further distilled by being brought into direct and intimate contact with hot coal-distillation gases. (B) Tar preheated as in (A) is discharged into a vapour box where the lower-boiling constituents are volatilised, the vapours being conducted to suitable condensing means. The residue is then brought in a preheated state into direct and intimate contact with hot fuel gases to effect further distillation of the tar.

A. B. MANNING.

**Apparatus for heating tar or bituminous matter liquefiable by heat.** H. L. DALE (B.P. 352,623, 10.5.30).—The apparatus comprises a horizontal, open-ended, steam-jacketed cylinder surrounded by another cylindrical or prismatic casing with closed ends and so perforated that the material flows longitudinally over the outside and through the inside of the heating jacket. The above apparatus is placed in a larger tank containing the material.

B. M. VENABLES.

**Preparation and treatment of aqueous emulsions of asphalt and the like, particularly for road-making.** METALLGES. A.-G. (B.P. 353,702, 28.7.30. Ger., 16.8.29).—Asphalt, emulsified in  $H_2O$  by the addition of  $Na_3PO_4$ , is flocculated by adding alkaline-earth salts, especially  $CaCl_2$ .

D. K. MOORE.

**Catalytic hydrogenation of hydrocarbon oils.** STANDARD OIL DEVELOPMENT CO., Assees. of R. P. RUSSELL and M. W. BOYER (B.P. 353,792, 16.10.30. U.S., 17.10.29).—Heavy oils are hydrogenated by heating to 400–470° and passing with  $H_2$  under pressure through a drum containing metallic oxides as catalysts. After a time the activity of the catalyst falls, and is restored by passing through the drum a light hydrocarbon oil, e.g., kerosene at above 400°, and  $H_2$  at a pressure above 20 atm.

D. K. MOORE.

(A) Treatment of carbonaceous materials with hydrogen in the presence of catalysts. (B) Refining of liquid carbonaceous materials such as petroleum products and residues, asphalt, etc.

H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 353,506—7, 25.4.30).—(A) Destructive hydrogenation of materials such as coal, the removal of O from org. materials, *e.g.*, PhOH, and the desulphurisation of petroleum products is effected by heating them to 460° in H<sub>2</sub> at 100 kg. per sq. cm. pressure in the presence of catalysts consisting of colloidal metallic compounds, *e.g.*, Mo oxide, adsorbed by carriers, *e.g.*, pumice, SiO<sub>2</sub> gel, metal hydroxides. (B) For the desulphurisation of petroleum oils the catalyst consists of colloidal Cr compounds adsorbed by active C, with or without colloidal Mo compounds adsorbed by active C.

D. K. MOORE.

**Refining of hydrocarbons.** E. W. ISOM and E. B. PHILLIPS, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,786,493, 30.12.30. Appl., 27.3.29).—A solid, absorbent, refining agent is suspended in a crude petroleum stock and the mixture passed rapidly through a heating zone, where it is heated to not above 400° but below cracking temp., and is discharged into a vaporising and digesting zone from which the vapours are removed. A substantial supply of liquid is maintained in this zone with the solid absorbent in suspension at a temp., approx. 326°, such that the residual product discharged therefrom has a Saybolt viscosity of > 100 at 99°. The refined viscous oil product is discharged after remaining in the vaporising and digesting zone for not less than 5 times as long as the raw absorbent stock mixture is in the heating zone.

H. S. GARLICK.

**Treatment of [hydrocarbon] oil.** D. G. BRANDT, ASSR. to DOHERTY RES. Co. (U.S.P. 1,784,087, 9.12.30. Appl., 31.1.22. Renewed 11.7.28).—Oil is pumped through a series of surface condensers, where it is heated to 204—232° by vapours passing in a countercurrent direction, to a dephlegmator maintained under approx. 100 lb. pressure, and thence into a cracking chamber in which is maintained a column of oil. As the mixture of incoming and residual oil passes downwardly through the cracking chamber, part is cracked and part vaporised, the vapours passing up through the oil into the dephlegmator. The residual oil is conducted to the lower drum of a cracking still in which a substantially fixed oil level is maintained and from which it passes rapidly through small tubes, located in a heating furnace, to a vapour-separating drum. The vapours are introduced into the lower portion of the cracking chamber and distributed through the oil therein by means of baffles and trays. As the oil is cracked and the lighter vapours are released, C and tarry matter is liberated and works downwardly over the baffles, finally accumulating in a chamber in the bottom of the cracking chamber, from which it may be drawn off.

H. S. GARLICK.

**Purification of hydrocarbon oils.** C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,787,570, 6.1.31. Appl., 29.6.25. Renewed 5.10.28).—Sulphurous compounds are removed from hydrocarbon distillates by passing the latter through a zone containing aq. Ca(OH)<sub>2</sub>, wherein Ca(OH)·SH is formed as a reaction product. The distillate is passed to a second zone similar to the first and is then returned to the first zone, wherein CS<sub>2</sub> contained in the distillate will react with the above compound. The distillate is thereafter

treated in an independent zone with Ca(OH)<sub>2</sub> and H<sub>2</sub>O to remove H<sub>2</sub>S therefrom.

H. S. GARLICK.

**Sweetening of sulphurous petroleum.** H. H. CANNON and W. W. GARY, ASSRS. to CANNON-PRUTZMAN TREATING PROCESSES, LTD. (U.S.P. 1,789,168, 13.1.31. Appl., 21.1.29).—The oil is treated with a powdered solid comprising an alkaline-earth hydroxide, *e.g.*, Ca(OH)<sub>2</sub>, and PbO, intimately mixed together, to convert the mercaptans into Pb mercaptides, and thereafter the Pb is precipitated from the mercaptides by adding S to the oil.

H. S. GARLICK.

**Treatment of petroleum oils containing sulphur.** G. J. ZISER and J. H. OSMER, ASSRS. to STANDARD OIL COMPANY OF CALIFORNIA (U.S.P. 1,784,215, 9.12.30. Appl., 9.1.26).—The oil is brought into intimate contact with an aerated dil. aq. solution of an ammoniacal Cu salt.

H. S. GARLICK.

**Treatment of crude [petroleum] oil.** K. W. HICKEY (U.S.P. 1,789,233, 13.1.31. Appl., 11.1.29).—A solution of NaHCO<sub>3</sub> (6 lb.) and borax (1 lb.) in H<sub>2</sub>O (8 gals.) is added to crude oil (250 barrels) simultaneously with HCl (14 oz.), to produce a violent reaction which causes precipitation of foreign matter and H<sub>2</sub>O.

H. S. GARLICK.

**Gum inhibitor for hydrocarbons.** W. S. CALCOTT and I. E. LEE, ASSRS. to E. I. Du PONT DE NEMOURS & Co. (U.S.P. 1,789,302, 20.1.31. Appl., 23.8.29).—The use of an acyl derivative of an arylamine of the type R·NH·CO·R', where R is an aryl group which may contain alkyl or alkoxy-substituents and R' is an alkyl, aryl, or aralkyl group, is claimed. Acetanilide is specially mentioned.

H. S. GARLICK.

**Refining of lubricating oils.** J. C. BLACK and W. D. RIAL, ASSRS. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,787,505, 6.1.31. Appl., 27.4.27).—Lubricating oil distillate from the vac. distillation of asphalt-base crude petroleum oil is treated with aq. NaOH, under pressure > 1 atm. and at approx. 137°, in quantities sufficient to combine with the emulsifying constituents. The mixture is allowed to separate and the oil is treated with sufficient H<sub>2</sub>SO<sub>4</sub> (*d* 1.83) to combine with all the unsaturated hydrocarbons. After separating the acid sludge the oil is treated at above 93° with a solid absorbent earth in order to absorb all the sulphonic acid constituents. The lighter grades of lubricating oil stocks are separated from the heavier oils by distillation in vac. and are finally clarified and decolorised as desired.

H. S. GARLICK.

**Increasing the viscosity and improving the lubricating properties of oils.** W. PUNGS and E. FRESE, ASSRS. to STANDARD I.G. Co. (U.S.P. 1,787,951, 6.1.31. Appl., 13.6.28. Ger., 25.6.27).—The addition of small quantities (0.25—0.75%) of montan wax bleached by oxidation with CrO<sub>3</sub> or salts thereof in the presence of H<sub>2</sub>SO<sub>4</sub> or org. acids considerably increases the viscosity of mineral oils, whilst the addition of larger quantities gives products of the consistency of petroleum jelly.

H. S. GARLICK.

**Lubrication of textile machinery.** A. E. BECKER, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,789,331, 20.1.31. Appl., 20.9.26).—A lubricant that is

easily washed out of the fabric being manufactured comprises a petroleum oil, preferably having a viscosity between 50 and 250 sec. Saybolt at 38°, containing 0.15–1.5% of  $H_2O$ -sol. alkali-metal soap.

B. M. VENABLES.

**Stabilisation of mineral oils and greases, animal and vegetable oils, and fats and soaps.** R. T. VANDERBILT CO., INC., Assees. of P. I. MURRILL (B.P. 353,874, 24.4.30. U.S., 4.3.30).—Small amounts of a phenoxide prepared from a phenylphenol and a base that is itself an antioxidant (e.g., 4-hydroxydiphenyl and diphenylethylenediamine) are added.

E. LEWKOWITSCH.

**Manufacture of concentrated emulsions of oil and water.** C. E. OLIVER (U.S.P. 1,790,070, 27.1.31. Appl., 5.4.26).—Solutions of 10 lb. of rosin in 5 gals. of oil, and 10 lb. of casein in 10 gals. of  $H_2O$  containing 1 lb. of NaOH, having approx. equal surface tension, are mixed together (1:1) to form a conc. emulsion, which may be diluted by the alternate addition of  $H_2O$  and oil, preferably in the proportions of 1:3. H. S. GARLICK.

**Coke pushers, coke levellers, and door extractors for use in coke ovens.** WELLMAN SMITH OWEN ENG. CORP., LTD., and S. STOCKDALE (B.P. 353,879, 28.4.30).

**Pressure gas burners for furnaces.** F. WOLFF, and SELAS A.-G. (B.P. 354,072, 19.8.30).

**Gas-detecting lamps.** R. J. PLUMMER, and CEAG, LTD. (B.P. 353,454, 22.4.30).

**Combustion apparatus for liquid fuels.** T. K. A. NORDENSSON (B.P. 353,457, 23.4.30. Swed., 24.4.29).

**Sorting of coal etc. Mixing phenol compounds. Centrifuging salts. Pipe still. Distillation control. Dephlegmator for vapours. Carrying out catalytic reactions. Determining m.p. [of coal ash etc.].—See I. Impregnating timber.—See IX. Coke-oven effluents.—See XXIII.**

### III.—ORGANIC INTERMEDIATES.

**Synthesis of aliphatic alcohols from mixtures of carbon monoxide and hydrogen.** G. NATTA and M. STRADA (Giorn. Chim. Ind. Appl., 1931, 13, 317–325).—When heated under pressure in presence of compounds of Zn and of alkali metals, mixtures of CO and  $H_2$  containing high proportions of the latter yield almost entirely mixtures of aliphatic alcohols in large part higher than MeOH. The use of catalysts free from elements of groups IV–VIII renders insignificant the production of harmful secondary reactions, especially the formation of  $CH_4$ . At temp. not above 400–420° the formation in the catalysts of alkali salts of fatty acids as intermediate compounds is confirmed. Particularly high catalytic activity is shown by mixtures of ZnO and alkali salts of fatty acids. When reduced by  $H_2$  in presence or absence of ZnO  $HCO_2K$  gives MeOH almost quantitatively; KOAc gives EtOH (from 26.2% of the salt decomposed),  $Pr^iOH$ , and  $COMe_2$  (from 31.8%);  $PrCO_2K$  gives *n*- and *iso*- $PrOH$ .  $K_2CO_3$  is reducible to  $HCO_2K$  by  $H_2$  in presence of ZnO. Confirmation is thus furnished of the hypothesis that the primary product of these syntheses from CO and  $H_2$  is MeOH, and that the higher alcohols are produced subsequently by interaction of MeOH and CO (in

presence of alkaline catalysts) to form firstly fatty acids (or their alkaline salts). These undergo either reduction or condensation (2 mols. of the acid), with elimination of  $CO_2$ , to ketones or aldehydes, which are then hydrogenated to give alcohols.

T. H. POPE.

**Analysis of mixtures of methyl and ethyl alcohols.** L. MARICQ (Bull. Soc. chim. Belg., 1931, 40, 333–337).—The total vol.-% of the alcohols is determined from the density, whilst conditions are described under which oxidation with  $K_2Cr_2O_7$  and  $H_2SO_4$  converts MeOH into  $CO_2$  and  $H_2O$ , and EtOH into AcOH. Combination of the results permits the determination of each alcohol. The process is recommended for the determination of MeOH in spirits.

A. A. LEVI.

**Recovery of volatile solvents from the viewpoint of modern adsorption methods.** W. HERBERT (Chem.-Ztg., 1931, 55, 577–578, 595–597, 615–617).—The recovery of volatile solvents by condensation alone is usually inefficient and the fire risk is considerable. In the case of methods of recovery involving absorption in a less volatile solvent with, in many cases, formation of a mol. compound, the difficulty is rather in the economical recovery of the absorbed solvent. The Bréguat process, using cresols, is one of the best, but is only advantageous with solvents containing O, as with hydrocarbons etc. no mol. compound is formed. Washing processes are suitable for high and const. vapour concentrations. Capital costs are usually high for small-scale plants. The third class of methods include adsorption on activated C,  $SiO_2$  gel, or  $Al(OH)_3$  gel. The properties of these adsorbents are functions of their enormous surface, activated C having been prepared with a surface of 1250 sq. m./g. or above 30% of that of a sheet 1 mol. thick. Their use is particularly advantageous for the recovery of volatile solvents present only in traces. Vegetable charcoal is heated with  $ZnCl_2$  to 500° or with steam and oxidising gases to 800–1000°. The Bayer adsorption process is described. The use of superheated steam for stripping has been abandoned as no appreciable benefit is obtained. The intermittent working of 2 units alternately is no disadvantage, and the process is now very widely used.

C. IRWIN.

**Volatility of cresols when heated on the water-bath.** N. J. STEKELENBURG (Pharm. Weekblad, 1931, 68, 719–724).—The loss of wt. of cresol, isolated from liquor cresoli saponatus by the Dutch Pharmacopœia method, during heating on the water-bath in Erlenmeyer flasks of various dimensions, has been determined. With short- and wide-necked flasks the loss may amount to 80% within 30 min., and even under good conditions to 2–5%.

H. F. GILLBE.

**Apparatus for experimental pyrolytic production of diphenyl.** A. W. HIXSON, L. T. WORK, H. V. ALESSANDRONI, G. E. CLIFFORD, and G. A. WILKENS (Ind. Eng. Chem. [Anal.], 1931, 3, 289–291).—The influence of temp., pressure, and rate of flow on the production of Ph·Ph when  $C_6H_6$  vapour is bubbled through a bath of molten Pb has been studied. The optimum temp. is about 740°. Pressures above 1 atm. favour the conversion into Ph·Ph. The % conversion decreases with increasing rate of flow, but less rapidly

under atm. than under higher pressures. The chemical efficiency of the process is high and may reach 98%. With the apparatus described, in which the vapour is bubbled through a column of Pb 17 in. high, the rate of production under atm. pressure and at moderate rates of flow was about 1.4 g./hr., whilst under higher pressures and with somewhat greater rates of flow the rate reached 50—75 g./hr.

A. B. MANNING.

**The indanthrone [alkali] melt.** V. Effect of water. VI. Catalytic action of the isomeric cresols. T. MAKI (J. Soc. Chem. Ind. Japan, 1931, 34, 249—253 B, 253—257 B; cf. B., 1931, 105).—V. In the fusion of 2-aminoanthraquinone with KOH in absence of PhOH the yield of indanthrone first rises and then falls as the H<sub>2</sub>O present decreases, the max. (15.6%) being reached at 19% H<sub>2</sub>O; there is a continuous fall in alkali-sol. product (alizarin) and rise in PhNO<sub>2</sub>-sol. (2-aminoanthraquinone). In the presence of PhOH (42% of wt. of 2-aminoanthraquinone) similar effects are observed, but the yield is much higher (51.5% max. at 19% H<sub>2</sub>O). As the PhOH is increased in proportion to the 85% KOH, a max. yield (51.6%) is reached at 15% (60% of the 2-aminoanthraquinone) at the expense of the PhNO<sub>2</sub>-sol. product; excess of PhOH acts as a diluent.

VI. *o*- and *p*-Cresols have about equal effects, the max. yields being 51.5% and 48.5% with 17.5% of *o*- and *p*-cresol, respectively (on the 85% KOH), and 44.4% with 25% of *m*-cresol; the amount of alkali-sol. product increases with the proportion of cresols more rapidly than with PhOH.

C. HOLLINS.

**Analysis of gaseous hydrocarbons.**—See II. **Syntheses under pressure.**—See VII. **Glycerin analysis.**—See XII. **Trinitronaphthalene.**—See XXII.

## PATENTS.

**Synthesis of oxygenated aliphatic compounds [from carbon monoxide and steam].** SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 349,193, 18.3.30. Fr., 28.1.30).—Mixed catalysts containing a rare-earth (except Ce) metal or compound, at least one alkali or alkaline-earth compound, and at least one Cu, Mn, Zn, Cd, Pb, V, Cr, Mo, Sb, Bi, B, Al, W, Si, Ti, Zr, or Th metal or compound, are used, with or without Ce, for conversion of CO and H<sub>2</sub>O into aliphatic oxygenated compounds below C<sub>7</sub>. Commercial Ce(CO<sub>3</sub>)<sub>2</sub> assaying 60% Ce, 30% Di, is dissolved with ZnCO<sub>3</sub> and Ba(OH)<sub>2</sub> in AcOH, Al(OAc)<sub>3</sub> is added, and after addition of NaOH the ppt. is washed with Ba(OH)<sub>2</sub> solution and dried at 100°. This catalyst gives 40% conversion at 350° with a space velocity of 8000.

C. HOLLINS.

**Production of vinyl halides [from acetylene and hydrogen halides].** J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,017, 12.2.30).—The active C catalyst is activated by preheating with the hydrogen halide at higher temp. (200—600°) than that used for reaction (100—300°). The catalyst may similarly be reactivated.

C. HOLLINS.

**[Preservation of] oxidisable organic compounds.** GOODYEAR TIRE & RUBBER CO., ASSEES. OF A. M. CLIFFORD (B.P. 352,652, 24.5.30. U.S., 27.8.29).—Oxidisable

substances such as rubber, transformer oils, and aldehydes are preserved by incorporating the reaction product of an aromatic amine, a naphthol, and CH<sub>2</sub>O. The reaction products yielded by aniline, *o*-toluidine, and  $\beta$ -naphthylamine with  $\beta$ -naphthol are cryst. and have m.p. 132—133°, 185—186°, and 187—189°, respectively. Their formula is C<sub>10</sub>H<sub>6</sub>(OH)·CH<sub>2</sub>·NHR, where R is an aromatic radical.

D. F. TWISS.

**Temp. control. Refrigerants.**—See I.

## IV.—DYESTUFFS.

## PATENTS.

**Manufacture of [acid] wool dyes.** I. G. FARN-ENIND. A.-G. (B.P. 348,902, 30.5.30. Ger., 17.8.29).—A 2:6-diamino-derivative of a sulphonated secondary amine of the C<sub>6</sub>H<sub>4</sub> series is condensed with a 2:4:6-trinitrobenzene containing a replaceable group (Cl, OMe) in position 1; the diphenylamine first formed loses NaNO<sub>2</sub> on prolonged heating in aq. NaOH, giving yellow-brown to violet-brown acid dyes. Examples of starting materials are: 2:6-diaminodiphenylamine-4-sulphonic acid with 2:4:6-trinitroanisole; 2:6-diamino-*n*-butylaniline-4-sulphonic acid with 1-chloro-2:4:6-trinitrobenzene; 4-chloro-2:6-diaminodiphenylamine-3'-sulphonic acid or 4-(2':6'-diamino-4'-sulpho-anilino)antipyrine with 2:4:6-trinitroanisole.

C. HOLLINS.

**Manufacture and application of stable diazo preparations.** SOC. CHEM. IND. IN BASLE (B.P. 349,339 17.6.30. Switz., 17.6.29).—The diazo compound is precipitated as arylsulphonate in presence of a salt of a metal of group II. Examples are: diazo compounds from 2:5-dichloroaniline or *o*-anisidine with Na 2:7-naphthalenedisulphonate and MgCl<sub>2</sub> or CaCl<sub>2</sub>; *o*-chloroaniline with Na 1:5-naphthalenedisulphonate and MgCl<sub>2</sub>. The products may be used in dyeing with ice colours.

C. HOLLINS.

**Diazo-type layers.**—See XXI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Yolk in New Zealand wool.** I. Effect of sheep covers on yolk production. W. G. SUTTON (J. Text. Inst., 1931, 22,  $\tau$  365—369).—The production of yolk (wool grease) by the skin is not significantly affected by protecting the sheep from rain etc. by means of light waterproof covers. A method for determining the constituents of greasy wool, which involves extraction with both Et<sub>2</sub>O and H<sub>2</sub>O, is described. B. P. RIDGE.

**Sulphur content of some New Zealand wools.** D. J. SIDEX (J. Text. Inst., 1931, 22,  $\tau$  370—373; cf. A., 1931, 1081).—In the few but strictly comparable cases examined, better spinning wool was higher in S than lower-class wool, and the S content was also in inverse proportion to the coeff. of variation and the contour figure. It therefore appears that there is a correlation between the spinning properties and the S content of a wool.

B. P. RIDGE.

**Determination of mixtures of textile fibres.** P. KRAIS and MARKERT (Rev. Gén. Mat. Col., 1931, 35, 281—285).—The material containing the mixed fibres is treated with a conc. solution of Ca(SCN)<sub>2</sub> for

1 hr. at 70°, whereby viscose-, cuprammonium-, acetate-, and nitro-cellulose rayons and natural silk are quantitatively dissolved, whilst raw or mercerised cotton and wool suffer a loss of wt. of only 2–4%. Thorough stirring during treatment is necessary, and the insol. residue is collected by filtration through a metal sieve, washed, dried, and weighed. Good agreement is obtained between the theoretical values for mixtures of known composition and those found experimentally. When used in conjunction with other known procedures, the method permits the analysis of mixtures containing more than two different fibres.

B. P. RIDGE.

**Chemistry of wood. III. I. Determination of lignin.** L. E. WISE and A. M. FAIRBROTHER. **II. Preparation of lignin from wood.** W. M. HARLOW and L. E. WISE (Ind. Eng. Chem. [Anal.], 1931, 3, 253–255).—I. The U.S. Forest Products Laboratory method, involving hydrolysis of the wood with 72%  $\text{H}_2\text{SO}_4$ , gives a uniformly higher lignin fraction from hard woods than does the Ross-Hill procedure using  $\text{CH}_2\text{O}$  and conc.  $\text{H}_2\text{SO}_4$ , but if the material is previously extracted with hot  $\text{H}_2\text{O}$  and  $\text{EtOH}-\text{C}_6\text{H}_6$  the results are in approx. agreement. For coniferous woods the Ross-Hill method gives the higher values. This method is, however, the more rapid and convenient.

II. When the procedures described above are followed, both methods give the same yield of lignin from the same sample of wood.

B. P. RIDGE.

**Action of dilute sodium hydrogen carbonate solution on "oxycelluloses" and denitrated cellulose nitrate.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 24–26; Chem. Zentr., 1931, i, 2412–2413).—The air-dried material (25 g.) was shaken for 15 hr. with 750 c.c. of 5% aq.  $\text{NaHCO}_3$ , the washed residue being 3 times similarly treated. The Cu no. of the product was not that characteristic of cellulose.

A. A. ELDRIDGE.

**Cellulose formate.** G. TOCCO and A. NYSSSENS (Giorn. Chim. Ind. Appl., 1931, 13, 325–330).—Esterification of cellulose by means of  $\text{HCO}_2\text{H}$  in presence of a dehydrating catalyst under good conditions always yields a cellulose diformate which remains in solution and may be separated in the same way as the acetate. Cellulose diformate is highly stable in dry air, but in ordinary air it begins to decompose after some weeks; decomp. is greatly retarded by immersion in  $\text{H}_2\text{O}$  or other neutral liquid. Cross and Bevan's hypothesis that cellulose diformate is the mono-substituted ester of a cellulose previously modified by acid is not confirmed. On evaporation of its solution in  $\text{HCO}_2\text{H}$ , a residue of pure cellulose remains. This behaviour is characteristic of the compound and serves for obtaining a pure cellulose pellicule by evaporation. The solvent properties of  $\text{H}_3\text{PO}_4$ ,  $\text{ZnCl}_2$ ,  $\text{HCO}_2\text{H}$ , readily-sol. thiocyanates, pyridine, etc. for the compound are confirmed. The behaviour of the more important solutions, especially of those in thiocyanates (which follows a cycle of ripening similar to that of viscose) and pyridine, is described.

T. H. POPE.

**Acid values of cellulose fatty acid esters, and rapid analysis of certain cellulose acetates.** T. F. MURRAY, C. J. STAUD, and H. LE B. GRAY (Ind. Eng.

Chem. [Anal.], 1931, 3, 269–273).—Of the more important methods available for the determination of the acetyl value of acetylated cellulose, that due to Eberstadt is considered the most satisfactory, although it requires a long time for the complete saponification of the material. Accurate results for cellulose triacetate are obtained only after a pretreatment with 75%  $\text{EtOH}$ . A more rapid modification of this method involves dissolution of the ester in pyridine before saponification with  $\text{NaOH}$ , and the time is then shortened to about 1 hr. This procedure can be used for materials containing 35.8–44.8% Ac, but the ester must be rendered easily sol. in pyridine by previous dissolution in  $\text{COMe}_2$  and reprecipitation. The results agree with those given by the slower method. Methods are described for the determination of  $\text{HCO}_2\text{H}$  and  $\text{AcOH}$  in the presence of each other and of one or more acids not volatile in steam.

B. P. RIDGE.

**Relation between chemical properties of cellulose nitrate and its suitability for use as a lacquer.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 15–22; Chem. Zentr., 1931, i, 2401).—Heating with  $\text{H}_2\text{O}$  under pressure has a greater action on the cellulose mol. of the product than has treatment with  $\text{NH}_3$ , but gives better lacquers.

A. A. ELDRIDGE.

**Pine oil in the cellulose industry.** W. FERMAZIN (Chem.-Ztg., 1931, 55, 613).—Pine oil—used up to the present chiefly for the manufacture of terpineol—consists principally of the oxidation products of turpentine and has b.p. 208–210°. A sample contained 54% of  $\alpha$ -terpineol, the rest being terpin hydrate, fenchyl alcohol, terpenes, and camphor. A 0.5% addition of pine oil to the size used for an acetate silk yarn improved the strength considerably. A similar effect was produced by an addition of 0.3% to the spinning bath, this proportion being an optimum. Pine oil is a good solvent for nitrocellulose and lacquers prepared with its use are of high quality.

C. IRWIN.

**Oxycellulose.**—See VI. **Stability of cellulose nitrate.**—See XXII.

## PATENTS.

**Dry disintegration and deposition of fibres.** F. W. MANNING, Assr. to FILTER FABRICS, INC. (U.S.P. 1,786,669, 30.13.30. Appl., 23.4.28).—Filtering media, e.g., cellulose fibres, asbestos, decolorising C, are deposited in a more or less dry condition on a fabric support from a current of air or other elastic fluid. Thus scrim passes round a cylindrical suction mould revolving at the upper end of a chamber up which the filtering medium is blown by a current of air, deposition being therefore against the force of gravity with the result that only the finely-divided material reaches the fabric. By inserting vertical partitions in the deposition chamber it is possible to apply successive layers of different filtering media. After leaving the suction mould the filter fabric is wound on a suction drum so that steam or air carrying atomised oil may be sucked through the material to promote felting.

D. J. NORMAN.

**Manufacture of rubber-impregnated fibrous material.** G. A. RICHTER and R. B. HILL, Assrs. to BROWN CO. (U.S.P. 1,787,952, 6.1.31. Appl., 27.11.28).

—Absorbent paper is impregnated with an aq. rubber dispersion, *e.g.*, latex, dried, and passed through a bath containing a rubber solvent to swell the rubber. The material is then rolled and heated to volatilise the solvent. Vulcanising agents and accelerators may be present in the solvent bath. The resulting product is less  $H_2O$ -absorbent and has a higher dry and wet strength than before treatment with the solvent.

D. J. NORMAN.

**Making cellulose acetate.** KODAK, LTD. From EASTMAN KODAK Co. (B.P. 351,118, 2.4.30).—Cellulose acetate is obtained in a fluffy form by including in the acetylation bath an inert,  $H_2O$ -immiscible, volatile solvent and subsequently precipitating the cellulose acetate in  $H_2O$  at or above the b.p. of the volatile solvent. The quantity of  $AcOH$  in the acetylation bath should be insufficient of itself to dissolve the cellulose acetate formed, complete dissolution being effected by adjusting the proportion of volatile solvent present. Suitable solvents are  $C_2HCl_3$ ,  $CHCl_3$ , and particularly  $C_2H_4Cl_2$ .

D. J. NORMAN.

**Acetylation of nitrocellulose.** H. A. GARDNER and C. A. KNAUSS, ASSRS. to H. A. GARDNER LABORATORY, INC. (U.S.P. 1,786,989, 30.12.30. Appl., 12.1.28).—Acetylnitrocelluloses containing 9.6–4.8% N and 20.6–41.2% OAc are obtained by acetylating nitrocellulose containing 1 or more unsubstituted OH groups and about 11% N. The acetylated product is then precipitated with  $C_2H_4Cl_2$  and the excess  $AcOH$  recovered by fractional distillation. Acetylnitrocelluloses so obtained are sol. in  $COMe_2$  and most of the usual solvents, give solutions of low viscosity and good film-forming properties, and are of low flammability.

D. J. NORMAN.

**Manufacture of nitrocellulose.** A. HOUGH, J. R. DUFFORD, and W. C. LEONHARD (U.S.P. 1,785,030, 16.12.30. Appl., 31.8.26).—Parchment paper, preferably made from  $\alpha$ -pulp, is comminuted and nitrated at 40° with, *e.g.*, an acid mixture containing  $H_2SO_4$  60%,  $HNO_3$  21–22%, and  $H_2O$  18–19%. The nitrated product is then washed, boiled with  $H_2O$  for about 3 hr., and then boiled with successive quantities of very dil. aq.  $NH_3$  until no further yellow colour develops. The product may be finally bleached with alkaline  $NaOCl$  solution.

D. J. NORMAN.

**Nitration of cellulose.** L. A. PINCK (U.S.P. 1,784,945, 16.12.30. Appl., 27.6.28).—Nitrating acid is made by passing N oxides, *e.g.*,  $NO_2$  and  $N_2O_4$  as obtained in  $N_2$  fixation and similar processes, into  $H_2SO_4$  until the mol. ratio  $N_2O_4 : H_2SO_4$  is 1:1.25–3.0. When the N oxides are practically free from  $HNO_3$  the concentration of the  $H_2SO_4$  may be 95–90%, but when about 15% of the  $N_2O_4$  is replaced by its equiv. of  $HNO_3$  the concentration of the  $H_2SO_4$  may be 95–85%, the presence of  $HNO_3$  serving to increase the degree of nitration at the same time permitting the use of a less conc.  $H_2SO_4$  without any deleterious effect on the cellulose or the nitrated product. Graphs are given showing the degree of nitration obtained with various acid mixtures. Sufficient nitrating acid should be used to give 15 pts. of  $N_2O_4$  per 1 pt. of cellulose, and a reaction time of 4–5 hr. at 25–30° is suitable. D. J. NORMAN.

**[Production of] pyroxylin compound sheets.** FIBERLOID CORP., ASSEES, of R. G. O'KANE and E. R. DERBY (B.P. 353,045, 9.4.30. U.S., 12.2.30).—Sheets with a smooth, sheeny surface are obtained by extruding a mass of pyroxylin gel containing a solvent through an orifice while under tension, removing part of the solvent by a blast of hot air, and finally while still in a plastic condition subjecting it to progressive transverse stretching.

F. R. ENNOS.

**Manufacture of mixed esters of cellulose.** C. S. WEBBER and C. J. STAUD, ASSRS. to EASTMAN KODAK Co. (U.S.P. 1,785,466, 16.12.30. Appl., 2.8.29).—Cellulose triacetate or the hydrolysed acetates containing <44.8% Ac are treated with aromatic org. acids having an ionisation const. above  $1.82 \times 10^{-5}$ , *e.g.*, *o*-, *m*-, or *p*-toluic acids, benzoic, mandelic, and cinnamic acids. The reaction may take place in any suitable medium, *e.g.*,  $H_2O$ , 1:4-dioxan, propionic acid,  $C_2H_4Cl_2$ , at 100–150°, and may be continued until a mixed ester of the desired solubility up to a  $H_2O$ -sol. product is obtained. The prep. of cellulose acetosalicylate (m.p. 248–271°) and acetomandelate is described.

D. J. NORMAN.

**Pigmentation of organic cellulose esters and ethers.** L. CLEMENT and C. RIVIÈRE (B.P. 350,924, 11.2.30).—The preformed pigment and the cellulose derivative are powdered and stirred together in a liquid medium comprising a solvent for the cellulose derivative in admixture with an equal vol. of  $H_2O$ . After a few hr. a large proportion of the pigment becomes fixed on the cellulose derivative and the latter is then filtered off, washed, and dried. The resulting product gives films in which the pigment grains are invisible. Metal powders may be introduced by this process.

D. J. NORMAN.

**Decolorisation of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 353,075, 23.4.30. U.S., 13.5.29).—The derivatives are treated, either in suspension or in solution, with a solution of polybasic aliphatic acids or their salts (oxalic, citric, tartaric, etc.). F. R. ENNOS.

**Manufacture of transparent or translucent films or foil.** A. H. CHIVERTON, and F.M., LTD. (B.P. 353,136, 15.5.30).—A mixture of fusel and gallipoli oils is applied by means of rollers to the "casting" surface (drum or band) before deposition of the film material (cellulose acetate solution), whereby a uniform film which is readily stripped off is obtained.

F. R. ENNOS.

**Production of artificial filaments or threads.** BRIT. CELANESE, LTD., H. DREYFUS, E. KINSELLA, J. BOWER, and W. I. TAYLOR (B.P. 352,922, 1.1.30. Addn. to B.P. 300,998, 304,674, and 326,232; B., 1929, 203, 353; 1930, 456).—Solutions of org. derivatives of cellulose are dry-spun into an evaporative medium, the whole or a definite part of which is made to pass through the immediate vicinity of the spinning orifices solely by means of the suction applied to withdraw it from the spinning cell, whilst additional evaporative medium is also introduced into the neighbourhood of the spinning orifices.

F. R. ENNOS.

**Treatment [saccharification] of cellulosic material.** H. DREYFUS (B.P. 353,028, 17.4.30).—Wood



etc., after purification with alkali and pretreatment with mineral and/or org. acid, is esterified with an org. acid anhydride in presence of  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$ , or other mineral acid, and afterwards hydrolysed, one or more stages of the treatment being so conducted, *e.g.*, by raising the temp. of reaction, as to degrade the cellulosic material to sugars. F. R. ENNOS.

**Manufacture of artificial horsehair, strips, and ribbons.** G. B. ELLIS. FROM SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 352,414, 7.4.30).—A large number of artificial filaments after spinning are assembled into the desired form and welded together by passing through a solvent or swelling agent, which may contain non-solvent liquids or solids in solution or emulsion (gums, resins, etc.), cellulosic material sol. in the solvent, or colouring matter, at such a speed that they are softened only at the surface. Pigments may also be incorporated in the filaments, if desired, before or after agglomeration. F. R. ENNOS.

**Production of hollow filaments from solutions of cellulose derivatives.** CELLULOSE ACETATE SILK Co., LTD., and H. C. CURTIS (B.P. 353,041, 19.3.30).—Solutions of cellulose derivatives are dry-spun through dies having conical holes which taper to a greater diam. on the outside face of the die, thus: metal thickness of dies 0.4–0.5 mm., holes 0.05–0.07 mm. inside and 0.09–0.11 mm. outside diam. F. R. ENNOS.

**Device for the stretch-spinning [with flowing precipitating liquor] of artificial silk.** BRIT. BEM-BERG, LTD. (B.P. 352,862, 6.11.30. Ger., 7.11.29).—Placed inside the cylindrical spinning vessel and ending in its outlet tube is a smooth conical insertion having several vertically arranged series of apertures, which are staggered with respect to one another and serve to equalise sharp variations in concentration of the precipitating liquor. F. R. ENNOS.

**Manufacture of cellulosic filaments having a structure and properties comparable with those of natural wool.** M. J. DASSONVILLE (B.P. 352,692, 18.6.30. Fr., 26.6.29).—By periodic variations in the supply or pressure of the cellulosic solution passing into the spinneret, a noded filament is produced which, immediately after initial solidification, is waved or crimped. Suitable apparatus is described. F. R. ENNOS.

**Manufacture of artificial threads and filaments [of reduced lustre] and products therefrom.** COURTAULDS, LTD., and C. DIAMOND, with, in (c), W. H. GLOVER (B.P. 352,412, 352,610, and 352,611, [A] 7.4.30, [B, C] 5.5.30. [A, B] Addns. to B.P. 338,269; B., 1931, 110).—Solutions of an org. ester of cellulose containing a small proportion of (A) a monohydric aliphatic ester of a higher fatty acid, *e.g.*, amyl stearate, (B) an org. acid ester of a saccharide sol. in org. solvents, *e.g.*, glucose penta-acetate, or (C) either (A) or (B) together with a small proportion of  $\text{TiO}_2$ , are dry-spun and treated with aq. soap solution at or near the b.p. F. R. ENNOS.

**Treatment of artificial threads and films made from cellulose esters and ethers.** K. WEISSENBERG and B. RABINOWITSCH (B.P. 352,445, 8.1.30. Ger., 18.2.29).—Finished artificial threads, yarns, or films or cellulose esters or ethers which have been treated

with a neutral org. swelling agent, *e.g.*, 50% aq. dioxan in the case of cellulose acetate, or threads of cellulose acetate spun from solutions containing dioxan, are gradually stretched 200% or more while still in contact with the swelling medium. Previous to, during, or immediately after these operations the threads may be subjected to transverse pressure, *e.g.*, by rolling, and they are finally treated with salt solutions and washed with  $\text{H}_2\text{O}$ . F. R. ENNOS.

**Devices for treating thread coils or similar annular masses of spun filaments of artificial silk.** M. SCHOENFELD (B.P. 352,771, 7.8.30. Ger., 9.8.29).—The coils etc. are mounted inside a container around an expanding column, which secures them, and after-treatment liquids and gases are passed by suction or pressure through the windings. F. R. ENNOS.

**Processing of raw cellulosic material.** G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,787,953—4, 6.1.31. Appl., 24.9.29).—(A) Pulp rich in  $\alpha$ -cellulose is obtained by digesting cellulosic material with a liquor which is initially acid and is subsequently made alkaline during the cook. Thus spruce chips are digested with  $\text{H}_2\text{SO}_3$  (5% free  $\text{SO}_2$ ) for about 2 hr. at  $120^\circ$  (2 hr. being allowed to reach this temp.), after which the  $\text{SO}_2$  is removed by raising the temp. rapidly and opening the relief valve, the pressure being gradually lowered meantime to atm. pressure. The residual  $\text{SO}_2$  is then neutralised and the cook completed at about  $168^\circ$  for 3 hr. in an alkaline liquor containing 24–40 g.  $\text{Na}_2\text{O}$  per litre, 40% of which should be present as  $\text{Na}_2\text{S}$ . (B) The  $\text{SO}_2$  remaining in the digester after the acid cook is oxidised to  $\text{SO}_3$  by introducing the requisite quantity of, *e.g.*,  $\text{NaOCl}$ ,  $\text{Na}_2\text{O}_2$ , etc. in order to avoid the formation of thiosulphates on adding an alkaline liquor containing  $\text{Na}_2\text{S}$ . D. J. NORMAN.

**Preparation of cellulose pulp.** R. B. WOLF, R. P. HILL, and R. S. HATCH (U.S.P. 1,788,555, 13.1.31. Appl., 8.2.29).—Lignocelluloses, *e.g.*, wood chips, are treated with anhyd. liquid  $\text{SO}_2$  or a non-aq. solution thereof at  $15$ – $38^\circ$  and 100–125 lb./sq. in. for a sufficient time (2–8 hr.) for the  $\text{SO}_2$  in conjunction with the moisture in the wood to convert the lignin into lignin-sulphonic acids. The liquid  $\text{SO}_2$  is then withdrawn under pressure and the cellulose freed from lignin-sulphonic acids by treatment with  $\text{H}_2\text{O}$  or alkalis. Under these conditions degradation of the cellulose is avoided, high yields are obtained, and the lignin-sulphonic acids are recovered in an unpolymerised form. D. J. NORMAN.

**Manufacture of chemical pulp or cellulose.** D. E. CABLE, ASSR. to OXFORD PAPER Co. (U.S.P. 1,790,260, 27.1.31. Appl., 27.10.27).— $\text{N}_2$  or other inert non-oxidising gas which is not absorbed to any great extent by the digestion liquor is used to supply pressure in the digester at the beginning of the cook and to maintain a suitable pressure throughout the digestion period. Pressure-boosting with  $\text{N}_2$  is particularly useful in sulphite cooking, especially when air-dry (10%  $\text{H}_2\text{O}$ ) chips are used, as it facilitates penetration of the chips, reduces the total cooking time, and improves the bleaching qualities of the pulp. The amount of  $\text{O}_2$  present in the digester may be reduced to a min. by exhausting

the air from the charged digester and admitting  $N_2$  before introducing the cooking liquor.  $N_2$  of sufficient purity is obtained from the stack at the outlet of the acid-making plant.

D. J. NORMAN.

**Pulp-filtering machine.** E. C. LOETSCHER (U.S.P. 1,787,047, 30.12.30. Appl., 12.9.29).—The stock is fed on to a travelling wire cloth as in the ordinary paper machine, sheet formation being assisted by inclining the wire upwards from the feed box end, providing water sprays about half way up the machine, and applying suction to the underside of the wire for practically the whole of its length. The wire must be suitably reinforced to withstand the suction.

D. J. NORMAN.

**Deriving high-grade cellulose from cottonseed hulls.** B. DORNER (U.S.P. 1,789,354, 20.1.31. Appl., 26.10.29).—The cleaned and washed hulls are completely dried at 103–105° to make them brittle and are then humidified just sufficiently long for the cotton fibres to regain their elasticity without any appreciable absorption of moisture by the hull itself. The material is then immediately milled and the cotton fibres are separated from the powdered hull by an air blast. The hulls are more easily powdered if they are soaked in a solution of a metal salt, e.g., 6–8% NaCl solution, before drying.

D. J. NORMAN.

**Manufacture of cellulose and textile fibres from vegetable matter.** C. A. BRAUN (U.S.P. 1,786,890, 30.12.30. Appl., 15.8.27. Fr., 4.7.27).—The action of alkali monosulphite liquors containing free caustic alkali on cellulosic material is considerably accelerated if there is added to the liquor alkali salts of weak acids, e.g., soaps, silicates, borates, aluminates, etc., which by dissociation at high temps. furnish free alkali for dissolving the incrustants and weak acid, the latter, in turn, liberating  $SO_2$  from the monosulphite. A small proportion of  $Na_2CO_3$  is preferably added to maintain a faintly alkaline reaction to the end of the cook. A suitable liquor for 1000 kg. of straw contains, per 2000 litres,  $Na_2SO_3$  85 kg.,  $Na_2SiO_3$  35 kg., NaOH 20 kg., and  $Na_2CO_3$  5 kg. With this liquor a boiling time at 160° of 4 hr. is sufficient as against 5.5–6 hr. with a liquor containing 35 kg. of NaOH and no silicate.

D. J. NORMAN.

**Production of high  $\alpha$ -cellulose fibre.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,790,170, 27.1.31. Appl., 6.5.25. Renewed 23.7.27).—The continuous recovery of heat and soda as applied to a plant producing  $\alpha$ -pulp by digestion of sulphite pulp with alkali, running in conjunction with plant producing kraft pulp, is described.

D. J. NORMAN.

**Preparation of parchment paper stock from corn-stalk pith.** E. R. DARLING, Assr. to CORNSTALK PRODUCTS Co., INC. (U.S.P. 1,790,002, 27.1.31. Appl., 19.10.28).—The cleaned pith is digested with  $H_2O$  under heat and pressure, screened to remove fibrous material, bleached, and beaten to the required degree of hydration at about 57° in an ordinary beater.

D. J. NORMAN.

**Manufacture of coloured and tinted parchment paper and the like.** R. I. COWEN (U.S.P. 1,790,367, 27.1.31. Appl., 6.3.28).—Dyes which are insol. or but slightly sol. in 70%  $H_2SO_4$ , but are sol. in  $H_2O$ , e.g.,

Eric Fast Scarlet (Colour Index No. 326), are suspended in the parchmentising acid so that they become fixed in the paper during parchmentising. The full colour of the dye develops during the washing operation.

D. J. NORMAN.

**Production of impregnated paper and the like.** CALLENDER'S CABLE & CONSTRUCTION Co., LTD., and H. HILL (B.P. 352,533, 10.4.30).—The impregnation of paper in reel form is facilitated by embossing nodules in the paper before reeling, to keep the layers slightly apart.

D. J. NORMAN.

**Manufacture of paper and paper board.** L. M. BOOTH (U.S.P. 1,788,790, 13.1.31. Appl., 5.10.28).—When using old rags and old papers in the furnish, decomp. of the material is arrested and the products of decomp. are precipitated in the pulp as fillers by adding hypochlorite and hydrated  $CaO$ , e.g., 2–5 lb. of  $Ca(OCl)_2$  and 1–3 lb. of  $Ca(OH)_2$  per ton of pulp, to the beater.

D. J. NORMAN.

**Manufacture of cigarette paper.** B. SEFERIADIS, and S. C. PATENTS, LTD. (B.P. 352,541, 11.4.30. Addn. to B.P. 325,125; B., 1930, 414).—The resinols etc. used in the prior process are obtainable in varying quantities from any gum or gum resin, natural or synthetic, by maceration with  $COMe_2$ , EtOH, liquid hydrocarbons, or mixtures thereof. In making the paper it is advantageous to add 1.5–2% of EtOH to the pulp before running on the machine.

D. J. NORMAN.

**Stencil sheets or stencil paper.** W. W. TRIGGS. From DITTO, INC. (B.P. 352,460, 8.4.30).—Yoshino paper is coated with a solution of an unpolymersed phenol resin (Baekeland's product B) in admixture with shortening agents, e.g., metal soaps, waxes,  $BaSO_4$ ,  $MgCO_3$ , etc., and softeners, e.g., oils, or mixtures of neatfoot oil with butyl phthalate or hydrogenated cottonseed oil, and the synthetic resin is subsequently polymerised (to product C) by heat.

D. J. NORMAN.

**Apparatus for treating [rubbing etc.] fibrous material.** I. TUDOR, and O. TUDOR-HART (B.P. 353,424, 17.1.30).

**Machines for spinning artificial silk.** C. HAMEL A.-G., and E. HAMEL (B.P. 353,803, 5.11.30).

**Filter blocks.**—See I. Lubricant for textiles.—See II. Mineral fibres.—See VII. Artificial wood.—See IX. Paper for roll films. Light-sensitive paper.—See XXI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Determination of oxycellulose in bleached cotton.** H. HEINRICH (Textilber., 1931, 12, 113–114).—1 g. of the air-dry bleached cotton is boiled with 20 c.c. of 0.6% NaOH for 1 hr. (the vol. of the alkaline liquor is maintained const. by additions of  $H_2O$ ), the liquor is diluted to 50 c.c., and the colour of the extract compared with that of extracts obtained similarly from bleached cottons having known oxycellulose contents. Bleached cotton if satisfactory yields a clear very pale yellow extract, overbleached cotton brown.

A. J. HALL.

**Lecithin in the textile industry.** O. MECHEELS (Textilber., 1931, 12, 123–124).—Lecithin (from soya beans) when used in the treatment of textile fibres

should be in the form of a neutral or preferably alkaline emulsion; such emulsions are stable in  $H_2O$  up to 15° hardness. Such emulsions may serve as lubricants for cotton and wool in spinning and as finishing agents in softening the handle of artificial silk materials. In finishing bleached cotton goods a bleached lecithin should be used. Increased depth of shade is obtained by the addition of lecithin to printing pastes, and brighter and more level dyeings (even with vat dyes) are produced by adding lecithin to the dye liquor.

A. J. HALL.

**Modern bleaching, dyeing, printing, and finishing machinery.** P. URMSTON (J. Soc. Dyers and Col., 1931, 47, 215—221).

**Textile sizes.**—See XVII.

## PATENTS.

**[Crêping of] textile materials.** BRIT. CELANESE, LTD., and W. A. DICKIE (B.P. 351,999 and 352,000, 24.12.29).—Crêpe fabrics are obtained by impregnating with a swelling agent which becomes effective on dilution with  $H_2O$  (A) fabrics woven with tightly-twisted yarns or (B) tightly-twisted yarns which are afterwards woven into fabrics, and then scouring or otherwise treating with aq. liquors. Suitable swelling agents for cellulosic materials include conc. solutions of  $ZnCl_2$  and  $Ca(CNS)_2$ ; org. swelling agents such as  $COMe_2$ ,  $AcOH$ , and  $EtOH$  suitably diluted with  $C_6H_6$  are satisfactory for yarns composed of cellulose esters and ethers, particularly cellulose acetate. A. J. HALL.

**[Crêping of] textile materials.** H. DREYFUS (B.P. 352,451, 5.3.30).—Yarns of artificial silk, cotton, silk, etc., which have been sized with a polymerised vinyl compound or an artificial resin together with a softening agent (olive oil), are tightly twisted and incorporated in a fabric; this is then scoured in an aq. soap bath or with a swelling agent for the twisted threads or for the size ( $EtOH$ , xylene dispersed in a sulphonated oil, etc.) without rendering the latter relatively soft or yielding. (Cf. B.P. 348,589; B., 1931, 716.) F. R. ENNOS.

**[Weighting of] animal fibres.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 353,033, 20.1.30).—Wool etc. is treated with an aq. solution of at least 10% of its wt. of an org., non-dyeing, halogenated compound having a mol. wt. above 200 and an affinity for the fibres, e.g., 2:3:6-trichloroanilinesulphonic acid, tetrachlorophthalic acid, chlorinated fatty acids, sulphonic acids of chlorinated paraffin waxes, etc.

F. R. ENNOS.

**Apparatus for treating artificial silk with liquids.** H. A. GILL. From GLANZSTOFF-COURTAULDS G.M.B.H. (B.P. 354,008, 19.6.30).

**Filter press.**—See I. **Stable diazo preps.**—See IV. **Tinted parchment.**—See V. **Coloured rubber.**—See XIV. **Colour photography.**—See XXI. **Insecticide.**—See XXIII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Nitration process of sulphuric acid manufacture.** M. MATSUI (Proc. World Eng. Cong., 1929, 31, 265—288).—A comprehensive summary of previous

work of the author (cf. B., 1923, 265; 1930, 238, 321, 903).

C. IRWIN.

**Titration of hydrofluoric acid containing hydrofluosilicic acid.** K. KOBAYASHI (Proc. World Eng. Cong., 1929, 31, 399—407).—About 1 g. of the sample is weighed in a covered Pt crucible containing 5 c.c. of distilled  $H_2O$  and poured into a large Pt dish, 10—15 c.c. of saturated aq.  $KNO_3$  are added, and the solution is titrated with  $N-NaOH$  solution and phenol-red until the colour does not fade in 5 sec.; it is then warmed to 90° and titrated to a permanent pink colour. The first figure gives  $HF + H_2SO_4 + \frac{2}{3}H_2SiF_6$ , the second the total acid. This method obviates the use of ice, which is necessary with phenolphthalein. The limit of accuracy is that of the sampling. C. IRWIN.

**Liquefaction of gases and syntheses under pressure. Synthesis of ammonia and methyl alcohol.** R. E. JORDAN (Proc. World Eng. Cong., 1929, 31, 139—152).—The Claude  $NH_3$  process is described. If 5—8% CO is left in the  $H_2$  derived from coke-oven gas and the mixture compressed to 1000 atm. a preliminary catalysis converts almost the whole of the CO into  $MeOH$ . The remaining compressed gases are then available for  $NH_3$  synthesis. By varying the catalyst higher alcohols or hydrocarbons may be obtained in place of  $MeOH$ . The first fraction on liquefaction of coke-oven gas consists chiefly of propylene, and has no present application. The second consists of  $C_2H_4$  and  $C_2H_6$  and forms 1.5—2.0% of the gas. The  $C_2H_4$  is used for the prep. of  $EtOH$ ; it is also possible to prepare instead  $Et_2O$  or  $EtOAc$  using the Ca salt. The synthesis of petrol from  $C_2H_4$  is possible, but not economic at present prices. That of glycol is more attractive if cheap  $Cl_2$  is available. The  $C_2H_6$  may be converted into  $C_2H_4$  with a yield of 80% by controlled oxidation with air. The  $CH_4$  may be decomposed into the elements at 1150° and the C produced is suitable for printing ink. With  $H_2O$  vapour at 1034° and in presence of Cu the reaction  $CH_4 + H_2O = CO + 3H_2$  occurs. The  $CH_4$  may also be chlorinated, or used for the production of cyanides or of  $CH_2O$ . C. IRWIN.

**Oxometer for the determination of hydrogen peroxide etc.** CHEM. FABR. PYRGOS G.M.B.H. (Chem.-Ztg., 1931, 55, 540).—The apparatus comprises a stoppered cylinder with a lower mark to which the solution to be tested is filled, and a scale graduated in 0.25% steps to 5% starting some distance above the lower mark. The vol. between the latter and the zero of the scale is filled with 50%  $H_2SO_4$  and the solution is then titrated with a  $KMnO_4$  solution supplied with the cylinder, the vol. of the latter used giving directly the %  $H_2O_2$  from the scale reading. The apparatus may also be used for  $Na_2O_2$  and Na perborate.

A. R. POWELL.

**Superphosphate enriched with ammonia.** S. I. VOLFKOVICH, L. E. BERLIN, I. L. HOFMAN, and A. A. IONAS (Udobr. Urozhai, 1930, 2, 556—569).—The preparation of ammoniated superphosphate from raw phosphate (Portland and Saratov),  $H_2SO_4$ , and  $(NH_4)_3PO_4$  is described. CHEMICAL ABSTRACTS.

**Chemical constitution of bleaching powder.** S. URANO (Proc. World Eng. Cong., 1929, 31, 171—180).

—A mixture of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaCl}_2$  loses the whole of its Cl under action of  $\text{CO}_2$  or of heat alone. The loss of Cl by the action of  $\text{CO}_2$  on bleaching powder is therefore no evidence as to the composition of the latter. Thermochemical tests show that bleaching powder in solution behaves in this respect as a mixture of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaCl}_2$ . Solid bleaching powder, however, is a compound of the two with a heat of formation of 2800 g.-cal. per mol. of  $\text{CaOCl}_2$ . A study of the system  $\text{Ca}(\text{OCl})_2$ – $\text{CaCl}_2$ – $\text{H}_2\text{O}$  showed that a triple point exists at  $48^\circ$  above which no double salt can exist. Between  $48^\circ$  and  $-50^\circ$  double salt can exist in equilibrium with either single salt, and below  $-50^\circ$  it can exist alone. The v.p. of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaOCl}_2$  coincide at about  $47^\circ$ . The reactions between  $\text{CaO}$  and  $\text{Cl}_2$  are therefore (1)  $\text{Ca}(\text{OH})_2 + \text{Cl}_2 = \text{CaOCl}_2 \cdot \text{H}_2\text{O} + 16,500 \text{ g.-cal.}$ ; (2)  $2\text{Ca}(\text{OH})_2 + 2\text{Cl}_2 = \text{Ca}(\text{OCl})_2 + \text{CaCl}_2 \cdot 2\text{H}_2\text{O} + 27,400 \text{ g.-cal.}$  The partial pressures of  $\text{Cl}_2$  calc. by the Nernst formula from the two equations become equal at  $45.3^\circ$ , which is therefore the transition point. Bleaching powder is a double salt with some  $\text{H}_2\text{O}$  of crystallisation. C. IRWIN.

**Utilisation of Korean alunite.** H. TANAKA (Proc. World Eng. Cong., 1929, 31, 289–290).—Alunite  $3(\text{Al}_2\text{O}_3 \cdot \text{SO}_3) \cdot \text{K}_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  is calcined at  $600^\circ$  and treated with hot aq.  $\text{NH}_3$  solution. A mixed solution of  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  is obtained and evaporated to crystallisation. The residue, together with 20–40 pts.  $\text{H}_2\text{O}$ , is treated at  $60$ – $70^\circ$  for 3–5 hr. with  $\text{SO}_2$ .  $\text{Al}_2\text{O}_3$  goes into solution,  $\text{SiO}_2$  remaining undissolved. The solution is heated at  $100^\circ$ , when basic sulphite is precipitated. This is filter-pressed and calcined at above  $900^\circ$ , yielding a pure  $\text{Al}_2\text{O}_3$  almost free from  $\text{Fe}_2\text{O}_3$ . C. IRWIN.

**Determination of the hydrolytic acidity of fuller's earth and its decolorising power.** H. UTERMÖHLEN (Chem.-Ztg., 1931, 55, 625–626).—The hydrolytic acidity of various samples of fuller's earth was determined by the  $\text{NaOAc}$  method, as with soil samples; the results agreed well with those of actual bleaching tests. This quantity is, in the case of a soil, a measure of the absorptive power to OH and Na ions, and the action of fuller's earth on oils is held to be analogous. C. IRWIN.

**Reports of investigations: [Canadian] non-metallic minerals.** R. K. CARNOCHAN and R. A. ROGERS (Canad. Dept. Mines, Repts. Ore Invest., 1929, 159–175).—Experiments designed to improve the quality of asbestos,  $\text{SiO}_2$  sand for glass making, a uraninite ore, mica, and gypsum are recorded. The methods used involved selective crushing, table dressing, and magnetic separation. A. R. POWELL.

**Manufacture of lead suboxide and some of its industrial applications.** G. SHIMADZU (Proc. World. Eng. Cong., 1929, 31, 325–344).—Pb lumps about 1 in. diam. are fed automatically into a gas-tight drum with hollow trunnions on which it revolves. Through the trunnion at the feed end passes a compressed-air pipe having a number of branches. The speed of the drum is so arranged that the Pb is in const. motion. Temp. rise occurs owing to friction, but chiefly to the formation of  $\text{Pb}_2\text{O}$ ; the optimum wall temp. is

$140$ – $150^\circ$ . The exit air passes through bag filters. Air control regulates the temp.  $\text{Pb}_2\text{O}$  as prepared by this process contains 95%  $\text{Pb}_2\text{O}$  and 5% Pb. It is a very finely-divided black powder which is gradually oxidised by air to  $\text{PbO}$ . It burns rapidly to the same product when lit with a match or agitated with compressed air.  $\text{PbO}$  can therefore be manufactured without the use of fuel. The rapidly oxidised product contains a little  $\text{Pb}_3\text{O}_4$  and is converted into the latter by igniting at  $400^\circ$  under oxidising conditions.  $\text{Pb}_2\text{O}$  with boiled linseed oil gives a paint of high value, and the  $\text{PbO}$  and  $\text{Pb}_3\text{O}_4$  prepared from it are exceptionally pure. C. IRWIN.

**Manufacture of oxygen, nitrogen, and the rare gases.** R. E. JORDAN (Proc. World Eng. Cong., 1929, 31, 131–138).—The Claude liquid air process starting with cooled air at 20 atm. divides it into two portions; one is liquefied, the other cooled, by expansion while performing work. The liquid air is fractionated first in a high-pressure column with bottom feed, and secondly in a low-pressure column. The use of Kuhni plates containing spirally wound ribbons of Cu, one of which is flat and one corrugated, further increases fractionating efficiency. The gaseous residue after washing with liquid N contains 37.5% Ne, 12.5% He, 50% N. The Ne and  $\text{N}_2$  are absorbed by coconut charcoal, from which the Ne is recovered by regulated heating. He which is not absorbed is obtained directly. A has b.p.  $-186^\circ$ , and a plate in the column can be found containing up to 4% A in liquid O. This liquid is further rectified, giving an 80% A gas which is freed from  $\text{O}_2$  by burning  $\text{H}_2$ . The Kr and Xe accumulate in the O vaporiser residues. If these are burned with  $\text{H}_2$ , 6000 litres of mixed gas yield 9 litres of Kr and 0.8 litre of Xe. They are absorbed in  $\text{SiO}_2$  gel cooled with liquid N. Kr is liberated by warming to air temp., Xe at  $200^\circ$ . They are purified by redistillation at the temp. of liquid air. C. IRWIN.

**Extraction of [pure] krypton and xenon from liquid air residues.** F. J. ALLEN and R. B. MOORE (J. Amer. Chem. Soc., 1931, 53, 2512–2522; cf. A., 1930, 1508, 989; 1928, 1209).—The compressed liquid air residues are condensed in charcoal at liquid air temp. and then boiled off slowly and passed over hot Cu and Ca, thus avoiding the use of large gas holders. Details of the fractionation are given. J. G. A. GRIFFITHS.

**$\text{CaF}_2$  for furnaces.**—See XI. Nitroglycerin waste acids.—See XXII. Determination of As.—See XXIII.

#### PATENTS.

**Manufacture of sulphuric acid by the lead-chamber or by the tower process.** E. ROTHAMMEL (B.P. 348,142, 5.2.30).—A portion of the gases from the Glover tower is withdrawn by a suction fan and forced with an injector action into the conduit supplying roaster gases to the system. One or more denitrators followed by one or more nitrators are arranged in front of the Glover tower and nitrosulphuric acid from the Gay-Lussac tower is led through the nitrators, and a portion of the  $\text{H}_2\text{SO}_4$  from these is returned in a closed circuit to the top of the nitrators. In the apparatus succeeding the Glover tower  $\text{H}_2\text{SO}_4$  ( $d$  1.7)

containing  $N_2O_3$  is sprayed under such conditions that the gases flowing through the first section take up  $N_2O_3$  from the acid and give up the gas again to the acid in the second section. Various other modifications of the process are indicated. A. R. POWELL.

**Preparation of phosphoric anhydride and phosphoric acid.** METALLGES. A.-G. (B.P. 352,367, 29.11.30. Ger., 29.11.29).—Molten P is atomised by allowing it to fall on a rotating disc together with a stream of  $H_2O$  while a current of cold air passes upwards round the disc to cool it. Combustion of the P- $H_2O$  spray affords  $H_3PO_4$  in any desired degree of concentration by adjusting the ratio of P :  $H_2O$ . A. R. POWELL.

**Production of sodium carbonate liquor.** H. W. MORSE (U.S.P. 1,791,281, 3.2.31. Appl., 29.4.29).—The mixture of  $Na_2CO_3$ ,  $Na_2SO_4$ , and NaCl obtained from the mother-liquors in the extraction of  $Na_2B_4O_7$  and K salts from Searles Lake brine is leached with small quantities of  $H_2O$  at 30–40°, whereby a solution containing 12%  $Na_2CO_3$  is obtained.

A. R. POWELL.

**Production of potassium nitrate, sodium bicarbonate, and compound fertilisers.** SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 352,307, 4.9.30. Fr., 15.11.29).—In the manufacture of  $KNO_3$  by the interaction of  $NaNO_3$  with KCl, sylvinit is used as the source of KCl, and after the greater part of the  $KNO_3$  has been recovered by crystallisation the mother-liquor is treated with  $NH_3$  and  $CO_2$  to ppt.  $NaHCO_3$ , leaving  $NH_4Cl$  and the remaining  $KNO_3$  in solution; evaporation of this liquor yields a valuable mixed fertiliser.

A. R. POWELL.

**Treatment of phosphate material.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 353,160, 27.5.30. Nor., 30.5.29).—After phosphate rock has been decomposed with  $HNO_3$  and  $Ca(NO_3)_2$  crystallised out, the mother-liquor is treated with  $NH_3$  or  $(NH_4)_2CO_3$  under heat and pressure, if desired,  $Ca_3(PO_4)_2$  or a mixture of  $Ca_3(PO_4)_2$  and  $CaHPO_4$  being precipitated and dissolved in  $HNO_3$  together with more phosphate rock. The mother-liquor may be freed from  $HNO_3$  by distillation before precipitation, and the precipitated phosphates may be treated with HCl to remove part of the  $Ca^{++}$  before dissolving it. The solution of  $NH_4$  salts is evaporated and the product mixed with K salts.

W. J. WRIGHT.

**Manufacture of ammonium phosphates.** UNION CHIM. BELGE, SOC. ANON. (B.P. 352,309, 5.9.30. Fr., 7.11.29).—Dry cryst.  $(NH_4)_3PO_4$  is treated with the theoretical quantity of  $H_3PO_4$  containing 50%  $P_2O_5$ , conc.  $HNO_3$ , conc. HCl, or conc.  $H_2SO_4$  to convert the salt into  $(NH_4)_2HPO_4$  or  $NH_4H_2PO_4$  and the mixture is dried.

A. R. POWELL.

**Production of ammonium phosphate.** KUNST-DÜNGER-PATENT-VERWERTUNGS A.-G. (B.P. 352,814, 19.9.30. Swed., 5.10.29).—Phosphate rock is leached with  $H_2SO_4$ , insol. matter is removed, and the clear solution neutralised with  $NH_3$ , mono- or di- $NH_4$  phosphate being obtained by recrystallisation. The mother-liquor is utilised in the leaching process, its  $(NH_4)_2SO_4$  content being available instead of a corresponding amount of  $H_2SO_4$ .

W. J. WRIGHT.

**Manufacture of triammonium phosphate.** E. URBAIN (B.P. 352,725, 4.7.30. Ger., 8.5.30).— $H_3PO_4$  is passed into the upper part of an absorption tower into which sufficient  $NH_3$  is introduced to give a solution of mono- and di- $NH_4$  phosphates, which is continuously returned to the  $H_3PO_4$  container. The solution is then treated with excess of  $NH_3$  in a cooled mixer, dry cryst.  $(NH_4)_3PO_4 \cdot 5H_2O$  being thus obtained. W. J. WRIGHT.

**Removal of calcium chloride liquor adhering to calcium hypochlorite.** I. G. FARBERIND. A.-G. (B.P. 354,093, 11.9.30. Ger., 17.9.29).— $Ca(OCl)_2$  crystals, after centrifuging or pressing, are stirred with a wt. of anhyd.  $Na_2SO_4$  equiv. to the  $CaCl_2$  in the adhering mother-liquor.

L. A. COLES.

**Preparation of calcium cyanamide.** N. CARO and A. R. FRANK (B.P. 352,264, 14.7.30. Ger., 18.12.29).—Finely-powdered  $CaC_2$  at least 10% of which passes 75-mesh, is compressed, rolled, or shaped, with the addition, if desired, of suitable solid or liquid agglomerants, into pieces of any size or shape, and these pieces are subjected to the action of  $N_2$  under pressure at a suitable temp. to form  $CaCN_2$ .

A. R. POWELL.

**Manufacture of chromium oxide.** J. E. DEMANT (B.P. 353,152, 22.5.30).—Cr or an alloy containing it, e.g., ferrochrome, is roasted in presence of  $O_2$ , or gases containing or evolving  $O_2$ . Fe and Si are removed from the product by treatment with acids or alkalis, or the product may be heated at a high temp. in a reducing atm. to reduce the Fe.

W. J. WRIGHT.

**Production of asbestos and mineral artificial fibres.** W. LÜDKE (B.P. 352,681, 13.6.30).—Solutions of sol. silicates, e.g., water-glass, to which are added salts that do not cause immediate coagulation, e.g.,  $NaAlO_2$ , are spun into threads of silicate jelly in suitable coagulating agents, e.g., org. liquids or hot gases. These threads are stabilised by heating with  $H_2O$  or a solution of an electrolyte, e.g.,  $MgCl_2$ ,  $CaCl_2$ ,  $KOH$ , HCl, under pressure.

D. K. MOORE.

**Depuration of waste water containing cyanide.** OESTERR.-ALPINE MONTANGES. (B.P. 353,184, 16.6.30. Austr., 24.6.29).—The cyanide is decomposed by bringing the waste water, without previous heating, in counter-current to kiln or other waste gases containing  $CO_2$ , in an irrigation plant, the hydrocyanic acid being suitably extracted from the waste gases.

W. J. WRIGHT.

**[Preparation of] substances containing sulphur in a chemically active condition.** BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. OF B. W. NORDLANDER (B.P. 353,970, 14.5.30. U.S., 14.5.29).—A mixture (preferably 1:1) of bentonite and  $SeS_2$  is heated at 125–150°, cooled, and pulverised; the product may be used as a vulcanising agent or in the manufacture of therapeutic products, fungicides, skin pastes, etc.

L. A. COLES.

**Production of aqueous dispersions or solutions of iodine.** D. McKIE and A. BENNETT (B.P. 353,892, 21.3.30).—Powdered I is stirred with a dil. aq. solution of a simple  $NH_3$ -compound, decomposed by halogens in the presence of  $H_2O$ , preferably with boiling 0.5–4.0% aq.  $NH_3$ , under conditions such that simultaneous or subsequent oxidation can occur, e.g., in the presence of air or  $O_2$ .

L. A. COLES.

Recovering crystals from liquors. Centrifuging salts. Liquefaction of gases. Carrying out catalytic reactions.—See I. Flux for Al etc.—See X. Electrolysis of aq. solutions.—See XI. Fertilisers. Manures.—See XVI.

### VIII.—GLASS; CERAMICS.

**Flow tests in small glass tanks.** A. L. MARDEN (J. Soc. Glass Tech., 1931, 15, 119—128 T).—The flow through a small glass tank giving rise to cordy glass was confined to a narrow section between bottom and top stagnant layers, in almost a direct line from the charging pot to the outlet. On forcing the glass down to the bottom, the results were more satisfactory if the surface was skimmed occasionally. Flow lines on other furnaces feeding machine blowers are described. C. A. KING.

**Investigation of the flow of glass in tank furnaces by the use of barium oxide.** E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1931, 15, 128—140 T).—The addition of  $\text{BaCO}_3$  to a  $\text{Na}_2\text{O}$ - $\text{CaO}$ - $\text{SiO}_2$  glass for tracing the flow of glass does not sensibly affect a normal working, neither does it provide a quant. measure of distribution complementary to the qual. method of adding  $\text{Co}_2\text{O}_3$ .  $\text{BaO}$  was first observed at the machines in 9—10 hr., which indicates a fast-moving current in the tank, and the actual max. of 20% of the theoretical max. suggests powerful mixing currents. Probable lines of flow are discussed and the results show that the position of a machine is a more important factor than its "pull" in relation to the rate of flow. C. A. KING.

**Clay mixtures for glass-melting pots. III. Flow at high temperatures of pot-clay refractories under stress.** J. H. PARTRIDGE and G. F. ADAMS (J. Soc. Glass Tech., 1931, 15, 190—208 T; cf. B., 1931, 805).—Under a const. stress, a higher content of  $\text{Al}_2\text{O}_3$  (20—36%) increased the temp. of failure. All specimens containing grog were less resistant, and particularly so when coarser grog was used. Rapid tensile tests proved to be misleading as under prolonged creep tests the more silicious clays withstood the more severe conditions of stress and temp. At temps. as low as  $1300^\circ$  aluminous clays flowed continuously, but silicious clay attained a const. dimension; the former is therefore recommended only for short-period heatings. C. A. KING.

**Use of ammonium sulphate as an accelerating agent in glass melting.** M. PARKIN, W. J. A. WARREN, and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 153—166 T).—Both the melting and refining processes of glasses containing  $\text{Na}_2\text{O}$  13.1—16.8% and  $\text{CaO}$  8.5—13.8% were accelerated by the addition of up to 10 pts. of  $(\text{NH}_4)_2\text{SO}_4$  per 1000 pts. of sand. The  $(\text{NH}_4)_2\text{SO}_4$  appears to undergo some reaction with the Na salts and to leave residual sulphates accompanied by the escape of  $\text{NH}_3$ , which probably causes a stirring effect.  $(\text{NH}_4)_2\text{SO}_4$  is considered a more effective accelerator than  $\text{Na}_2\text{SO}_4$ . C. A. KING.

**Use of ammonium sulphate in glass making.** A. E. J. VICKERS [with G. H. WHITING, R. MILLER, and V. BATES] (J. Soc. Glass Tech., 1931, 15, 166—172 T).—Preliminary trials showed that about 0.7% of  $(\text{NH}_4)_2\text{SO}_4$

in a glass batch may increase output to a machine and give a longer working range with a reduced working temp. Glass of more brilliant colour and increased fluidity was made with rapid elimination of seed, and a possible reduction in Na content would produce a more durable glass. C. A. KING.

**Influence of manganese oxide on some properties of glass.** A. A. CHILDS, V. DIMBLEBY, F. WINKS, and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 172—184 T).—In a parent glass of composition  $\text{SiO}_2$  72%,  $\text{CaO}$  10%,  $\text{Na}_2\text{O}$  18%, the  $\text{Na}_2\text{O}$  was progressively replaced by  $\text{MnO}_2$ . The melting rate was increased distinctly by  $\text{Fe}_2\text{O}_3$  ( $\text{FeO}$ ) up to 2%, but not by  $\text{MnO}$ , which, however, greatly reduced the refining period, though  $>0.5\%$  had no further effect. Fluidity was increased with 2.5—3%  $\text{MnO}$  without further effect up to 5%, but  $\text{MnO}$  was not so effective as  $\text{FeO}$  in this respect. Although glass with 2.5—3%  $\text{MnO}$  is opaque, considerable light is transmitted if 2%  $\text{FeO}$  is present and opacity is not reached with 5%  $\text{MnO}$  + 2%  $\text{FeO}$ . The thermal expansion diminished with increasing  $\text{MnO}$ , but the resistance to corrosion by alkali increased. C. A. KING.

**Constitution of glass based on the laws of solutions.** O. KNAPP (J. Soc. Glass Tech., 1931, 15, 140—152 T).—The mol. ratio of the oxides does not indicate the properties of a glass, which is considered as a genuine solution of silicates in which, generally, free  $\text{SiO}_2$  is dissolved. The optical const. and the compositions of glasses indicated according to the highest silicate hypothesis give constitutive rules, and expressions are worked out for the weathering of  $\text{CaO}$  and  $\text{Pb}$  glasses and the tarnishing of the latter for both mono- and di-silicised forms. C. A. KING.

**Density of sodium metasilicate-silica glasses.** F. WINKS and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 185—190 T; cf. B., 1930, 767).—The densities of  $\text{Na}_2\text{O}$ - $\text{SiO}_2$  glasses containing 50—92%  $\text{SiO}_2$  were determined and compared with the calc. values from known formulæ. An error of  $<0.02$  was obtained by using Winkelmann and Schott's formula if the factors 3.38 for  $\text{Na}_2\text{O}$  and 2.20 for  $\text{SiO}_2$  were applied for glasses containing up to 35%  $\text{Na}_2\text{O}$ . Two formulæ by the Bureau of Standards gave closer values for 50—80%  $\text{SiO}_2$  glasses, but failed with glasses containing  $>85\%$   $\text{SiO}_2$ . C. A. KING.

**Heat-absorbing glass.** A. F. DUFFON (J. Soc. Glass Tech., 1931, 15, 208—212 T).—The criterion of efficiency hitherto adopted for heat-absorbing glass does not take into account that the glass becomes warm and so transmits heat to the atm. A modified empirical transmission factor  $F = T + (I - R - T)/\{2 + (I + E)/K\}$ , in which  $I$  is the transmission for the solar radiation,  $E$  the emissivity,  $K$  the conductivity, and  $R$  the reflecting power for solar radiation, enables windows of different kinds of glass to be graded. Double-glazing with heat-absorbing glass and ordinary glass which is opaque to radiant heat reduces the heat communicated to a room. C. A. KING.

**Transparency of glasses to ultra-violet rays.** P. GILARD, P. SWINGS, and A. HANTOT (Bull. Acad. roy. Belg., 1931, [v], 17, 593—602).—Experiments

with industrial glasses having the general composition 5 or 6( $\text{SiO}_2, \text{B}_2\text{O}_3$ ),  $\text{MO}, \text{M}_2\text{O}$ , or near, confirm, in general, the previous conclusions (A., 1931, 546) regarding the influence of different ingredients. Fe, Mn, La, and Ce oxides reduce the transparency, whilst Pr and Nd oxides exert little effect. The reduction in the transparency caused by increasing the thickness of the specimen, and by previously irradiating it with a strong beam of ultra-violet light, has been studied.

N. H. HARTSHORNE.

**Hydrolytic stability of chemical glassware.** F. H. ZSCHACKE (Chem.-Ztg., 1931, 55, 580—581).—Standardisation by the boiling  $\text{H}_2\text{O}$  test is recommended.

C. IRWIN.

**Static rigidity of plastic clays.** C. TERZAGHI (J. Rheology, 1931, 2, 253—260).—Plastic flow is preceded by a gliding flow, having its origin in the highly viscous film of  $\text{H}_2\text{O}$  surrounding the points of adhesion. The force available for plastic flow is equal to the difference between the total stress and the shearing resistance ("static rigidity"). E. S. HEDGES.

**Canadian non-metallic minerals [ $\text{SiO}_2$  sand].**—See VII. **Boundary fields [and glass tubing].**—See X.

#### PATENTS.

**Lehrs for annealing glassware.** BRIT. HARTFORD-FAIRMONT SYND., LTD. From HARTFORD EMPIRE CO. (B.P. 353,245, 19.7.30).—The lehr is of the type in which a metal tunnel is surrounded by an insulated metal casing, the tunnel being supported by heat-insulating blocks which allow of relative and expansional movement.

C. A. KING.

**[Fortifying] treatment of [frosted] glass articles.** R. D. SMITH, Assr. to CORNING GLASS WORKS (U.S.P. 1,791,066, 3.2.31. Appl., 30.4.30).—The articles are washed in an aq. solution containing not more than 750 pts. per  $10^6$  of a simple Al salt of an aliphatic org. acid, e.g., Al tartrate (cf. U.S.P. 1,772,965; B., 1931, 395).

L. A. COLES.

**Drying of clay ware or kindred products.** W. W. TRIGGS. From HARROP CERAMIC SERVICE CO. (B.P. 352,381, 30.12.29).—The dryer is of the continuous tunnel type. The hot gas enters at the discharge end and by means of baffles is made to flow tortuously for about half the length of the tunnel. It then enters a chamber above the tunnel, passes through a number of ports along the length of the remainder of the tunnel into the tunnel, and is withdrawn at outlet ports opposite to the inlet ports, to be recirculated in the inlet part of the dryer.

D. K. MOORE.

**Production of material for moulding and firing, from raw material containing lime.** B. JIROTKA (B.P. 352,665, 29.5.30. Ger., 29.5.29).—Raw clay pressed into a bar is fed on to a rapidly rotating grinding device and the ground material is thrown off into moulds. Foreign matter, e.g., stones, is thrown off at different velocity and thus separated.

D. K. MOORE.

**Manufacture of porcelain.** BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of L. E. BARRINGER (B.P. 352,763, 1.8.30. U.S., 10.8.29).—Porcelain made from china clay, ball clay, felspar, and pyrophyllite is less

abrasive and has greater mechanical and dielectric strength than has ordinary porcelain. D. K. MOORE.

**Manufacture of porcelain for use in cooking.** G. FUSCHI and M. KORACH (B.P. 352,723, 3.7.30).—To eliminate the fracture of porcelain owing to the difference of coeffs. of expansion of the mass and the glaze, the moulded articles instead of being glazed are polished before and after burning. Such porcelain may be fired in piles if a layer of non-fusible substance is placed between the articles.

D. K. MOORE.

**Manufacture of refractory products from magnesium silicates.** METALLGES. A.-G., and V. M. GOLDSCHMIDT (B.P. 352,881, 7.7.30. Cf. B.P. 340,487; B., 1931, 350).—Granular Mg silicates the vol. of which remains const. or nearly so on being fired are used in the place of the orthosilicates mentioned previously.

D. K. MOORE.

**[Synthetic] moulded articles such as grinding wheels.** BAKELITE CORP., Assecs. of R. S. DANIELS (B.P. 353,019, 17.4.30. U.S., 19.4.29).—A composition for moulding abrasive articles contains 3—10 pts. of abrasive grains and 1 pt. of a phenolic resinoid binder including a finely-divided ingredient (5—35%).

C. A. KING.

**Manufacture of [thin-walled] glass articles.** MACBETH-EVANS GLASS CO., Assecs. of T. H. SLOAN (B.P. 353,770, 26.9.30. U.S., 28.9.29).

**Electric glass furnaces.**—See XI.

#### IX.—BUILDING MATERIALS.

**Prediction of cement durability.** E. T. CARLSON and P. H. BATES (Eng. News-Rec., 1931, 107, 130—132).—A check study of Merriman's method (B., 1930, 191) of titrating a suspension of cement in sugar solution for CaO confirms to some extent the previous results, but the method is subject to inaccuracy owing to lack of sharpness of the end-point and the time factor. No correlation with actual durability has been proved and the method is not regarded as a reliable accelerated test.

C. A. KING.

**Synthesis of calcium aluminates and their hydration.** VIII. S. NAGAI and R. NAITO (J. Soc. Chem. Ind. Japan, 1931, 34, 159—161).—The strongest cement of the celite type is  $4\text{CaO}, \text{Al}_2\text{O}_3, \text{Fe}_2\text{O}_3$  followed by  $6\text{CaO}, \text{Al}_2\text{O}_3, 2\text{Fe}_2\text{O}_3$ . These cements consist of the ferrite  $2\text{CaO}, \text{Fe}_2\text{O}_3$  and the aluminate  $2\text{CaO}, \text{Al}_2\text{O}_3$  when the original material has been heated above  $1200^\circ$ .

A. R. POWELL.

**Determination of depth of penetration of mercuric chloride in impregnation of round wood.** M. DREIFUSS and A. STAAB (Chem.-Ztg., 1931, 55, 497—498).—A cylindrical section is cut out of the wood (e.g., a fir mast) and split with a wooden wedge; the split surface is then treated with  $(\text{NH}_4)_2\text{S}$ , which causes blackening where the  $\text{HgCl}_2$  solution has penetrated. For the quant. determination 100 g. of wood are destructively distilled in an Fe retort, the tarry matter is removed from the distillate with  $\text{COMe}_2$ , and the Hg dissolved in  $\text{HNO}_3$  and precipitated with  $\text{H}_2\text{S}$ . The  $\text{HgS}$  is collected, washed free from  $\text{HNO}_3$ , and shaken with  $\text{CS}_2$  and I-KI solution and the excess I is titrated with  $\text{Na}_2\text{S}_2\text{O}_3$ .

A. R. POWELL.



**Trass and sand as additions to cement.** R. GRÜN (Chem. Erde, 1931, 6, 402—415).—A continuation of previous work (B., 1930, 558). Tests were also made with Rhine sand and powdered basalt.

L. J. SPENCER.

**Effect of extractives on the strength of wood.** R. F. LUXFORD (J. Agric. Res., 1931, 42, 801—826).—In certain woods, the extractive contents of which were artificially varied, the compression strength parallel to the grain, the bending strength, and, to a small extent, the shock-resistance increased with the extractive content.

A. G. POLLARD.

**Heat-absorbing glass.**—See VIII. **Pig-Fe [slag].**—See X.

#### PATENTS.

**Treatment of concrete or the like.** M. GIESE and F. HELE (B.P. 352,676, 6.6.30. Ger., 11.7.29).—The adhesiveness and fluidity of concrete may be improved by submitting it to the action of high-speed rotary beaters.

D. K. MOORE.

**Manufacture of bricks.** A. PAUL, JUN. (U.S.P. 1,791,372, 3.2.31. Appl., 29.5.29).—A mixture of 10—15 pts. of CaO with 90—85 pts. of volcanic ash,  $\frac{1}{16}$ — $\frac{3}{4}$  in. diam., is treated with H<sub>2</sub>O in a closed vessel to retain liberated steam, and the product, after moulding into bricks, is treated with steam under pressure.

L. A. COLES.

**Making coloured granules [for building purposes].** FISKE & Co., INC., and R. L. ATKINSON (B.P. 352,789, 27.8.30. U.S., 13.1.30).—Crushed and screened shale is impregnated with a solution of an inorg. salt to give the required colour and fired in a rotary kiln.

D. K. MOORE.

**Manufacture of artificial wood.** A. DE SAMSONOV (B.P. 353,186, 16.6.30).—Vegetable materials, e.g., reeds, cane trash, etc., having the fibres arranged in one direction, are immersed in a natural or artificial resin and compressed. Fungicides may be incorporated.

C. A. KING.

**Impregnation of timber.** PEASE & PARTNERS, LTD., and G. W. WILLIAMS (B.P. 352,992, 10.4.30).—Timber is impregnated by circulating creosote oil around the wood at above 100° and allowing the oil to cool below 100° after moisture has been expelled. Creosote oil from benzol scrubbers may be used after the benzol-recovery process, and, after cooling, is returned to the scrubbers.

C. A. KING.

**Preservation of wood.** H. BAKER (B.P. 352,650, 23.5. and 2.10.30).—An aq. solution of K<sub>3</sub>Fe(CN)<sub>6</sub> or K<sub>4</sub>Fe(CN)<sub>6</sub>, with or without the addition of crude EtOH, is used as the preservative. The addition of tannic acid renders the wood more resistant to fire and more impervious to salt water.

D. K. MOORE.

**Tubular furnace. Drying plant for roads etc.**—See I. **Asphalt emulsions.**—See II. **Al coating [for wood].**—See X. **Insecticide.**—See XXIII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Stationary and tilting open-hearth furnaces with mixed gas firing.** H. BANSEN (Stahl u. Eisen, 1931, 51, 989—995).—The effect of varying H<sub>2</sub>O content in the

gas mixture (blast-furnace and coke-oven gas) on the economics of the open-hearth steel process has been investigated over a prolonged period, and the results are reproduced in tables and graphs, which show the effect of varying furnace construction on the operation of the process and the necessity for close control of the gas analysis for the economical running of the furnace.

A. R. POWELL.

**Smelting [iron ores] in the Saar with coke from small chamber ovens.** E. BERTRAM (Stahl u. Eisen, 1931, 51, 883—885).—Coke produced from Saar coal in small chambers 20 in. wide, fired at 1300° for half the usual period, is almost as good as regards size and crushing strength as that produced by the usual process. The new coke gives equally good results in smelting minette, provided that its ignition temp. does not fall below a certain min. value and provided also that the ore and coke are approx. of the same size.

A. R. POWELL.

**Fine structure of various preheated phosphate slags and its relation to the citric acid solubility.** H. SCHNEIDERHÖHN (Arch. Eisenhüttenw., 1931—2, 5, 9—15; cf. B., 1929, 358).—Addition of fluorspar to basic slags in the Thomas converter results in conversion of the citric acid-sol. silico-carnotite into insol. fluorapatite and  $\beta$ -Ca<sub>2</sub>SiO<sub>4</sub> and the proportion of sol. P<sub>2</sub>O<sub>5</sub> falls from the normal (90—95%) to 3—4% with only 1% F in a slag containing about 20% P<sub>2</sub>O<sub>5</sub>. Basic open-hearth slag contains part of the P<sub>2</sub>O<sub>5</sub> as silicocarnotite and part as two other phosphates of unknown composition both of which are relatively insol. in citric acid; addition of CaF<sub>2</sub> to this slag converts all three P<sub>2</sub>O<sub>5</sub> compounds into fluorapatite. Max. solubility of the P<sub>2</sub>O<sub>5</sub> is obtained with the mol. ratio 5CaO : 1P<sub>2</sub>O<sub>5</sub> : 1SiO<sub>2</sub>; a slight excess of SiO<sub>2</sub> does no harm, but with a deficiency of SiO<sub>2</sub> part of the P<sub>2</sub>O<sub>5</sub> is converted into less sol. Ca tetraphosphate and with an excess of CaO the solubility is reduced.

A. R. POWELL.

**Methods of determining sulphur in iron pyrites.** D. P. LIEBENBERG and S. LEITH (J. S. Afr. Chem. Inst., 1931, 14, 47—51).—Comparative tests on the determination of S in pyrites by the HNO<sub>3</sub>-KClO<sub>3</sub>, HNO<sub>3</sub>-Br, and HNO<sub>3</sub>-HCl methods with and without removal of the Fe with aq. NH<sub>3</sub> showed that equally good results were obtained by all the methods when Fe was removed. A more rapid method than the above comprises fusion of 0.5 g. of ore with 5 g. of Na<sub>2</sub>O<sub>2</sub> in a Ni crucible, dissolving the mass in HCl, reducing the FeCl<sub>3</sub> with KI, and treating the solution with BaCl<sub>2</sub> after expelling the I by boiling.

A. R. POWELL.

**Desulphurisation of cast iron by means of soda ash. Harmful effects of sulphur as an impurity in cast iron** (Imperial Chem. Industries, Ltd., 1931, 12 pp.).—Proprietary compounds sold usually in the form of bricks for the desulphurisation of Fe act by virtue of their alkaline content. The brick form is not necessary, as dense soda ash of low dust content is quite suitable. It is placed in the bottom of the ladle and molten metal run on to it, when the evolution of CO<sub>2</sub> produces vigorous agitation. Eventually the alkaline slag is thickened with ground limestone and removed. About 1% (on the wt. of Fe) of Na<sub>2</sub>CO<sub>3</sub> and 0.5% of CaCO<sub>3</sub> is sufficient in

ordinary cases. About 60% of the total S was removed in foundry trials from cast Fe of low S content. The action is complete in a few min. The Mn content of the Fe is not affected, the Si is slightly reduced, and the physical properties of the metal are improved. The use of soda ash for the direct desulphurisation of molten steel is not recommended. The cost of its use for cast Fe is about 1s. 6d. per ton, and it enables cheaper raw materials to be used whilst complying with a rigid specification for the product.

C. IRWIN.

**Formation of graphite in cast iron.** H. HANEMANN (Stahl u. Eisen, 1931, 51, 966—967).—Quenching of cast Fe (4—15% C, 0.09% Si, 0.18% Mn, and < 0.03% P and S) from above the eutectic point (1141°) produced a purely ledeburitic structure, and from just below the m.p. a completely grey structure. Complete decomp. of the cementite in the same metal required 1 hr. at 1140° and 6 hr. at 1000°. These results indicate that graphite is present in the Fe immediately after solidification, and is not formed by the decomp. of primary cementite, but separates directly from the liquid metal.

A. R. POWELL.

**Case-hardening.** H. MÜLLER (Arch. Eisenhüttenw., 1931—2, 5, 57—62).—Recent work on case-hardening steel with C and N compounds is critically reviewed and in certain respects amplified by further work. CO carburises slowly but evenly without over-carburising the outermost layer; its action may be intensified by increasing the pressure. Hydrocarbons, on the other hand, carburise rapidly and the surface of the metal rapidly becomes converted into carbide which diffuses only slowly inwards unless a high temp. is employed. Nitrogenisation is most effective when pure N<sub>2</sub> is used, but the method is costly and its efficiency depends to some extent on the composition of the steel. Immersion in NaCN baths hardens the surface of steel by absorption of both C and N, but the effect of the latter preponderates. The best case-hardening temp. is 900°, cooling should be rapid, and the hardened articles should be heat-treated below the A1 point (e.g., at 650°) and subjected to a skin-hardening treatment so as to induce a uniform transition from the hard outer layer to the softer core.

A. R. POWELL.

**Precipitation hardening of iron by titanium.** R. WASMUTH (Arch. Eisenhüttenw., 1931—2, 5, 45—56).—Ti alone produces precipitation hardening in Fe only when about 6% is present, but in the presence of Si or Ni only 1—3% Ti is necessary. The presence of C reduces the hardening effect owing to the formation of Ti carbides. The hardness of Fe containing 1.3% Mn, 3.2% Si, and 2.7% Ti after quenching from 1250° is 260, but on annealing at 500° it rises rapidly to a max. of 570 in 12 hr. and at 600° to a max. of 510 in 2—3 hr. Lower quenching temps. and higher Ti to Si ratios result in a decreased precipitation hardening. Addition of 2.3% Ti to V2A steel causes an increase in hardness from 280 to 480 on quenching from 1250° and annealing at 500°. The high temp. from which Ti—Si steels must be quenched before hardening produces a coarse-grained structure, but this may be refined without reducing the precipitation hardness by the addition of Mn, Ni, or Cr. Measurements of the changes of hardness, electrical

conductivity, and magnetic properties of Ti steels after various heat treatments indicate that the mechanism of the precipitation hardening is similar to that of the age-hardening of duralumin. Most of the hardened Ti steels retain their hardness and high tensile strength up to 500°, but on prolonged heating above this temp. the steels become soft again.

A. R. POWELL.

**Ageing of hardened carbon steel.** S. STEINBERG and W. SUBOW (Stahl u. Eisen, 1931, 51, 911—913).—The hardness of steel with 0.98% C rises slowly over a period of several months when the steel is stored after quenching from 750°. Similar increases in hardness occur when the steel is annealed at 75°, 100°, or 125°, but much more rapidly; in all cases the hardening effect is accompanied by a decrease in vol. which reaches a max. of nearly 0.12% at 125° in 30 min. For practical purposes ageing at 125° for 30 min. is recommended, the bending strength is thereby increased from 145 to 195 and the Rockwell C hardness from 65 to 66.

A. R. POWELL.

**Effect on the properties of a metal [nitrogen-iron alloy] of the combined action of cold-work and disperse precipitation [ageing].** W. KÖSTER (Z. Metallk., 1931, 23, 176—177).—The coercivity of a slowly cooled steel containing N in solid solution rose from 3 to 7 gauss in a smooth curve by subjecting it to an elongation of up to 20% in the tensile machine; on subsequent ageing at 100° for 14 days to cause precipitation of the nitride in a finely-dispersed form a further increase of coercivity was obtained depending on the degree of stretching and reaching a max. with 5% elongation. The abs. increase in coercivity decreased linearly with increasing elongation, although the electrical conductivity of all the specimens was the same, showing that equal amounts of nitride had been precipitated. Subsequent annealing at 300° to redissolve the nitride resulted in the restoration of the original coercivity curve, showing that it is possible to redissolve the nitride without removing the effect of cold-work. In a second series of tests the metal was aged at 100°, then stretched up to 20% elongation; the coercivity curve obtained resembled that obtained with the specimens stretched before ageing, but the actual coercivity values were somewhat higher. Subsequent annealing at 300° reduced the coercivity to the figures obtained in the first test. Hence it appears that the effects of cold-work and of ageing on the coercivity of steel are strictly additive.

A. R. POWELL.

**Effects of carbon and iron in blue powder on sherardising.** M. KURODA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 16, 125—126).—Additions of C, particularly 2%, have a slight beneficial action on the rate of formation of a Zn coating by sherardising. Additions of Fe are disadvantageous, causing decrease in rate of coating and entanglement of Fe particles which lead to local rusting.

E. H. BUCKNALL.

**Example of an unstable condition in [nickel-iron] alloys which cannot be stabilised by heating at 1000°.** L. ANASTASIADIS and W. GUERTLER (Z. Metallk., 1931, 23, 189—190).—The ( $\alpha + \beta$ )- $\gamma$  transformation in Ni—Fe alloys with 5—25% Ni is accompanied by hysteresis phenomena which are particularly

marked in the changes which occur in the electrical resistance. In alloys with 15–25% Ni there is a metastable condition which cannot be completely stabilised by prolonged annealing at 1000°. A. R. POWELL.

**Determination of nitrogen in steel and iron alloys.** P. KLINGER (Arch. Eisenhüttenw., 1931—2, 5, 29–32).—The N in Fe alloys containing Cr, W, Ti, V, or Si is not completely recovered as  $\text{NH}_3$  by the dissolution method, and that in Ti, V, or Al alloys is not completely removed by dissociation on heating in vac. at 1100°. Complete expulsion of the N as such is obtained by fusion of the fine drillings with a 1:1 mixture of  $\text{Na}_2\text{O}_2$  and  $\text{Na}_2\text{CO}_3$  in vac.; fusion is carried out in a hard glass tube connected with a heated tube packed with Cu turnings to absorb the  $\text{O}_2$  evolved. The gases are passed backwards and forwards over the Cu until no further reduction in pressure takes place, then freed from  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{H}_2$  in the usual way, and the  $\text{N}_2$  is measured. A. R. POWELL.

**Formation of hard zinc and iron salts [in hot galvanising].** K. TAUSSIG (Stahl u. Eisen, 1931, 51, 885–886).—The presence of  $\text{FeCl}_2$  on the Fe sheets when they are immersed in the Zn bath leads to the formation of Fe–Zn alloy in the bath by interaction between the Zn and  $\text{FeCl}_2$ ; practically no  $\text{FeCl}_2$  is lost by volatilisation, so that it is essential to wash thoroughly the sheets before immersion in the bath. A. R. POWELL.

**Soil corrosion. Rates of corrosion and pitting of bare ferrous specimens.** K. H. LOGAN and V. A. GRODSKY (Bur. Stand. J. Res., 1931, 7, 1–35).—The extent to which soil conditions are responsible for the corrosion of underground pipes has been investigated by determining the rate of loss of wt. of 1300 specimens removed from 70 test stations. The extent of corrosion depends largely on the character of the soil, and whilst the rate appears to vary from year to year, the general tendency is to decrease with time of exposure. The data do not indicate that any one of the commonly used pipe materials is markedly superior to the others for general use underground, although it seems that certain materials have some advantage in certain locations. Several causes appear to be responsible for the corrosiveness of soils, and it is improbable that a single satisfactory method for determining soil corrosiveness can be developed. E. S. HEDGES.

**Internal stresses in brass tubes.** A. KŘEČEK (Z. Metallk., 1931, 23, 178–185).—Experiments are recorded which show that even small stresses produced by finishing operations may lead to season-cracking in ( $\alpha + \beta$ )-brass; a deformation of 5% in fully-annealed brass or of only 1.5% in brass which has had a low-temp. anneal to remove internal stress may be dangerous. A. R. POWELL.

**Properties of drawn wires and the power consumption in wire-drawing.** W. LINICUS (Z. Metallk., 1931, 23, 205–210).—The effect of drawing conditions and the type of dies used on the mechanical properties of Fe and brass wires has been investigated. A. R. POWELL.

**Behaviour of phosphorus and oxygen in copper-tin melts and alloys.** R. FALKENSTÖRFER (Metall-

Wirts., 1931, 10, 130–131; Chem. Zentr., 1931, i, 2259).—When brass containing Cu 85, Sn 8, Zn 5, Pb 2% is melted in a reducing atm., the strength and extensibility diminish rapidly when the P content exceeds 0.12%. With up to 0.15% P the solid solution is homogeneous, but with more than 0.21% P the phosphide eutectic appears. P is not removed from the heated mass at 1100°, when loss of Zn did not exceed 0.3%. The reducing action of P is obvious when oxidation has occurred during fusion.

A. A. ELDRIDGE.

**Welding of monel metal in the construction of chemical apparatus.** R. MÜLLER (Chem. Fabr., 1931, 4, 310–312).—As  $\text{Cu}_2\text{O}$  is sol. in molten monel metal and renders the alloy brittle and less resistant to corrosion, it is essential that welding be carried out under non-oxidising conditions. In  $\text{O}_2$ - $\text{C}_2\text{H}_2$  welding, the gas should be passed through a solution of  $\text{H}_3\text{BO}_3$  in EtOH and a slight excess of  $\text{C}_2\text{H}_2$  should be present in the gas mixture. In arc-welding, the work should be made the negative pole and the welding rod should be coated with powdered Mg–Mn–Si or Ti–Ca alloy. Good welds are also obtained by welding with at. H, using W electrodes. A. R. POWELL.

**Influence of pressure conditions in the oxy-acetylene welding torch on economy and quality of the weld.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 60–64; Chem. Zentr., 1931, i, 2418).—Changes in the mixture ratio in welding torches are due partly to change in pressure owing to rise of temp. and partly to back-pressure. The quality of the weld is unaffected. A. A. ELDRIDGE.

**Reports of investigations: [Canadian] metallic ores.** C. S. PARSONS, J. S. GODARD, A. K. ANDERSON, D. S. HALFORD, and G. B. O'MALLEY (Canad. Dept. Mines, Rept. Ore Invest., 1929, 5–158).—The results of table concentration, ore flotation, cyaniding, and amalgamation tests on numerous samples of Canadian Au, Ag, Sn, Cu, Pb, and Zn ores both simple and complex are recorded, and in most cases suitable methods of treatment are indicated. A. R. POWELL.

**Reports of investigations: [Canadian Mines Laboratory] hydrometallurgical and electrochemical section.** I. Treatment of titaniferous magnetite from Mine Centre for recovery of iron, titanium, and vanadium. R. J. TRAILL, W. R. MCCLELLAND, and J. D. JOHNSTON. II. Separation of copper from molybdenum in molybdenite concentrates. R. J. TRAILL. III. Treatment of zinc concentrates from various Canadian ores. R. J. TRAILL, W. R. MCCLELLAND, and J. D. JOHNSTON. IV. Treatment of mixed concentrate from base-metal sulphide ore. W. E. HARRIS, R. J. TRAILL, and H. C. MABEE. V. Use of coking coals in the metallisation of iron ores. W. R. MCCLELLAND (Canad. Dept. Mines, Rept. Ore Invest., 1929, 178–185, 186–187, 187–191, 192–199, 199–201).—I. An ilmenite ore containing 48.4% Fe, 12.8%  $\text{TiO}_2$ , 0.9%  $\text{V}_2\text{O}_5$ , and 6.4% Si was heated with coal and coke at 1050° for 5 hr. and the reduced product gave a magnetic product containing 61.2% Fe (58.3% metallic Fe),

17.6%  $\text{TiO}_2$ , and 1%  $\text{V}_2\text{O}_5$ . Over 50% of this product was dissolved by  $\text{FeCl}_3$  solution, leaving 99% of the  $\text{TiO}_2$  and 97% of the  $\text{V}_2\text{O}_5$  in a residue containing 20.8% Fe, 35.3%  $\text{TiO}_2$ , and 2.1%  $\text{V}_2\text{O}_5$ . Roasting of this residue with  $\text{NaCl}$  and  $\text{Na}_2\text{CO}_3$  rendered 75% of its V content sol. in  $\text{H}_2\text{O}$ , and the insol. material is available for the extraction of  $\text{TiO}_2$  by heating with  $\text{H}_2\text{SO}_4$ .

II. An ore containing 4.6% Cu as chalcopyrite and 2.8% Mo as molybdenite gave only a poor extraction of Cu on leaching with  $\text{FeCl}_3$ , but after roasting with S in a reducing atm. over 85% of the Cu was extracted with  $\text{FeCl}_3$ ; after leaching with dil.  $\text{HCl}$  to remove Ca and Mg the residue gave a good Mo extraction on roasting and leaching with  $\text{NaOH}$  solution.

III. The results of a preliminary investigation into the formation of ferrites on roasting  $\text{ZnS}$  concentrates containing FeS are recorded. The determining factors in ferrite formation are the roasting temp., the Fe content, and the mineralogical nature of the ore.

IV. A mixed sulphide flotation concentrate containing 30% Zn, 5.5% Cu, 3.4% Pb, 19.4% Fe, and 7.5 oz. (Au + Ag) per ton was roasted in a multiple-hearth furnace and the product leached with dil.  $\text{H}_2\text{SO}_4$  until the solution contained less than 0.5% of free acid. The solution contained 80% of the Zn and 50% of the Cu; it was purified by treatment with Zn dust to ppt. Cu, then with  $\text{MnO}_2$  and  $\text{CaCO}_3$  to ppt.  $\text{Fe}(\text{OH})_3$ , and filtered. The filtrate contained 120–130 g. of Zn per litre and 2 p.p.m. of Ni which was removed by treatment with 0.01% of  $\text{HgSO}_4$ , 0.15% of  $\text{H}_2\text{SO}_4$ , and 0.15% of Zn dust; the purified solution was then electrolysed for Zn, the outflowing liquor containing 3.6% Zn and 12.5%  $\text{H}_2\text{SO}_4$  being returned to the leaching. The residue from leaching containing 17.4% Zn, 8.3% Cu, and 5.9% Pb, was treated by the Waelz process, whereby 97% of the Zn and over 95% of the Pb were recovered in the fume, leaving a residue containing 0.6% Zn, 11.8% Cu, and 15.6 oz. Au + Ag per ton suitable for smelting for the recovery of Cu, Ag, and Au.

V. A mixture of finely-ground Fe ore and coking coal in a 1:1 ratio was heated at 636–946° for several hr. and the products of destructive distillation were removed continuously so as to maintain a slightly reduced pressure in the furnace. The resulting Fe sponge was of good quality after magnetic separation from the silicious residue.

A. R. POWELL.

**Boundary fields of metallography.** K. SCHRÖTER (*Z. Metallk.*, 1931, 23, 197–201).—For the examination of W powder the material is mixed with 3 times its wt. of Cu powder and the mixture sintered in  $\text{H}_2$  in an electric furnace at 1250°. As Cu does not alloy with W the structure of the latter remains unaltered; a section of the sintered mass is embedded in solder, polished in the usual way, and etched with alkaline  $\text{K}_3\text{Fe}(\text{CN})_6$  solution. To examine the mechanism of sintering of W bars, samples of the bar at various stages of the sintering process are immersed in molten Cu, which fills up the pores and thus facilitates polishing of a section; in this way grain growth during sintering may be readily followed. The structure of W wires may be examined by coating them electrolytically with Cu, embedding the wire in asphalt, and polishing as usual; the surface Cu is then dissolved in dil.  $\text{HNO}_3$  and the remainder is

blackened by  $(\text{NH}_4)_2\text{S}$  solution. In this way the structure of the W wire shows up sharply against the black background; a similar device serves for the examination of thin Ni and Ni-Fe alloy wires. Methods for detecting hair-cracks in and the riffling of glass tubing etc. are also described.

A. R. POWELL.

**Effect of iron content of blende on its flotability.** L. KRAEBER (*Mitt. Kaiser Willh.-Inst. Eisenforsch.*, 1930, 12, 343–352; *Chem. Zentr.*, 1931, i, 2257).—Max. flotability is reached when the  $p_{\text{H}}$  of the  $\text{H}_2\text{O}$  is 6.0–7.0, the optimal val. being the lower the higher is the Fe content of the blende. The varying rapidity of flotation is not attributable to the Fe content, neither is there a definite relation between the effects of KCN and  $\text{CuSO}_4$  and the Fe content.

A. A. ELDRIDGE.

**Effect of small quantities of addenda on the behaviour of soft lead towards acids.** A. BUCKHARDT (*Metall-Wirts.*, 1931, 10, 181–187; *Chem. Zentr.*, 1931, i, 2258).—Min. strength is rapidly reached by storage for 1 month at 70–200°. Except with alloys containing >0.1% Ag or 0.005% Li, the elongation also falls. On exposure to  $\text{H}_2\text{SO}_4$  the metal exhibits no change in strength or elongation. Fe, Sb, Sn, Te, Cu, and Ni decrease, whilst Li, Ag, Zn, Cd, and Bi increase, the corrosion. Tl and Hg have little or no effect. Pb containing Cu, Ni, or Te is least resistant towards  $\text{HCl}$ , pure Pb being most resistant. Moist  $\text{Cl}_2$  causes greater corrosion than  $\text{HCl}$ .

A. A. ELDRIDGE.

**Determination of lead in slags.** W. WITTER (*Chem.-Ztg.*, 1931, 55, 550).—The slag is decomposed with 1:1  $\text{HCl}$  and  $\text{NH}_4\text{F}$ , the solution boiled to expel  $\text{SiF}_4$ , and any sulphide oxidised with  $\text{KMnO}_4$ . Pb is then separated as  $\text{PbS}$ , which is converted into  $\text{PbSO}_4$  and the analysis finished by the usual  $(\text{NH}_4)_2\text{MoO}_4$  titration.

A. R. POWELL.

**Heat conductivity, electrical conductivity, and Lorenz value of some light alloys.** W. MANNCHEN (*Z. Metallk.*, 1931, 23, 193–196).—The thermal and electrical conductivities and the Lorenz values for Al and Mg and for 16 Al and 20 Mg light alloys at 87°, 273°, 373°, and 476° are tabulated. The thermal conductivities of the alloys are all lower than that of the basic metal, but the temp. coeffs. are positive for the alloys and negative for the pure metals. Addition of Mn or Si to Mg results in a considerable decrease in both conductivities; this is explained on the assumption that a limited series of solid solutions is formed, although previous workers have stated that Mn and Si are completely insol. in solid Mg. The Lorenz value for alloys is usually much less than for the pure metals, but in Cu–Al and Mn–Mg alloys the vals. are approx. the same as for Al and Mg respectively.

A. R. POWELL.

**Surface treatment and electroplating of aluminium.** ANON. (*Chem.-Ztg.*, 1931, 55, 579–580).—Al articles are freed from a coating of oxide by treatment with  $\text{NaOH}$  solution followed by dil.  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ – $\text{HNO}_3$  mixture or by treatment with a  $\text{NaOH}$ – $\text{NaCl}$  mixture. Al previously cleaned may be polished by steel balls in revolving drums. After cleaning, Al can be lacquered in the same way as other metals. There is great difficulty in producing a coherent coating on Al by electroplating, and such a coating cannot be relied

on as a complete protection. Articles which are not to be wetted can be electroplated in the same way as other metals, but the oxide surface layer must be removed. Coatings of Fe, Cu, Zn, Cd, Cr, and Ni can be obtained directly and used for the superposition of other metals. Ni may be deposited from a bath containing glycerin with a c.d. of 0.5 amp./sq. dm. Al may be sprayed with other metals by the Schoop process.

C. IRWIN.

**Blast-furnace coke. Welding with  $C_2H_2-O_2$  mixtures.**—See II. Metals and milk.—See XIX.

## PATENTS.

**Furnace [for brazing, annealing, etc.].** (A) C. L. IPSEN and J. L. MCFARLAND, (B) J. L. MCFARLAND, ASSRS. to (A, B) GEN. ELECTRIC CO. (U.S.P. 1,789,177 and 1,789,187, 13.1.31. Appl., [A] 29.11.27, [B] 19.4.29).—In (A) a furnace, and in (B) a method of operating it, are described, the object being the annealing or brazing of metals in an explosive atm. The furnace is heated nearly up to the working temp. while filled with air, a non-explosive gas, *e.g.*,  $N_2$  or  $N_2-H_2$  mixture, is used to sweep out the air, and finally pure explosive gas, *e.g.*,  $H_2$ , is passed in. In cooling, the reverse process is adopted. The furnace comprises an elongated shell suitably lined and provided with openings at the ends through the bottom for inlet and outlet (respectively) of goods which are placed on trucks and pushed longitudinally through by a ram. Near the inlet a zone is electrically heated, the remainder being for cooling; the zones for the various gases are divided off by transverse partitions which fit closely to the trucks, and it is arranged that, whenever possible, there is a truck in each opening.

B. M. VENABLES.

**Annealing or other heat treatment of metals, more especially iron and steel.** N. G. ELFSTROM (B.P. 352,677, 10.6.30).—The metal is heated in a closed chamber, the atm. of which is provided by blast-furnace, producer, or other fuel gas passing through glowing C heated electrically. Control of the temp. of the C provides either a carburising, inert, or decarburising atm.

C. A. KING.

**Agglomeration of finely-divided ferruginous ores, concentrates, etc.** A. FOLLIET and N. SANDERICHIN (B.P. 352,832, 3.10.30. Fr., 30.10.29).—A mixture of ore and fuel is fed forward in a thin layer and in a certain zone meets penetrating jets of air at 650–800° which promotes combustion.

C. A. KING.

**Treatment of metallurgical slimes.** METALLGES. A.-G. (B.P. 353,212, 27.6.30. Ger., 28.6.29).—Slimes are mixed with solid fuel or other coarse agglomerate which favours the drainage of contained  $H_2O$ , and are then transferred direct to the blast-roasting process.

C. A. KING.

**Conversion of metallic ores.** H. EDWARDS, O. J. PARKER, and R. ARNOT (B.P. 354,098, 13.9.30).—The ore, with or without a preliminary mechanical concentration treatment, is heated at a temp. below that at which sintering occurs with a powdered carbonaceous reducing agent and with a compound which combines with or decomposes  $H_2O$ , *e.g.*,  $CaO$ ,  $CaC_2$ , or  $CaCN_2$ . Thus an ore containing  $CuCO_3$  in a basic gangue is heated with

coal and  $CaCN_2$  at 400–450° and the product is treated magnetically to separate Fe, leaving a residue from which Cu can be recovered by wet dressing.

A. R. POWELL.

**Moulding processes [for metals] and moulds made thereby.** J. B. DURAND (B.P. 351,592, 3.4.30. Ger., 4.4.29).—Moulds or cores for use in metal casting are made by the process described in B.P. 319,777 (B., 1931, 354) except that artificially produced sand or old sand, produced by grinding used moulds or cores made by the same process, is used. [Stat. ref.]

A. R. POWELL.

**Granulation of liquid slag.** A. L. MOND. From VEREIN. STAHLWERKE A.-G. (B.P. 351,591, 3.4.30).—The liquid slag is run down a channel in which it meets jets of  $H_2O$  directed at various angles into the slag stream and the mixture of hardened slag and  $H_2O$  falls into a centrifugal machine comprising a conical chamber with a rotating central axis carrying arms the length of which increases from top to bottom.

A. R. POWELL.

**Manufacture of sponge iron.** I. RENNERFELT and I. C. S. G. BOHM (B.P. 353,180, 12.6.30).—Finely-divided Fe ore (concentrates) is mixed with approx. the theoretical quantity of solid reducing agent (high-grade charcoal) and heated in coke-oven chambers, reducing gas being admitted during the later stages of reduction. The sponge Fe is then pushed out of the chamber, with protection from the atm.

C. A. KING.

**Production of high-strength grey cast iron.** INTERNAT. NICKEL CO., INC., ASSEES. OF F. B. COYLE (B.P. 352,510, 4.1.30. U.S. 5.1.29).—A charge consisting of >60% of steel, >12% of coke, and scrap Fe is melted and graphitised by adding 2.5–7% of Ni or Si, preferably >5.5% Ni. The tensile strength of the product exceeds 50,000 lb./sq. in.

C. A. KING.

**Manufacture of material suitable for resisting rifle bullets and other projectiles etc.** (SIR) R. A. HADFIELD (B.P. 352,548, 12.4.30).—Steel containing C 0.35–0.6%, Mo or W 0.25–1.5%, Mn 0.25–3%, and Si 0.25–2.5% is annealed, rolled, reheated to 875–900°, quenched, reheated to 530–590°, and cooled slowly to remove strains. The alloy may be tempered from about 650°.

C. A. KING.

**Cobalt high-speed tool steel.** OESTERR. SCHMIDTSTAHLWERKE A.-G. (B.P. 352,951, 14.4.30. Addn. to B.P. 343,508; B., 1931, 418).—Co steels containing not more than 1.5% Cr, preferably <0.5%, are claimed. [Stat. ref.]

C. A. KING.

**Magnetic [iron-nickel] material.** J. E. HARRIS, ASSR. to BELL TELEPHONE LABS., INC. (U.S.P. 1,790,704, 3.2.31. Appl., 24.3.30).—Dust for the manufacture of magnetic cores is made by grinding an alloy of 25% Co, 27% Fe, 45% Ni, 3% Sn, and 0.0625% Bi, or an alloy of 25% Co, 26% Fe, 45% Ni, 4% Mo, and 0.25% Pb, until at least 90% passes 120-mesh.

A. R. POWELL.

**Iron-titanium alloys.** W. KROLL (B.P. 352,964, 7.2.30. Ger., 16.2.29).—Alloys with >50% Fe, 0.5–8% Ti, up to either 40% Ni or 8% Mn, and additions of up to 25% Cr, 15% Co, 5% Mo, 3% V, 4% Si, or 25% W are quenched between 700° and 1350° and artificially aged by annealing at 320–600°.

C. A. KING.

**Production of corrosion-resisting ferrous alloys.**

HALL & PICKLES, LTD., and J. SMITH (B.P. 351,532, 26.3.30).—Fe or mild steel containing 0.25–4% Si, 0.1–2% Cu, and 0.1–2% As is claimed. The Cu and As are added when the charge is melted down and the Si as Fe-Si when the metal has been refined. A small amount of Al is added just before casting to deoxidise the charge. A. R. POWELL.

**Joining zinc-aluminium alloys to steel.** H. C. HALL (B.P. 351,977, 25.1.30).—The steel surface is galvanised by the hot-dipping process and after heating to 250° the galvanised surface is covered with a layer of molten Al-Zn alloy containing 70–99% Zn. A. R. POWELL.

**Composition for treating metals [iron to prevent rusting].** E. K. WALLACE, ASSR. to RUST PREVENTION CHEM. CO. (U.S.P. 1,790,960, 3.2.31. Appl., 7.3.29).—The pickling solution comprises a mixture of 42.5%  $H_3PO_4$ , 56%  $H_2O$ , and 1.5% casein. A. R. POWELL.

**Continuous copper-melting furnace.** R. P. HEUER (B.P. 351,740, 28.6.30).—The flue gases from the melting furnace are separated into two parts, one of which is used for preheating the air for combustion and the other for preheating the incoming charge to the furnace to a temp. just below the m.p. This is effected by feeding the charge on to conveyor which moves along a circular track in the flue. A. R. POWELL.

**Copper-silicon-zinc alloys.** HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 352,639, 19.5.30. Ger., 13.2.30).—Castings of high tensile strength contain Cu 65–80%, Si 2–6%, with one or more of the metals Al, Mn, Co, Fe, Pb, Ni, Cr, Zr, W, Mo, and Ti to the limit of 0.1–3%. C. A. KING.

**Reclaiming and re-using metallic scrap mixtures [copper-tungsten or -molybdenum welding rods].** FORD MOTOR CO., LTD., ASSEES. OF R. H. MCCARROLL and G. VENNERTHOLM (B.P. 353,377, 4.2.31. U.S., 4.2.30).—The scrap ends of Cu-Mo or Cu-W welding rods are ground in a disc or ball mill to pass 20–100-mesh and the powder is reduced at 1000° for 15 min. in  $H_2$ . The resulting mixture of W or Mo grains coated with Cu is pressed into electrodes which are sintered until coherent and then used again for welding. A. R. POWELL.

**Reduction of zinciferous materials.** NEW JERSEY ZINC CO., ASSEES. OF E. H. BUNCE (B.P. 353,371, 29.12.30. U.S., 30.12.29).—A mixture of Zn ore and powdered coal is fed continuously into Chilean mills to which tar or pitch is added to bind the mixture, which then passes to a briquetting machine. The briquettes fall on to a conveyor belt which transfers them to a coking furnace, the hot discharge from which is fed automatically to vertical retorts of the gas-fired recuperator type. The exhaust hot gases from the recuperator are mixed with a quantity of unburnt fuel gas and the mixture is used to heat the coking furnace. A. R. POWELL.

**Reduction of zinciferous materials containing lead and other contaminating metals.** L. MELLSHER-JACKSON. From NEW JERSEY ZINC CO. (B.P. 352,005, 3.2.30).—Briquettes of Zn ore and coke are fed downwards through a long vertical retort the lower

half only of which is heated to a temp. at which the Zn distils, whilst the upper half is heat-insulated so that an appreciable length of the briquette column is maintained at 800–900°, whereby the Pb, Bi, etc. which distil in the lower zone are condensed again in the charge and a relatively pure Zn vapour passes to the condensers. A. R. POWELL.

**Refining aluminium, magnesium, and their alloys.** H. C. HALL (B.P. 352,199, 5.6.30).—Molten Al or Al alloy is treated with a tablet, stick, or lozenge of fused anhyd.  $ZnCl_2$  coated with wax, grease, oil, or varnish which prevents it from deliquescing during storage. Mg alloys are treated with  $MgCl_2$  preparations which have been similarly treated. A. R. POWELL.

**Manufacture of aluminium alloys.** S. JUNGHAUS (B.P. 353,891, 29.1.30. Ger., 31.1.29).—Alloys of Al with 5–20% Cu, 1–3% each of W, Mn, and Cr, 0.1–3% Ni, and 0.1–2% Na, K, Li, Be, Mg, Ca, Sr, or Ba are claimed. In addition, 0.1–3% Co, <5% Ag, <10% Cd, and <3% V or Mo may be added. A. R. POWELL.

**Melting flux for purifying aluminium, its alloys and other non-ferrous metals.** F. DUBBERLEY, SEN. and JUN., C. A. DUBBERLEY, and E. H. TYSON (B.P. 353,867, 26.3. and 12.4.30).—The molten metal is treated with a flux comprising (a) 10%  $K_4Fe(CN)_6$ , 2.5% KOH, 10%  $Na_3AlF_6$ , 45%  $CaF_2$ , and 32.5% NaCl, or (b) 18%  $K_4Fe(CN)_6$ , 2% KOH, 48%  $CaF_2$ , and 23% NaCl. A. R. POWELL.

**Metallic [aluminium] protective coating.** S. U. MCGARY, W. F. ROGERS, and S. GILL, ASSRS. to GILL PRODUCTION CO. (U.S.P. 1,790,643, 3.2.31. Appl., 11.10.29).—Timber or metal articles are protected from corrosion by coating them with a thin sheet of Al (0.0005–0.002 in. thick) which is held on to the article by means of a layer of a plastic adhesive material. A. R. POWELL.

**Lead alloy.** R. J. SHOEMAKER, ASSR. to S. & T. METAL CO. (U.S.P. 1,791,148, 3.2.31. Appl., 2.8.28).—A Pb alloy containing 0.01–0.03% Li, 0.1–0.3% Ca, 0.25–0.5% Sn, and 0.02–0.1% Al is claimed. A. R. POWELL.

**Hard-metal compositions.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF S. L. HOYT (B.P. 354,028, 9.7.30. U.S., 16.7.29).—A mixture of 100 pts. of WC and 15 pts. of U in the form of fine powder is heated in a C mould at 1500–1600° and a pressure of 1000 lb./sq. in. is applied to compress the mixture into a sintered slug suitable for use, after shaping, as a cutting tool. A. R. POWELL.

**Recovery of refractory metals from ores [zirconium from zircon].** C. V. IREDELL, ASSR. to WESTINGHOUSE LAMP CO. (U.S.P. 1,791,272, 3.2.31. Appl., 7.12.28).—Finely-powdered  $ZrSiO_4$  is fused with coke, graphite, or charcoal at 1900–2500°, whereby a mixture of SiC and ZrC is obtained and most of the Fe is volatilised. The product is digested with dil.  $HNO_3$  (1:1) to obtain a solution of  $Zr(NO_3)_4$  and a residue of SiC. A. R. POWELL.

**Purifying or refining non-ferrous metals and alloys.** D. R. TULLIS and P. OAKLEY (B.P. 352,671,

4.6.30).—Vapour of V halide is introduced into a molten metal, e.g., Al, or its alloys. C. A. KING.

**Apparatus for treatment of ores [especially those containing precious metals].** N. LESLIE (B.P. 352,931, 10.3.30. Austral., 11.3.29).—The apparatus comprises a vertical chamber with a conical bottom, an adjustable upcast pipe in the vertical axis of the chamber, and a circulating pipe entering the chamber at the point of the cone and being directed in the style of an injector into the upcast pipe so that by passing compressed air through the jet the contents of the chamber are thoroughly agitated. The apparatus is suitable for amalgamation, cyanidation, or flotation processes. A. R. POWELL.

**Cathodes for the electrolytic refining of metals.** H. WADE. From UNITED STATES METALS REFINING Co. (B.P. 352,104, 9.4.30).—Cathodes for the electrolytic refining of Cu consist of thin Cu sheets stiffened by means of horizontal corrugations at the top and bottom and vertical corrugations down the middle. Corrugations and fastening of the suspending loops to the sheet are made in one stamping operation. A. R. POWELL.

**Electrolytic production of metals, particularly magnesium.** A. C. JESSUP (B.P. 351,510, 28.2.30).—Electrolysis is carried out in a square tank with a central anode, whereby the circulating currents set up in the  $MgCl_2$  electrolyte are sufficient to cause the deposited Mg on the cathodes to become detached and to be driven into cooled recesses which are provided at the four corners of the tank. The metal is protected against the  $Cl_2$  evolved at the anode by surrounding the latter with partitions which dip just below the surface of the electrolyte. A. R. POWELL.

**Electrolytic production of magnesium.** I. G. FARBENIND. A.-G. (B.P. 351,503, 20.1.30. Ger., 6.2.29).—The bath is provided with a series of plane anodes and cathodes extending nearly across the whole cross-section and separated from one another by non-conducting partitions extending less than half-way down the bath, the partitions being closer to the cathodes than to the anodes and forming a gas-collecting chamber round the anode. The lower sides of the partitions are bevelled upwards away from the anode and the cathodes or extensions thereof are disposed just below these bevelled portions. The bath is replenished with fused  $MgCl_2$  free from suspended C and  $SiO_2$ . A. R. POWELL.

**Electrolytic zinc bath.** G. BIANCO (U.S.P. 1,791,082, 3.2.31. Appl., 20.4.27).—Zn is melted with 1–15% Hg and the granulated alloy is dissolved in a solution containing  $H_2SO_4$ ,  $(Al, Fe)_2(SO_4)_3$ ,  $H_3PO_4$ , and NaOH to give an approx. neutral solution of Zn salts. A. R. POWELL.

**Operation of furnaces for brazing, annealing, etc.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. L. MCFARLAND (B.P. 353,062, 16.4.30. U.S., 19.4.29).—See U.S.P. 1,789,187; preceding.

**Magnetic materials [nickel-iron alloys].** ELECTRICAL RESEARCH PRODUCTS, Assees. of G. W. ELMEN (B.P. 352,019, 31.3.30. U.S., 2.4.29).—See U.S.P. 1,757,178; B., 1930, 1033.

**Purification of magnesium or magnesium alloys.** C. ARNOLD. From DOW CHEM. Co. (B.P. 346,271, 5.12.29).—See U.S.P. 1,754,788; B., 1930, 914.

**Furnaces [for heating leaf springs].** BURDONS, LTD., and J. McQUARRIE (B.P. 353,699, 26.7.30).

**Tubular furnace. Hg boilers.**—See I. Welding electrodes. Separation of foundry sand. Determining K in spaces. Coated Cu articles.—See XI. [Adhesive] reaction products of rubber.—See XIV.

## XI.—ELECTROTECHNICS.

**Electric furnace of fluorite.** A. DAMIENS and L. DOMANGE (Compt. rend., 1931, 192, 1711–1713; cf. B., 1931, 720).—The  $CaF_2$  porcelain being permeable to gases at high temp., the furnace is constructed of pure fluorite in the shape of a tube wound with nichrome wire, and placed in an Fe tube with the intervening space packed with powdered fluorite. The ends are water-cooled, and one is closed with a plate of transparent fluorite; temp. is determined by radiation. Work with  $F_2$  can be carried on at  $1000^\circ$ .

C. A. SILBERRAD.

**Synthetic galena as a radio-detector.** W. OGAWA (Proc. World Eng. Cong., 1929, 31, 291–304).— $PbS$  was precipitated from  $Pb(OAc)_2$  acidified with  $AcOH$ , filtered, washed, dried, and fused either alone or with other substances.  $Ag_2S$  and  $Tl_2S$  was found to improve sensitivity, the optimum addition of the former being 7.5%. The effect is associated with reduction to Ag. By moulding with solutions, salts of alkali metals were found to be equally effective. For use with a galena crystal, a sharp needle of electropositive metal or alloy is preferable to Pt. The direction of rectification is determined by the chemical composition of the contact portions of both electrodes, and is of a relative nature. Synthetic galena is a better conductor than the natural crystal, but resistance is increased by addition of Ag. Determinations of thermoelectric powers of galena and other mineral crystals used as detectors give no support to any thermoelectric theory of their action. The author's theory is that rectification is caused by the difference of electron emissions from the pairs of substances which make a couple. This difference must be as large as possible, and the resistance of the electrodes must be great enough to minimise metallic conduction. The emission of electrons from a galena crystal has been demonstrated and other evidence in support of the theory is given. C. IRWIN.

**Electrification in coal-dust clouds.**—See II. Welding of monel metal. Conductivity of light alloys. [Resistance of] Al alloys. Plated Al.—See X.

## PATENTS.

**Electric furnaces and methods for making glass and the like.** J. K. B. RAEDER (B.P. 350,930, 6.3.30).—The charge is fed into a vertical chamber comprising an upper melting zone and a lower refining zone with spaced electrodes projecting into the molten glass at the junction of the two zones. The refining zone is connected at its lower end with a conditioning chamber which permits the escape of gas bubbles from the



molten glass, and is also independently electrically heated.

A. R. POWELL.

**Welding electrodes and coatings therefor.** P. S. D. SMITH. From HOLLUP CORP. (B.P. 352,086, 5.4.30).—The sheath comprises a flexible material, *e.g.*, licorice tubes or macaroni, with which is incorporated ground glass as a flux. The tube is split longitudinally so that it can be fitted round the electrode as and when required. The sheaths are specially adapted for covering mild steel welding rods.

A. R. POWELL.

**Production of negative electrodes for electric accumulators.** SOC. ITAL. BATTERIE ELETTICHE CELLINO (B.P. 352,604, 2.5.30. Ger., 2.5.29).—Amalgamated Zn is deposited upon a metallic sheet, *e.g.*, of Pb, from an electrolyte containing a colloidal alkaline silicate, *e.g.*, Na silicate and  $\text{HgSO}_4$ , in which a Zn anode is immersed.

J. S. G. THOMAS.

**[Preventing loss of electrolyte in] electrical accumulators, polarisation cells, electrolytic rectifiers and condensers.** BRITANNIA BATTERIES, LTD. From ACCUMULATOREN-FABR. A.-G. (B.P. 352,394, 1.4.30).—Gas-discharge tubes lead from the upper part of the accumulator vessel etc. to the bottom of an adjacent protecting vessel partly filled with oil or other liquid or with granular absorbent or porous material, *e.g.*, glass wool, pumice stone, etc.

J. S. G. THOMAS.

**Electrolytic purification of aqueous solutions.** J. BILLITER (B.P. 352,109, 11.4.30. Ger., 12.4.29).—The removal of salts from  $\text{H}_2\text{O}$  is effected by electrolysis in a cell divided by diaphragms in such a way that no electro-osmotic flow through the diaphragms occurs. This is effected by the use of a mean p.d. of 6–18 volts per liquid compartment and by weakening or neutralising the membrane potentials by suitably directed liquid movements through the diaphragms, care being taken that the difference of level of liquid in the various compartments is sufficient to drive the liquid by hydrostatic pressure in the direction through the diaphragms which the membrane potential would drive it.

A. R. POWELL.

**Electrical precipitation apparatus [for gases].** H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,790,961, 3.2.31. Appl., 12.6.29. Austral., 1.8.28).—Apparatus comprising a number of precipitation units with a main flue extending alongside, and means for closing down one or more units of the series and diverting the gas stream to the next following or to the delivery outlet, is claimed.

J. S. G. THOMAS.

**Electrical precipitator [of suspended particles from gases].** H. A. WINTERMUTE, Assr. to RESEARCH CORP. (U.S.P. 1,791,338, 3.2.31. Appl., 12.4.27).—Baffles projecting from the collecting electrodes form pockets of "dead" gas along the surface of the electrodes, and means are provided for moving the baffles and for removing adherent precipitated material in the pockets.

J. S. G. THOMAS.

**[Controlling the humidity of gases in the] electrical purification of gases, particularly waste furnace gases.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 353,025, 17.4.30. Ger., 19.4.29).—A device indicating the gas temp. is adjustably connected with a lever controlling the moistening device.

J. S. G. THOMAS.

**Magnetic separation of materials [iron from foundry sand].** H. H. THOMPSON, A. E. DAVIES, and W. E. BOX (B.P. 352,172, 21.5.30).—The sand is elevated by a bucket elevator passing over an upper magnetic pulley so arranged that the sand is discharged at the top and the magnetic material at a slightly lower level. The buckets are made of Fe which is cut away at the belt side and fingers of Fe protrude from the belt and from the sides of the bucket into the interior thereof.

A. R. POWELL.

**Electric-discharge apparatus [with heated electrodes].** GEN. ELECTRIC CO., LTD., Assees. of PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 353,253, 24.7.30. Ger., 30.8.29).—The shell enclosing each electric-heater winding for heating the separate electrodes is made of a sintered mixture of refractory metal and one or more non-metallic substances of high electronic emissivity. Thus the shell may be made of W (70–85%) and a mixture of BaO and CaO (30–15%).

J. S. G. THOMAS.

**[Cold electrodes for] gaseous electric-discharge devices.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of T. E. FOULKE (B.P. 352,756, 28.7.30. U.S., 27.7.29).—"Cold" electrodes are coated with a compound of an alkali or alkaline-earth compound of low work function, *e.g.*, KOH, which is subsequently reduced partly to the oxide and partly to the metal.

J. S. G. THOMAS.

**Revivifying used electric luminous discharge tubes.** G. CLAUDE (B.P. 353,135, 15.5.30. Fr., 21.5.29).—Gases absorbed by the envelope and electrodes are driven out by heat.

J. S. G. THOMAS.

**[Sealing-in wire for] electric incandescence lamps and electric-discharge tubes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 353,335, 15.10.30. Holl., 30.11.29).—A single-crystal W wire is claimed.

J. S. G. THOMAS.

**Photoelectric cells.** WESTINGHOUSE LAMP CO., Assees. of H. C. RENTSCHLER (B.P. 353,554–5, 6.5.30. U.S., 6.5.29).—Photoelectric cells comprise in (a), or in (b) in parenthesis, an envelope constructed wholly (wholly or partly) of a material and/or made of such thickness as to be permeable to radiation of wave-length less than about 4500 Å. (3900 Å.), a cathode composed of one of the metals of the Ce group (Th), preferably deposited upon Au and photosensitive only to the same radiation, and an anode.

J. S. G. THOMAS.

**Photoelectric cells [sensitive only to ultra-violet radiation].** WESTINGHOUSE LAMP CO., Assees. of H. C. RENTSCHLER and D. E. HENRY (B.P. 353,981, 23.5.30. U.S., 12.6.29).—Cells comprising a cathode of U, preferably deposited upon a film of Au coating the envelope which is transparent to radiation of wave-length about 2900 Å., is claimed.

J. S. G. THOMAS.

**Photoelectric cells.** ELECTRICAL RESEARCH PRODUCTS, INC., Assees. of (A) A. R. OLPIN, and (B) G. R. STILWELL (B.P. 352,012 and 352,057, [A] 27.3.30, [B] 29.3.30. U.S., [A] 6.4.29, [B] 18.4.29).—(A) The cell contains an electrode coated with Co the light-sensitivity of which is increased by exposing it in vac. to the action of S vapour in the presence of a trace of  $\text{H}_2\text{O}$  vapour with or without a small quantity of the

vapour of an organic dye, preferably of the cyanine group. (B) Na is used as the light-sensitive metal; after one treatment with S vapour, air is admitted to the cell, which is then again evacuated and a second layer of Na and S is formed on the first.

A. R. POWELL.

**Apparatus for determining the potassium content in spaces containing potassium.** W. KOLHORSTER (B.P. 351,266, 8.7.30. Addn. to B.P. 340,231).—The ionisation set up by the  $\gamma$ -rays emitted by the K are measured by means of an electrical device including a thermionic valve circuit to amplify the current impulses and a rotating impulse relay in the form of a rotative selector as used in automatic telephony.

A. R. POWELL.

**[Manufacture of] piezo-electric substances.** P. FREEDMAN and W. J. RICKETS (B.P. 352,419, 7.4.30).—Preferentially-oriented hemihedral crystals of dielectric material are deposited from a liquid under the combined influence of an electrical field, preferably compounded of direct and alternating components, and high-frequency alternating mechanical stresses. (Cf. B.P. 311,055; B., 1929, 649.)

J. S. G. THOMAS.

**Manufacture of electric insulators.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 353,632, 13.6.30. Ger., 13.6.29. Addn. to B.P. 350,429; B., 1931, 767).—The heat treatment (described previously) for hardening the resin consists in contacting the strip with a heated, rotatable drum or roller.

J. S. G. THOMAS.

**Coating of articles [copper wires with insulating compositions].** ELECTRICAL RES. PRODUCTS, INC., Assees. of T. K. COX (B.P. 353,911, 29.4.30. U.S., 8.6.29).—Tinned Cu wire is treated with oleic, stearic, or palmitic acid so as to form a film of Sn salt on its surface. A layer of rubber insulating material is then extruded on the surface of the wire and subsequently vulcanised whereby it adheres strongly to the wire owing to the binding action of the Sn salt.

A. R. POWELL.

**Method of and composition for cleaning and vulcanising materials [used in insulating electrical conductors].** ELECTRICAL RES. PRODUCTS, INC., Assees. of F. S. MALM (B.P. 353,437, 22.4.30. U.S., 30.4.29).—Ends of submarine cables etc. to be joined are washed with a solution composed of a solvent, e.g.,  $\text{CS}_2$  and/or  $\text{CCl}_4$ , a vulcanising agent, e.g., S, and an ultra-accelerator, e.g., Zn butyl xanthate or one of the Zn alkyl xanthate or dithiocarbamate group.

J. S. G. THOMAS.

**[Winding of coreless] electric induction [crucible] furnaces.** ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of C. S. WILLIAMS (B.P. 353,270, 6.8.30. U.S., 14.8.29).

**Thermo-electric batteries.** J. NEALE (B.P. 353,521, 25.4.30).

**[Grinding of] piezo-electric [quartz] crystals.** ELECTRICAL RES. PRODUCTS, INC., Assees. of R. A. HEISING (B.P. 351,940, 31.3.30. U.S., 2.4.29).

**Manufacture of loaded electrical conductors.** ELECTRICAL RES. PRODUCTS, INC., Assees. of W. M. BISHOP (B.P. 353,472, 24.4.30. U.S., 6.6.29).

**Radiation pyrometer. Safety mixers. Determining m.p. Examining cryst. bodies.**—See I. Porcelain.—See VIII. Fe-Ni alloy Cathodes for refining metals. Mg. Zn.—See X. Photographic emulsion for electronic discharge.—See XXI.

## XII.—FATS; OILS; WAXES.

**Glycerin analysis committee report.** J. T. R. ANDREWS (Oil & Fat Ind., 1931, 8, 297—301).—The apparent glycerol content (of conc. glycerins), as determined by the dichromate method, is only 0.3% (about the experimental mean error) lower than that obtained from sp. gr. (Bosart and Snoddy tables) determinations; a bigger discrepancy (1.1% low) between the values from the acetin (I.A.M. method) and the sp. gr. figures is confirmed, and appears to be due to a defect in the acetin method (? loss of glycerol during saponification).

E. LEWKOWITSCH.

**Susceptibility of fats to oxidative rancidity.** D. P. GRETTE and R. C. NEWTON (Oil & Fat Ind., 1931, 8, 291—294).—A weighed amount of fat is distributed over filter paper and heated at 100° in a standardised air current; the volatile products are collected in acid permanganate, the amount of reduction being measured by titration. Great care in manipulative detail is essential for reproducibility.

E. LEWKOWITSCH.

**Absorption and desorption of water from individual soaps.** E. L. LEDERER (Allgem. Oel- u. Fett-Ztg., 1931, 28, 207—210).—The theoretical implications of the experiments of Godbole and Joshi (*ibid.*, 1930, 27, 76) on the adsorption of  $\text{H}_2\text{O}$  by dry soaps are discussed; inverse experiments on the drying out of aq. soap solutions similarly show a two-stage process, part ( $\frac{2}{3}$ ) of the  $\text{H}_2\text{O}$  being loosely held (straight-line desorption), whilst part ( $\frac{1}{3}$ ) is tenaciously retained in the intermicellar spaces. Hence it is to be expected that curd soaps should dry out less readily than cold-process (or soft) soaps. Alcohol-soap solutions behave similarly in type, but the permanation coeff. is so much greater that the soap-alcohol gel must be practically free from retaining spaces.

E. LEWKOWITSCH.

**Drying oils. XV. Oxidation of linseed oil up to gelation.** J. S. LONG and W. S. W. McCARTER (Ind. Eng. Chem., 1931, 23, 786—791; cf. B., 1931, 401).—The gelling time and  $\text{O}_2$  absorption of linseed oil were studied, the following factors being varied: temp., acid val. (admixture of linseed oil acids), complexity (heat-bodied oils and different esters of linseed oil acids), unsaturation, presence of driers and inhibitors. Gelation is shown to occur when a certain degree of complexity or polarity of the mols. is reached, oxidation being a contributory factor, although not reaching completion. Free fatty acids unite with linseed oil, aiding gelation although diminishing  $\text{O}_2$  absorption. Elevation of temp. and presence of driers accelerate one or more reactions leading to gelation (e.g., Pb has a specific associating function), whilst inhibitors merely retard oxidation. The partition of  $\text{O}_2$  between gel and volatile products and the composition of the latter are also recorded.

S. S. WOOLF.

**Coagulation and analysis of stand oils containing tung oil.** H. WOLFF and I. RABINOWICZ (Chem. Umschau, 1931, 38, 213—216).—Erratic results were obtained by the modified (and improved) Scheiber  $\text{SnCl}_4$  method (Farbe u. Lack, 1930, 513) for stand oils and varnishes containing raw and/or bodied tung oil: even 50% mixtures of the latter with rape and arachis oils gave values 43% and 37%, respectively. The coagulate obtained contains other acids (including stearic acid) besides eleostearic acid.

E. LEWKOWITSCH.

**Re-liquefaction of coagulated tung oil.** R. BÜRSTENBINDER (Chem. Umschau, 1931, 38, 229—230).—Recovery of overheated tung oil may be possible if condensation (cf. B., 1930, 217) has not proceeded too far; alcoholic HCl, raw oil, etc. assist resolution by trans-esterification.

E. LEWKOWITSCH.

**Specifications for oil-extraction solvents.** R. H. FASH (Oil & Fat Ind., 1931, 8, 311—312, 317).—The amount of extract obtained (e.g., from cottonseed) increases with the temp. of extraction. A solvent of pentane type, containing a min. of isopentane, isohexane, and hexane (initial b.p. between 35° and 40°, at least 95% distilling below 55° and not over 85% below 40°), is preferred. Cottonseed extracted at 80° yielded a solid dark resinous material on re-extraction in the usual manner.

E. LEWKOWITSCH.

**Sandal-seed oil.** M. SREENIVASAYA and N. NARAYANA (Proc. XV Indian Sci. Cong., 1928, 160).—The oil (50—55%) thickens in light; at 130—140° S is absorbed and a rubber-like compound is obtained. The oil had  $n_D^{20}$  1.4790,  $d_4^{30}$  0.9304, I val. 130—140, sap. val. 185—195, acid val. 20—25, Helmer val. 96.97, Reichert-Meißl val. 1.5, Polenske val. 0.2—0.3; mol. wt. of fatty acids 288.5.

CHEMICAL ABSTRACTS.

**Some lesser known Brazilian palm oils.** I. F. W. FREISE (Chem. Umschau, 1931, 38, 216—219).—The botanical identity, yields, application, and consts. of the oils from 34 native varieties are tabulated.

E. LEWKOWITSCH.

**Kapok oil and the Halphen test.** H. P. TREVITHICK and W. H. DICKHART (Oil & Fat Ind., 1931, 8, 305, 317).—Halphen tests on mixtures with sesame, olive, palm-kernel oils, etc. were made; in admixture with kapok oil the colour is developed more rapidly and the depth (after 5 hr.) is equivalent to that produced by ten times the proportion of cottonseed oil. With >1% of kapok or >5% of cottonseed oils the colour is too deep for quant. estimation of the contaminant.

E. LEWKOWITSCH.

**Solubility of fats in various solvents. Solubility of (V) camellia oil, (VI) rape oil, in isopropyl alcohol of various concentrations.** K. HASHI (J. Soc. Chem. Ind. Japan, 1931, 34, 224—225 B, 226—227 B; cf. B., 1931, 550).—Solubilities are given for alcohol concentrations from 85% to 100%. Crit. temps. with corresponding oil concentrations rise rapidly with dilution of the alcohol, the values for 100% alcohol being (V) 7.8°, (VI) 29.3°.

C. IRWIN.

**Detecting the ageing of oils, particularly castor oil, by capillary analysis.** A. BOUTARIC and (MLLE.)

M. ROY (J. Pharm. Chim., 1931, [viii], 14, 113—123).—The diminution in the surface tension between 0.002N- $\text{Na}_2\text{CO}_3$  and a 1% solution of the oil in  $\text{C}_6\text{H}_6$  forms a more rapid and accurate means of detecting ageing than does the determination of the acid val.; moreover, a very small amount of sample is required. Results obtained with  $\text{Na}_2\text{CO}_3$  are more reliable than those with NaOH, but both are influenced by concentration and the time taken for the drops to form.

T. MCLACHLAN.

**The sulphur monochloride reaction of fatty oils. II. Nature of the reaction product. III. Thermal behaviour of their fatty acids. IV. Evolution of hydrogen chloride.** E. H. HARVEY and H. A. SCHUETTE (Trans. Wisconsin Acad. Sci., 1931, 26, 225—229, 231—232, 233—239; cf. B., 1931, 727).—II. The min. quantity of  $\text{S}_2\text{Cl}_2$  necessary to convert oils into solid products cannot be correlated with the I val. With olive, rape, cottonseed, tung, and cod-liver oils the whole of the S (with corn oil 90%) but only apart of the Cl of the  $\text{S}_2\text{Cl}_2$  added was found in the factice. Substitution as well as addition appears to have occurred.

III. In the reaction under standardised conditions in the presence of a diluent (xylene) the max. temp. rise is greater, and the time required to attain it is less, for the fatty acids than for the respective parent oils.

IV. The amounts of HCl evolved during reaction for 1 hr. (in  $\text{CHCl}_3$ ) cannot be correlated with the Ac or I vals. of the oils, nor are the figures additive for mixtures. Absence of sunlight retards the substitutive reaction.

E. LEWKOWITSCH.

**Sulphonated oils. V. Preparation and properties of the chemically pure alkali acid salts of ricinoleic sulphuric ester and chemistry of acid-salt formation in the sulphuric acid esters of the hydroxy-acids. VII. Properties of commercial sulphonated oils.** K. NISHIZAWA and K. WINOKUTI (Tech. Rep. Tôhoku, 1931, 10, 93—115, 116—130).—V. [With T. KIKUTI.] A full account in German of work published in Japanese (A., 1930, 321; cf. B., 1930, 568; 1931, 307). The formation and the prep. of the salts by treatment of ricinoleic sulphuric ester with alkali salts, including  $\text{Na}_2\text{SO}_4$ , and the properties of the salts (including crystallographic data) and of their aq. solutions (surface tension, viscosity, etc.) are detailed. The acid Na salt tends to form an anhydride (carboxylic) when its alcoholic solution is dried with anhyd.  $\text{Na}_2\text{SO}_4$ .

VII. [With S. IGARASHI.] A German translation with full details of the Japanese paper, Part IX (cf. B., 1931, 211).

E. LEWKOWITSCH.

Yolk in wool.—See V.

## PATENTS.

**Manufacture of shaving soaps.** A. C. J. PARENT (B.P. 354,056, 6.8.30. Fr., 28.8.29).—5—10% of non-toxic (e.g., Et) esters of phthalic acid and/or of *p*-aminobenzoic acid is added to the soap. E. LEWKOWITSCH.

**Semi-continuous dry-rendering and meal manufacturing process.** W. B. ALLBRIGHT (U.S.P. 1,791,439, 3.2.31. Appl., 30.3.25).—The high-grade fat is drained off after dry-rendering the material, until the moisture

content of the residue is 20–40%, i.e., greater than the crit. amount (about 16%) below which discoloration occurs, and the material is re-treated until sufficiently dry for the remaining second-grade fat to be easily expressed. E. LEWKOWITSCII.

**Heat treatment of linseed oil [for varnishes, linoxyn, etc.].** E. ASSER, and G. RUTH A.-G. (B.P. 353,949, 30.4.30. Ger., 1.5.29).—Linseed oil is pre-heated to 280° and blown with air while heating is continued up to 310°. E. LEWKOWITSCII.

**Stabilisation of greases etc. Lubricant for textiles.**—See II. Disinfection.—See XXIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Turpentine from pines.** T. H. McCOMBS (New Zealand J. Sci. Tech., 1931, 12, 333–337).—*Pinus radiata* and *P. pinaster* produced a first-grade turpentine. Yields and characteristics of the products are recorded. A. G. POLLARD.

**“Chalking” [of paint films]. V. Practical trials of chalking of oil paints. II.** R. KEMPF (Farben-Ztg., 1931, 36, 1972–1975, 2010–2013).—A further interim report is given after 20 months' exposure of the tests previously reported up to 10 months (B., 1931, 552). Full details of degree of chalking and extent of cracking over the whole period are quoted and graphed. No striking developments occurred in the second 10 months, the general superiority of  $\text{PbCrO}_4$  mixtures being maintained. White lead–lithopone mixtures, however, show increased chalking. The tests continue. S. S. WOOLF.

**Evaluation of “chalking” of paint [films].** H. WOLFF and G. ZEIDLER (Farben-Ztg., 1931, 36, 1930–1932).—The loss of wt. during 17 months and the degree of “chalking” (by Kempf's method) of films of white and grey paints based on basic sulphate and basic carbonate white lead, with and without  $\text{Pb}_3\text{O}_4$  priming, were determined. Grey paints, in general, lose less wt. than do white. Chalking is shown to be a surface effect only, not necessarily paralleled by the loss of wt. which is a function of the whole film, as is illustrated by the differences between the results with and without the  $\text{Pb}_3\text{O}_4$  undercoat. Results on a further series of mixed-pigment paints are also quoted. Kempf's “stamping” method for measuring chalking is approved, in general, and its application to the measurement of freedom from “tack” is indicated. The integration of the chalking curve as recommended by Kempf is, however, considered unsound, since the chalking process is not regular and false evaluation results from integration. S. S. WOOLF.

**Difficulties in the manufacture of oil and lacquer colours.** R. BÜRSTENBINDER (Farbe u. Lack, 1931, 122).—Faults in ready-mixed paints or paste colours may be traced to inaccurate additions of constituents or to unsuitable combinations of dry colours. The breaking down of paint films is frequently due to faulty drying of the media, and the rough matt surface given by some lacquers can be attributed to a reaction with the plasticiser. In the case of mixed pigments, cloudiness of the paint film and variations in tone are generally the result of a reaction between the colours themselves

and/or the medium, or to a chemical change in one of the constituents during drying. F. C. HARWOOD.

**Test for predicting the durability of varnishes. [The photochemical embrittling test.]** J. H. WILSON (Bur. Stand. J. Res., 1931, 7, 73–83).—The film of varnish is exposed to an intense source of light for a definite time, after which it is bent double at a prescribed temp. over a rod of fixed diameter. The more durable is the varnish, the longer it may be exposed to the light before it will crack on bending. A number of advantages over the methods commonly in use are given, particularly in simplicity and speed. The test is not sufficient for pigmented coatings which also fail by chalking, fading, loss of gloss, etc. E. S. HEDGES.

**Rosin in lacquer and oil colours.** R. SCHWARZ (Farbe u. Lack, 1931, 123–124).—For use in lacquers rosin is heated with tung oil to 275–300°, followed by an addition of  $\text{CaO}$  or  $\text{ZnO}$  etc. at about 250°. The  $\text{CaO}$  required for hardening is calc. from the acid val. of the rosin, and must be freshly slaked, finely powdered, and free from  $\text{CaCO}_3$ , or the rosin boilings become mouldy and lumpy. Addition of a small quantity of a solution of rosin in  $\text{C}_6\text{H}_6$  or turpentine to a tung oil lacquer remedies polymerisation. Resin esters formed by the addition of approx. 10% of glycerin to the rosin give a more durable lacquer film. F. C. HARWOOD.

**Cellulose nitrate for lacquers. Pine oil.**—See V.  **$\text{Pb}_2\text{O}$ .**—See VII. **Drying oils.**—See XII. **Colours for rubber.**—See XIV.

### PATENTS.

**Polishing [of cellulose lacquer surfaces].** BRIT. CELANESE, LTD. (B.P. 352,547, 12.4.30. U.S., 26.4.29).—A polish for surfaces composed of an org. derivative of cellulose and a synthetic resin consists of an abrasive, a diluent ( $\text{C}_6\text{H}_6$ ), and a liquid which has a softening effect on the surface. For resins made from  $\text{CH}_2\text{O}$  and substituted sulphonamides the latter are used as softening liquids; for phenol- $\text{CH}_2\text{O}$  resins Et phthalate, diphenylolpropane, or triacetin may be used. D. K. MOORE.

**Manufacture of plates, section pieces, etc. from phenol-formaldehyde artificial resins.** DR. F. RASCHIG GES. M.B.H., Assecs. of DR. F. RASCHIG CHEM. FABR. (B.P. 353,870, 22.4.30. Ger., 20.4.29).

**Mixing phenol compounds.**—See I. **Pigmented cellulose esters etc. Stencil sheets.**—See V. **Grinding wheels.**—See VIII. **Linseed oil.**—See XII. **Reaction products of rubber [as lacquer]. Vulcanisation moulds.**—See XIV.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Colours used in the rubber industry.** G. F. THOMPSON and E. V. BRATBY (J. Oil Col. Chem. Assoc., 1931, 14, 218–241).—A review of some of the desired characteristics in colours for rubber is followed by detailed reference to particular materials or types.

D. F. TWISS.

**Action of titanium dioxide in rubber mixings.** R. DITMAR (Caoutchouc et Gutta-Percha, 1931, 28, 15,640–15,643).—The methods used for preparing amorphous

TiO<sub>2</sub> are reviewed. Experiment shows that rubber containing TiO<sub>2</sub> and S<sub>2</sub>Cl<sub>2</sub> has a good white colour, but the pigment tends to soften the rubber and a slightly greater degree of vulcanisation is necessary. In heat-vulcanisation, TiO<sub>2</sub> resembles ZnO in activating org. accelerators, but has the additional advantage of being non-toxic. The softening influence of TiO<sub>2</sub> in larger proportions is effective in preventing scorching of rubber mixtures during the preliminary milling operations.

D. F. TWISS.

#### Increase of the resistance of vulcanised [rubber] products to wear by tin and cadmium selenides.

R. DITMAR and C. H. PREUSSE (Caoutchouc et Gutta-Percha, 1931, 28, 15,639—15,640).—In rubber vulcanised with S<sub>2</sub>Cl<sub>2</sub> or with S in the absence of an org. accelerator, CdSe and SnSe have neither reinforcing nor accelerating influence. In the presence of most org. accelerators, however, vulcanised products containing either of these selenides show marked reinforcement and increased resistance to abrasion.

D. F. TWISS.

#### PATENTS

#### Manufacture of natural rubber and artificial rubber-like masses.

A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 352,549, 12.4.30. Cf. B.P. 333,941; B., 1930, 1017).—Aliphatic aldehydes are condensed with  $\alpha$ -naphthylamines or their substitution derivatives in the presence of H<sub>2</sub>O-sol. alcohols and acids, and the primary resin-like products are treated with H<sub>2</sub>O, preferably with heating. The products, which are yellowish inodorous powders, sparingly sol. in EtOH, are used as antioxidants for rubber and do not interfere in any way with the normal course of vulcanisation.

D. F. TWISS.

#### Manufacture of rubber or similar material.

DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., and E. W. MADGE (B.P. 351,937, 31.3.30).—Porous or microporous articles of vulcanised rubber are obtained by adding to compounded latex one or more reagents which are substantially non-coagulants, but which effect coagulation on the application of heat. NH<sub>4</sub> salts of strong acids act in this way in latex containing ZnO. Coagulation or gelling is then induced by heat in one or more operations such as moulding, spreading, or dipping. Vulcanisation is effected under conditions preventing the escape of the dispersion medium from the pores of the product.

D. F. TWISS.

**Manufacture of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 352,781, 21.8.30. U.S., 22.8.29).—Rubber articles are protected against the effects of heat as well as of O<sub>2</sub> by incorporating a compound of the formula NH<sub>2</sub>·R'·NH·R''·Y, where R' and R'' represent aromatic residues and Y represents H, OH, or NH<sub>2</sub>. The aminodiphenylamines and *N*-aminophenyl-naphthylamines are typical.

D. F. TWISS.

**Manufacture of rubber and the like. [Anti-ageing compounds.]** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,391, 29.8.30. U.S., 30.8.29).—Tetra-aminobenzhydrols, carrying, if desired, nuclear alkyl or alkoxy or *N*-alkyl or -aralkyl substituents, are added to a rubber mix or applied to vulcanised rubber as antioxidants.

C. HOLLINS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 351,938, 31.3.30. Addn. to B.P. 327,451; B., 1930, 626).—Using the method of procedure described previously an aq. dispersion of rubber containing disintegrated org. material such as ground rubbered cotton waste, cork powder, or leather dust, is converted into a crumb-like coagulum by the addition of compounding ingredients of the type already indicated. The crumb-like compositions may be shaped as such or dried and shaped, *e.g.*, by moulding, with or without previous milling.

D. F. TWISS.

#### Manufacture of coloured rubber masses.

A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 352,506, 10.4.30).—An alkaline-earth salt of a sulphonated azo dye prepared from a nitroaniline and an acetoacetic arylide, *e.g.*, the Ca salt of the azo compound derived from 5-nitro-2-aminobenzoic acid and acetoacetic-2-anisidide-sulphonic acid or from *o*-nitroaniline-4-sulphonic acid and acetoacetic-2-anisidide, is used exclusively or in part as a colouring agent for manufactured rubber.

D. F. TWISS.

#### Manufacture of reaction products of rubber.

DUNLOP RUBBER CO., LTD., S. A. BRAZIER, E. H. HURLESTON, and E. V. BRATRY (B.P. 352,080, 5.4.30).—The products obtained by treating rubber with an org. nitroso-compound, *e.g.*, *p*-nitrosodimethylaniline, in substantially greater proportions than those used for satisfactory acceleration of vulcanisation have strong adhesive properties and can be applied as a lacquer or bonding agent for rubber to rigid impermeable surfaces such as of metal.

D. F. TWISS.

#### Process and compounds for treating rubber.

ROESSLER & HASSLACHER CHEM. CO. (B.P. 352,746, 18.7.30. U.S., 2.8.29).—The hexamethylenetetramine benzyl ester of a mercaptothiazole, obtained by the interaction of the additive product of a benzyl halide and hexamethylenetetramine with a metal salt of a mercaptothiazole, is applied as an accelerator of vulcanisation. The product from mercaptobenzthiazole has m.p. 140°.

D. F. TWISS.

#### Vulcanisation accelerators [for rubber] and application thereof.

IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 352,165, 17.5.30).—Vulcanisation is accelerated

by a compound of the formula R' <  $\begin{smallmatrix} N \\ S \end{smallmatrix} \end{smallmatrix} \text{S} \cdot \text{S} \cdot \text{C} \begin{smallmatrix} N \\ S \end{smallmatrix} \text{R}''$ , (R' and R'' being aromatic residues) with or without the additional presence of a basic org. accelerator such as diphenylguanidine. The products are obtained by the action of a compound R <  $\begin{smallmatrix} N \\ S \end{smallmatrix} \end{smallmatrix} \text{SCl}$  (R being an arylene radical) on a 2-mercaptoarylenethiazole. They are free from tendency to prevulcanisation.

D. F. TWISS.

**Vulcanisation of rubber and rubber-like substances.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 352,133, 26.4.30).—The reaction products of 2-mercaptoarylenethiazoles and a halogenated benzoquinone, *e.g.*, chloranil, containing a reactive halogen atom, accelerate vulcanisation without inducing scorching during

the preliminary milling operations. They are preferably used in conjunction with a basic org. accelerator, e.g., diphenylguanidine. D. F. TWISS.

**Vulcanisation moulds.** BAKELITE CORP., ASSECS. OF G. W. CROSBY (B.P. 352,519, 6.3.30. U.S., 7.3.29).—A mixture of a thermo-setting phenolic resinoid (approx. 40%) with finely-divided mica (5–60%), the balance, if any, consisting of org. or inorg. fillers such as wood flour, asbestos, or Al or other metallic powder, is used to form the contact surface in a mould for the vulcanisation of rubber. D. F. TWISS.

**Determining plasticity.**—See I. Rubber-impregnated fibres.—See V. Substances containing S.—See VII. Coated Cu wires.—See XI.

## XV.—LEATHER; GLUE.

**Behaviour of leather towards free sulphuric acid.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 84–88; Chem. Zentr., 1931, i, 2427).—Immerheiser's method is unacceptable. In Paessler's method with ox or calf leather the value of the acid found diminishes with diminution of the concentration of the  $\text{H}_2\text{SO}_4$ . Calf leather takes up about 50% more  $\text{H}_2\text{SO}_4$  than does ox leather. The extraction loss of the leather and the N content of the Procter extract fall with decreasing  $\text{H}_2\text{SO}_4$  concentration.

A. A. ELDRIDGE.

**Testing of leather preservatives and impregnating materials.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 89–92; Chem. Zentr., 1931, i, 2427–2428).—The composition of commercial materials is recorded. To test whether the impregnating material causes any change in the leather the former is removed and the total N,  $\text{H}_2\text{O}$ -sol. N, and extraction loss of the latter are compared with values obtained for untreated leather. A. A. ELDRIDGE.

PATENTS.

**Apparatus for manufacturing pellet glue.** T. K. LOWRY (B.P. 353,881, 28.4.30).

**Insecticide.**—See XXIII.

## XVI.—AGRICULTURE.

**Secular and seasonal changes in soils.** J. S. BURD and J. C. MARTIN (Hilgardia, 1931, 5, 455–509).—The soils examined were maintained under controlled conditions. If water is withheld between seasons losses of N from low-fertility soils under continuous cropping are very small and some gain in N may occur. Under similar conditions substantial increases in N occur in fallowed uncultivated soils. The C:N ratio of continuously cropped soils remains the same as when prolonged fallow periods intervene between crops. The total concentration of the soil solution is reduced by cropping and usually, but not invariably, increased by fallowing. The  $[\text{SO}_4^{--}]$  tends to increase in both fallow and cropped soils.  $\text{NO}_3^-$  increases in fallow and falls in cropped soils. A significant decline in the  $[\text{K}^+]$  occurs in many cropped soils. After depletion by cropping the subsequent increase in the concentration of the soil

solution may be fairly rapid. Whatever their previous history, cropped soils lose more  $\text{NO}_3^-$  than is absorbed by the crops, the loss being very heavy in high- $\text{NO}_3^-$  soils. The cropping of soils after prolonged fallowing results in a rapid decline in total concentration and a corresponding reduction in crop yield. A. G. POLLARD.

**Characteristics of an eroded soil.** G. W. MUSGRAVE and H. DUNLAVY (J. Amer. Soc. Agron., 1931, 23, 245–252).—Eroded soil exhibited a downward trend of moisture and relative  $\text{H}_2\text{O}$ -holding capacity, loss of org. matter, decrease in colloids and moisture equiv., and a lower percentage of clay. CHEMICAL ABSTRACTS.

**Podsollic process in soils.** T. J. DUNNEWALD (J. Amer. Soc. Agron., 1931, 23, 259–265).—Laboratory experiments indicate that org. matter is not concerned in the podsollic accumulation of  $\text{SiO}_2$ , but is concerned in the translocation of bases and sesquioxides. Extraction of the bases and formation of acid conditions appear to be necessary preliminaries to deposition of  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  in subsoil. Timbered and grass soils are compared. CHEMICAL ABSTRACTS.

**Humus formation and destruction by certain higher filamentous fungi in forest soils.** R. FALCK (Forst-Archiv, 1930, [16]; Bied. Zentr., 1931, 60A, 252–253).—The action of fungi on the lignin and cellulosic matter of beech leaves may result in a "corrosion" process ultimately yielding humus, or a destructive process characterised by the production of sugars and org. acids, notably  $(\text{CO}_2\text{H})_2$ . The formation and maintenance of forest humus is examined in the light of these facts. A. G. POLLARD.

**Variability of the buffer value of soils.** S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1931, 21A, 257–270).—The buffer power of a soil is dependent on the proportion of sol. basic or neutral salts present, and values recorded by different methods for its determination vary accordingly. Soils highly saturated with bases have lower buffer capacities than those deficient in bases. The reduction of the buffer powers of soils by liming is due to the resultant increase in particle size. The utilisation of these results in the quant. examination of the effects of CaO on the physical properties of soils is discussed.

A. G. POLLARD.

**Base-exchange property of organic matter in soils.** W. T. McGEORGE (Arizona Agric. Exp. Sta. Tech. Bull., 1930, No. 30, 181–213).—The exchange capacity of highly org. soils is a linear function of their C content, but bears no relation to the N content or C:N ratio. Base exchange in highly org. soils takes place in chemically equiv. proportions. Loss of exchange capacity following treatment with  $\text{H}_2\text{O}_2$  is a linear function of the amount of org. matter destroyed. Digestion with  $\text{H}_2\text{O}_2$  does not affect the exchange capacity of natural bentonites, but reduces that of synthetic zeolites. Lignin, lignohemicellulose, and lignocellulose function as exchange compounds and each has a definite equiv. exchange capacity. Tervalent ions (e.g.,  $\text{Al}^{+++}$ ) cannot enter the org. exchange complex by replacement of mono- and bi-valent ions. Lignin-like substances suffer a partial loss of exchange capacity

by leaching with  $\text{AlCl}_3$  solution. This is regained by treatment with a solution of a hydroxide or of a salt yielding  $\text{OH}$  ions by hydrolysis. A. G. POLLARD.

**Organic compounds associated with base-exchange reactions in soils.** W. T. McGEORGE (Arizona Agric. Exp. Sta. Tech. Bull., 1931, No. 31, 215—251).—The base-exchange capacity of soil lignins and "ligno-humates" varies with soil type. The aq.-alkali-sol. ligno-humate has a capacity of 382 milliequiv. and the alcoholic-alkali-sol. portion 116 milliequiv. per 100 g. Leaching with  $\text{HCl}$  increases the capacity of lignin and ligno-humates, probably by hydrolysis. "Lignic acid" and "ligno-humic acid" (H-saturated lignin and ligno-humate) are, respectively, di- and tetra-basic. The amount of base absorbed from an acetate solution by lignin is equal to that necessary to neutralise the lignic acid. The degree of ionisation of the Na and K compounds of the org. complexes is relatively high and that of Ca, Ba, and H compounds low. Xylan has a slight and synthetic humus made from xylan a considerable exchange capacity. Xylan, lignin, and ligno-humates absorb colour from basic dyes and the colour is replaceable by the base of a neutral salt. Dry-ground lucerne has a definite exchange capacity which is not easily destroyed by  $\text{H}_2\text{O}_2$  and increases considerably during the spontaneous decomp. of the material. The lignin content of the org. matter of highly org. soils is a linear function of the base-exchange capacity. No similar relationship exists with the cellulose or hemicellulose. A. G. POLLARD.

**Dispersion of soils by a supersonic method.** L. B. OLMSTEAD (J. Agric. Res., 1931, 42, 841—852).—The mechanical dispersion of soil in  $\text{H}_2\text{O}$  is produced by means of supersonic waves. The degree of dispersion is of the same order as that obtained in the rubbing method, but is more quickly reached. By neither method is the extraction of colloidal matter complete, small and decreasing amounts being obtained by successive treatments. A. G. POLLARD.

**Determination of the total surface area of soils, clays, and similar substances. III. Effect of the salt content on the water absorption of soils and clays.** H. KURON (Z. Pflanz. Düng., 1931, 21A, 271—300; cf. B., 1931, 37).—Both the capillary-bound and the adsorbed  $\text{H}_2\text{O}$  of clays etc. can act as solvent for salts. In the system Na-clay- $\text{NaCl-H}_2\text{O}$ , all the bound  $\text{H}_2\text{O}$  has solvent powers only when the  $\text{NaCl}$  concentration is high. In the system Na-clay- $\text{Na}_2\text{CO}_3\text{-H}_2\text{O}$  there is a positive adsorption of  $\text{NaOH}$  by the clay. The nature of the adsorption isotherm of a Na-clay is not affected by the addition of Na salts. In the systems Ca-clay- $\text{NaCl-H}_2\text{O}$  and Ca-clay- $\text{Na}_2\text{CO}_3\text{-H}_2\text{O}$  the initial exchange reaction is followed by increased adsorption. Changes in the  $\text{H}_2\text{O}$ -absorbing power of clays saturated with Ca, Mg, or  $\text{H}$  brought about by the addition of increasing amounts of  $\text{Na}_2\text{CO}_3$  may be utilised for the indirect determination of the base-adsorptive capacity. The application of such a method to soils rich in  $\text{CaCO}_3$  is noted. A. G. POLLARD.

**Determination of total carbon [in soils].** E. WINTERS and D. C. WILMER (J. Amer. Soc. Agron., 1931,

23, 280—285).—The loaded boat is moved magnetically into the centre of the furnace after the flow of  $\text{O}_2$  has been started. The error is  $\pm 0.55\%$ .

CHEMICAL ABSTRACTS.

**Plant-soluble phosphoric acid in soils in relation to geological soil structure.** L. NEUBERGER (Kunst-dünger u. Leim, 1931, 28, 77—78; Chem. Zentr., 1931, i, 2383).—Degrees of P requirement are computed.

A. A. ELDRIDGE.

**Determination of the potash requirement of soil by means of *Aspergillus niger*.** H. NIKLAS, H. POSCHENREDER, and G. VILSMEIER (Arch. Pflanzenbau, 1930, A, 5, 152—165; Bied. Zentr., 1931, 60A, 272).—Results of the *Aspergillus* method were in good general agreement with those of the Neubauer test, but there is some divergence in soils of low K content. Repeated use of *Aspergillus* cultures on K-deficient soils leads to a decreased development of mycelium and *vice versa*. Relationships between the wt. of mycelium produced and the K assimilated are not always strictly regular. It is suggested that after the initial growth the more difficultly-sol. K may be assimilated. A. G. POLLARD.

**Effect of superphosphate on the reaction, degree of saturation with bases, and buffer power of acid mineral soils.** L. SCHMITT (Superphosphat, 1930, 6, 219—227; Bied. Zentr., 1931, 60A, 257—258).—Addition of superphosphate to soil produces a temporary increase in acidity which, however, quickly returns to the initial value even in definitely acid soils. The duration of the acid condition is insufficient to incur plant injury. Continuous applications of superphosphate during 29 years had no unfavourable effect on soil reaction, degree of saturation with bases, or the buffer capacity. A. G. POLLARD.

**Solubilisation of plant nutrients by drying and other treatments of soil.** F. ZUNKER (Kulturtechniker, 1930, 6, 591—603; Bied. Zentr., 1931, 60A, 255).—The increased solubility of mineral soil constituents during drying is ascribed to the reduced adsorptive power per unit surface area of the particles. Relationships between the adsorptive power of soils, changes in concentration of the adsorbed and capillary fluids, and the effects of humic acid are discussed. A. G. POLLARD.

**Explosive properties of fertiliser salts containing (1) calcium nitrate, (2) ammonium nitrate and various amounts of oil.** HAID, GOETZE, SELLE, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 108—113, 113—115; Chem.-Zentr., 1931, i, 2381).—The stability of  $\text{Ca}(\text{NO}_3)_2$  depends on the  $\text{H}_2\text{O}$  content; the fertiliser containing 13.2%  $\text{H}_2\text{O}$  is incompletely decomposed in the Pb block test even when the oil content reaches 4%, and it is not detonated by shattering explosives or direct heating. Technical urea- $\text{Ca}(\text{NO}_3)_2$  behaves similarly. Nitrophoska and Leuna saltpetre, when oiled, are not explosive, whilst the explosibility of  $\text{Ca NH}_4$  saltpetre is increased by the presence of oil. A. A. ELDRIDGE.

**Fertilisers and soils in relation to Concord grapes in south-western Michigan.** N. L. PARTRIDGE and J. O. VEATCH (Michigan Agric. Exp. Sta. Tech. Bull., 1931, No. 114, 42 pp.).—Characteristics of the vine soils



are recorded. Application of N fertilisers affected vines 15 ft. (but not at 25 ft.) from the margin of the fertilised area. Variations in the thickness of the humus layer of the surface soil had more influence on the growth and productivity of vines than any other soil factor. Vines weaker at the beginning of a fertiliser trial remained relatively weaker throughout whether fertilised or not. Application of  $(\text{NH}_4)_2\text{SO}_4$  or  $\text{NaNO}_3$  increased the wt. of prunings more than that of matured fruit. The effects of the two fertilisers were similar. Increased growth and productivity followed applications of either superphosphate or KCl with N. Use of both P and K with N gave little additional yield. Increased production following use of fertilisers was primarily the result of actual growth increase, vines of similar vigour yielding similar crops irrespective of the fertiliser used. The response to fertilisers was more marked in weak than in vigorous vines and differences in yield between individual vines is thus reduced considerably. A. G. POLLARD.

**Black cotton soil under different fertiliser treatments.** R. D. REGE (Proc. XV Indian Sci. Cong., 1928, 50—51).—Comparative experiments are described.

## CHEMICAL ABSTRACTS.

**Nitrification of stall manure in arable soils.** VI. C. BARTHEL and N. BENGTSON (Centralanst. Versuchswesen Geb. Ackerb. bact. Abt. Medd., 1930, No. 381, 18 pp.; Bied. Zentr., 1931, 60A, 254—255).—The rates of nitrification of the N-constituents of bacterial tissue by pure cultures of various organisms are recorded. Simpler amino-acids are nitrified more quickly and more completely than the more complex. Differences between this type of nitrification and that of cattle manure (50% of the N of which is calc. to be bacterial matter) are ascribed to the presence of colloidal matter which retards bacterial activity, and to the less favourable C : N ratio of the org. matter. Moreover, the varied decomposability of the C compounds of manure introduces a time factor into the action of the bacteria.

A. G. POLLARD.

**Effect of application of manurial salts on the suction force of soil.** H. E. STEINER (Fortsch. Landw., 1930, 5, 550—552; Bied. Zentr., 1931, 60A, 258).—Manuring with 40% K salts and with  $\text{NaNO}_3$  increased the suction force of soil, the effect increasing with falling soil-moisture content.

A. G. POLLARD.

**Treatment of lime-induced chlorosis with iron salts.** J. P. BENNETT (California Agric. Exp. Sta. Circ., 1931, No. 321, 12 pp.).—Methods of spraying, soil treatment, and injection are described.

A. G. POLLARD.

**Effects of various plant foods on growth activities and development of oats.** C. K. McCLELLAND (J. Amer. Soc. Agron., 1931, 23, 304—311).—P increased the number of tillers, heads, and spikelets and the yield and the seed size of oats; it caused early tillering and growth. N behaved similarly; K rather restricted the tillers, heads, and yield.

## CHEMICAL ABSTRACTS.

**Diseases of grain and their control.** W. W. MACKIE (California Agric. Exp. Sta. Bull., 1931, No. 511, 87 pp.).—Methods and formulæ for chemical treatment of seed are recorded.

A. G. POLLARD.

**China aster seed treatment and storage.** W. O. GLOYER (New York State Agric. Exp. Sta., Tech. Bull., 1931, No. 177, 41 pp.).—For the control of fungal diseases, aster seed is soaked for 30 min in  $\text{H}_2\text{O}$  at  $38^\circ$ , drained, and steeped in 1 : 1000  $\text{HgCl}_2$  solution for a further 30 min. at  $38^\circ$ , washed with  $\text{H}_2\text{O}$ , and spread out to dry for 24 hr. Org. Hg compounds were less efficient.

A. G. POLLARD.

**Phosphate slags. Soil corrosion.**—See X.

## PATENTS.

**Fertiliser manufacture.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 352,739, 14.7.30. U.S. 15.7.29).—Phosphate rock is partly decomposed with  $\text{H}_2\text{SO}_4$ , further decomp. being then effected by addition of  $\text{HNO}_3$ . Loss of the latter through evolution of nitrous fumes is thus avoided.

W. J. WRIGHT.

**Conservation of unstable chemical fertilisers by coating.** A. MENTZEL (B.P. 352,258, 11.7.30. Ger., 19.7.29).— $\text{NH}_4\text{HCO}_3$  crystals are coated with  $\text{NH}_4\text{Cl}$  by subliming the latter on to the crystals, which are kept cool and stirred during the condensation. Urea may be used instead of  $\text{NH}_4\text{Cl}$ , but sublimation must then be effected in vac.

A. R. POWELL.

**Manufacture of manures.** J. I. BRONN, and CONCORDIA BERGBAU A.-G. (B.P. 352,476, 4.4.30).—Waste  $\text{CaCl}_2$  liquors are caused to react with  $\text{NH}_3$  and  $\text{CO}_2$ , or  $(\text{NH}_4)_2\text{CO}_3$ , or with gas mixtures containing  $\text{NH}_3$  and/or  $\text{CO}_2$ , preferably under pressure, the solution being crystallised or evaporated to give a product consisting of  $\text{CaCO}_3$  and  $\text{NH}_4\text{Cl}$ .

W. J. WRIGHT.

**Fungicide and bactericide.** J. W. ROBERTS (U.S.P. 1,791,430, 3.2.31. Appl., 12.10.28).—The product contains  $\text{ZnSO}_4$  and  $\text{CaO}$ , together with, e.g.,  $\text{CaO}$ -casein and alum. The above in the form of a spray is effective against *Bacterium pruni*.

L. A. COLES.

**Treatment of phosphate material. Compound fertilisers.**  $\text{CaCN}_2$ . Substances containing S.—See VII. Insecticide.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Determination of the sugar content of cane.** P. NEUVILLE (Internat. Sugar J., 1931, 33, 326—328).—That the use of the Java ratio may give an inexact value in calculating the sugar content of the cane was shown by crushing cane in two laboratory mills throughout an entire season, when the polarisation of the juice thus obtained (15.33) differed distinctly from that of the factory juice (14.94). It is concluded that the calculation of the Java ratio based on the juice obtained from small mills cannot be applied to the primary juice of the factory. In two factories in Egypt the sucrose of the sugar bagged, plus that left in the molasses, plus the polarisation of the bagasse, gave a sum which did not correspond to the sugar content of the cane as calc., using values of 85.2 and 85.8. An appreciably higher ratio should have been used, and it would have been raised yet higher had the sucrose content (by double polarisation) been applied in its calculation, instead of the direct polarisation.

J. P. OGILVIE.

**[Sugar juice] carbonatation studies. I. Comparison of the Pšenička and Hruđa processes.** L. DOSTÁL (Z. Zuckerind. Czechoslov., 1931, 55, 555—563).—Using a small-scale plant, comparative experiments were made with the Pšenička and Hruđa processes, using in different series 0.75, 1.25, and 2.25% CaO. No difference was to be observed in the results given by the two methods so long as the same amount of CaO was used. In the series using 0.75% CaO there was distinctly more colour and a higher content in Ca salts than with the two larger quantities of CaO. On the whole, the 2.25% CaO series showed the best results.

J. P. OGILVIE.

**Course of the first carbonatation [of beet juices]. V. Neutralisation of sugar solutions containing lime, using oxalic acid. VI. Inactivation of calcium oxalate.** J. DĚDEK and D. IVANČENKO (Z. Zuckerind. Czechoslov., 1931, 55, 527—532, 593—596; cf. B., 1931, 560).—V. Laboratory experiments imitating factory conditions of carbonatation, but using oxalic acid for neutralisation, showed the course of the operation to proceed as when CO<sub>2</sub> is used. Entrainment of CaO by the ppt., however, took place only at high temps.

VI. On formation of a ppt. of CaC<sub>2</sub>O<sub>4</sub> in a solution of sugar at temps. below 70°, CaO is carried down, but this property is lost on heating, and in a few min. at temps. above 70°. Addition of alkalis to the suspension of the ppt. hinders this "inactivation."

J. P. OGILVIE.

**Glycerophosphoric acid in the liming and saturation [of beet juice].** B. DLOUHÝ (Z. Zuckerind. Czechoslov., 1931, 55, 431—435).—Factory scum cakes from the first carbonatation contained 1—1.5% P<sub>2</sub>O<sub>5</sub>, including 0.1—0.3% organically combined, probably as glycerophosphate and distearyl-glycerophosphate, not directly precipitable by magnesia mixture or molybdate. Cakes from the second and third carbonatations contained 0.56 and 0.19% P<sub>2</sub>O<sub>5</sub>, including 0.46 and 0.11% organically combined. Small quantities of Ca glycerophosphate, added to 15% sucrose solutions, were removed to the extent of over 90% by the usual liming and carbonatation operations, but mainly by adsorption, undecomposed. Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> readily adsorbs Ca glycerophosphate.

J. H. LANE.

**Value of the addition of phosphate in difficult [sugar] juice clarification.** H. D. LANIER (Internat. Sugar J., 1931, 33, 418).—In a factory in Cuba the average phosphate content of the raw juice was 0.022 g. P<sub>2</sub>O<sub>5</sub> per 100 c.c. of juice, but on adding  $\frac{1}{4}$ — $\frac{1}{2}$  lb. of superphosphate (of 50% solubility) per ton of cane ground, and liming to  $p_H$  7.5, a considerable improvement in the brilliancy of the clarified juice was observed. The best results were obtained when the P<sub>2</sub>O<sub>5</sub> content of the juice was raised to 0.033%, which addition was estimated to have increased the capacity of the factory by 10%.

J. P. OGILVIE.

**Influence of composition of sugar juices on their darkening during evaporation.** O. REISS (Z. Zuckerind. Czechoslov., 1931, 55, 515—526).—Employing Staněk's laboratory evaporator (B., 1926, 961) and Šandera's photocolormeter graduated in Stammer degrees (B., 1930, 1126), the author conc. 400-c.c.

portions of sugar solutions and beet thin-juices from 15° to 60° Brix in 1 hr. and determined the increase in colour at the original dilution and  $p_H$  val. With solutions of refined sugar no measurable darkening occurred at neutral or acid reactions, even after addition of 0.05% of invert sugar and 0.05% of amino-acid (aspartic acid). At initial reactions of  $p_H$  8.72—10.33, colours of 0.2—0.46° Stammer (at 15° Brix) were produced, with a fall of 0.3 in  $p_H$  val.; these effects were intensified by initial addition of 0.05% of invert sugar and much more by addition of 0.05% of aspartic acid as well. Beet thin-juices suffered a slight fall in  $p_H$  val. and an increase of 30—80% in their colour. This increase was much lessened by initial addition of 0.02% of Na<sub>2</sub>SO<sub>3</sub>, but much increased by addition of 0.001% of FeSO<sub>4</sub>. Raw beet sugars in 15% solution darkened less than thin-juices. Repeated evaporations after redilution to 15° Brix had in most cases about the same darkening effect as the first, or rather less.

J. H. LANE.

**Number of micro-organisms in diffusion [sugar] juice.** J. VONDRÁK (Z. Zuckerind. Czechoslov., 1931, 55, 601—605).—Counts of the micro-organisms present in the diffusion juices from five beet factories gave figures from 800,000 to 12,000,000 per c.c., the lowest being observed at the beginning of the campaign. *Diplococci* colonies were much in evidence, and in some cases a considerable number of *Torula* colonies developed.

J. P. OGILVIE.

**Factors affecting the ash content of [sugar] factory juices.** L. WICHERS (Arch. Suikerind. Nederl.-Indië, 1931, 39, I, 472—477).—The nature of the soil, and its content in mineral matter, particularly CaO, Cl, SO<sub>3</sub>, affect considerably the ash content of the juice, as does also the variety of cane grown. It is a general experience in Java factories that the introduction of the POJ 2878 variety has been accompanied by an increase in the ash content of the factory molasses, as compared, e.g., with cane variety EK 28.

J. P. OGILVIE.

**Chlorine as a decolorant in sugar analysis.** L. R. BLISS (Rev. Ind. Agr. Tucumán, 1930, 20, 180—186).—After treating sugar solutions with basic Pb acetate for their polarisation, dry Cl<sub>2</sub> is passed in, Al(OH)<sub>3</sub> cream added if necessary to remove traces of colloidal Pb, and the liquid finally filtered.

J. P. OGILVIE.

**Conductometric determination of the affining values of raw [beet] sugars.** K. ŠANDERA and C. A. RUŽICKA (Z. Zuckerind. Czechoslov., 1931, 55, 423—431).—A standardised affination test for raw sugars would often furnish more useful information than the conventional rendement. Comparative study of Šandera's test (B., 1928, 344) and that of Spengler and Brendel (B., 1927, 730; 1928, 30), as applied to 120 Czechoslovakian raw sugars, shows the necessity for an agreed definition of affining value, taking account of the various factors involved.

J. H. LANE.

**Comparative analysis of [beet] sugars in the usual way and by the method of single weighing with conductometric ash determination, and the times required.** A. DOLINEK, J. LINHART, and M. ŠMIDINGEROVÁ (Z. Zuckerind. Czechoslov., 1931, 55,

439—440).—With 265 raw beet sugars, not including after-products, determinations of polarisation and conductometric ash from a single weighing led to rendements differing in no case by more than 0.55% and in 208 cases by not more than 0.25% from the rendements determined in the usual way involving two weighings and an incineration. The rendement could be determined in  $\frac{1}{2}$  hr. by the former method, compared with  $1\frac{1}{2}$  hr. by the latter.

J. H. LANE.

**Analysis with Wood's light and the products of sugar manufacture.** G. MEZZADROLI and E. VARETON (Atti III Cong. Naz. Chim., 1929, 580—581; Chem. Zentr., 1931, i, 2277).—The use of the fluorescence method in analysis and control is described.

A. A. ELDRIDGE.

**Behaviour of cane molasses during storage.** G. L. C. LA BASTIDE (Arch. Suikerind. Nederl.-Indië, 1930, 38, II, 701—703).—Periodic analysis of a good number of samples of molasses from defecation, sulphitation, and carbonatation factories showed that the polarisation gradually falls, though sometimes the sucrose (by double polarisation) remains const. The content of reducing sugars falls, fermentation apparently taking place. Glucose increases, generally in the first month, contents of from 2.22 to 5.67% being observed.

J. P. OGILVIE.

**Influence of reducing sugars on the caramelisation test.** J. PUCHERNA (Z. Zuckerind. Czechoslov., 1931, 55, 663—669).—Reducing sugars are decomposed in the caramelisation test parallel with sucrose, but in the presence of the salts and amides of amino-acids, and also of alkali salts, coloured decomp. products are more readily formed from reducing sugars than from sucrose under identical conditions, and they have a more intense colour.

J. P. OGILVIE.

**Devitrification of sugar products [e.g., fruit drops].** F. HALLA and E. MEHL (Z. Zuckerind. Czechoslov., 1931, 55, 551—553).—That the change undergone by fruit drops and like vitreous confectionery articles on being exposed to moist air is due to surface recrystallisation is shown by six X-ray photographs, the amorphous ring being exhibited clearly in the case of freshly made drops, and the interference rings in material which had undergone the recrystallisation change.

J. P. OGILVIE.

**Determination of the acidity of potato flour.** A. P. SCHULZ (Z. Spiritusind., 1931, 54, 193).—Discordant values of the acidity of potato starch are due to the amount of NaOH required to neutralise a fixed quantity of starch decreasing to a const. as the vol. of  $H_2O$ , with which the starch paste is made, is increased to 100 c.c. The determination of the acidity is standardised by mixing 20 g. of starch with 100 c.c. of "neutralised"  $H_2O$ , adding 10 drops of 1% phenolphthalein, and titrating with 0.1N-NaOH until the red colour is permanent for 1 min. after stirring for 3 sec. New standards of quality of potato flours are chosen as 0.6, 0.8, and 1.2 c.c. of N-NaOH per 100 g. of starch for "fine," "superior," and "first" quality, respectively.

C. RANKEN.

**Hydrolysis of starch by oxalic acid in the preparation of [textile] sizes.** W. SIEBER and S. GÄRT-

NER (Textilber., 1931, 12, 105—107).—Results are given showing the amount of dextrose formed by heating potato starch with various concentrations of oxalic acid at 100° for 3 hr. Under such conditions about 50% and 15% of the starch is hydrolysed by 5% and 0.5% solutions of the acid, respectively. The view that  $(CO_2H)_2$  is of special value in preparing sol. starch for sizing purposes because dextrose is not formed simultaneously is shown to be erroneous.

A. J. HALL.

**Active carbons.**—See II. Naringin in marmalade.—See XIX.

## PATENTS.

**Treatment of saccharine fluids [with invertase].** L. WICKENDEN, Assr. to J. J. NAUGLE (U.S.P. 1,783,551, 2.12.30. Appl., 3.5.27).—Sugar syrups to be inverted, e.g., affination syrups, are treated, preferably at  $p_H$  4—5 and temp. 55—65°, with syrupy invertase products prepared according to U.S.P. 1,689,607 (B., 1929, 70), and are afterwards clarified if necessary.

J. H. LANE.

**Treatment of saccharine fluids.** (A) L. WICKENDEN and (B) J. J. NAUGLE, (A) Assr. to (B) (U.S.P. 1,787,502, 6.1.31. Appl., 28.1.26).—A fluid containing two or more types of impurities is treated by the addition of subdivided adsorbent material of varied degrees of fineness derived from leached carbonised lignin residues, in order to adsorb different types of such impurities, the mixture being stirred and heated to about 90°. Each fraction is separately treated for the removal of the adsorbent material.

J. P. OGILVIE.

**Treatment of sugar solutions.** H. DE F. OLIVARIUS, Assr. to CALIFORNIA PACKING CORP. (U.S.P. 1,788,628, 13.1.31. Appl., 27.12.27).—In recovering sucrose from its impure solutions containing invert sugar, it is concentrated to 35—65° Brix, and the invert sugar fermented without material inversion of sucrose. Yeasts of the *Saccharomyces apiculatus* group are not used.

J. P. OGILVIE.

**Treatment of sugar juices or syrups.** S. COLE. From F. CHAMIER (B.P. 350,118, 4.4.30).—Raw diffusion juices or syrups, e.g., from suitably or partly dried cane or beet slices, are acidified to  $p_H$  4.3—4.5 with  $H_2SO_4$  at 20—50° to ppt. coagulable matters, then clarified centrifugally, adjusted to  $p_H$  7.4—7.6 with CaO and  $Na_2CO_3$ , and again centrifuged. Further known treatments, e.g., with charcoal, may precede boiling.

J. H. LANE.

**Sugar process.** R. E. POSPISIL, Assr. to E. KUREK (U.S.P. 1,772,911, 12.8.30. Appl., 19.5.28).—The process of U.S.P. 1,352,084 (B., 1920, 732) is adapted to enable raw cane sugar to be worked up with beet juice for the production of white granulated sugar without the use of bone char and during an extended working season.

J. H. LANE.

**Monosaccharide material [from starch].** T. B. WAGNER (U.S.P. 1,784,402, 9.12.30. Appl., 30.7.23).—Green starch or other amylaceous material is hydrolysed with acid until about 90% of dextrose has been formed and until the dextrans present have lost their gummy properties. After a crop of cryst. dextrose has been obtained by known procedure, the mother-liquor, containing about 20.5% of  $H_2O$ , 57.5% of dextrose,

19.5% of non-fermentable carbohydrates, and 2.5% of ash, constitutes a specially good material for the manufacture of bakers' yeast. J. H. LANE.

**Manufacture of sugar.** DORR Co. (B.P. 350,459, 4.12.29. U.S., 4.12.28).—See U.S.P. 1,755,165; B., 1930, 1085.

**Lixiviation of beetroot slices.**—See I. Saccharification of cellulose.—See V. Artificial wood [from cane trash].—See IX.

## XVIII.—FERMENTATION INDUSTRIES.

**Physical chemistry of the proteins, and some applications in brewing.** I. General. II. Swelling and protection, with special reference to isinglass and finings. R. H. HOPKINS (J. Inst. Brew., 1931, 37, 413—419, 419—424).—I. A review is given of the physical properties of proteins with special reference to the reaction of the medium and to the relationship of the isoelectric point to coagulation, turbidity, viscosity, and surface tension.

II. Although the max. amount of swelling of isinglass in acid solution varies with different acids, the point of max. swelling occurs for all acids at  $p_H$  3.3—3.5 within the swelling gel. The isoelectric point of finings made from isinglass is between  $p_H$  4.8 and 5.2, and the protective action of the finings is at a min. at that point.

C. RANKEN.

**Protein degradation during mashing.** W. WINDISCH, P. KOLBACH, and E. SCHILD (Woch. Brau., 1931, 48, 253—260, 267—273, 286—289, 298—300).—The optimum temp. for production of "permanently sol. N" falls with increasing duration of mashing from about 62° with a 15-min. mash to about 56° with a 3-hr. mash. The corresponding optima for formol-N are 56—60° and 47—51°. The latter action is considered to be in part due to the more heat-sensitive peptidase. A "congress" mash has approx. the same composition as regards total, permanently-sol., and formol N as a 2-hr. mash at 60°. The % formol-N in the permanently-sol. N produced falls from about 40 to 20% as the temp. is raised from 40° to 70°. This may be due to increasing inhibition of the peptidase or to a relatively less formation of simpler decomp. products by the proteinase at higher temps. With increasing duration of mashing the ratio of formol to permanently sol. N increases, probably owing to the continued action of the proteinase on the products first formed, since the effect is very marked at  $p_H$  5.1, at which reaction the peptidase action would be very weak. The optimal  $p_H$  zone for production of permanently-sol. N rises from 4.3—4.7 at 30° to 4.9—5.3 at 70°, that for formol-N being slightly lower at high temps. (4.6—5.0 at 70°). This optimum is referred to the proteinase, since at this reaction the peptidase is inactive, but below 50° the action of the latter is indicated by the higher proportion of formol-N produced at higher  $p_H$  values. The permanently-sol. N is increased by 6% on raising the mashing concentration from 1:5.3 to 1:2.0, in agreement with Schjerning (B., 1913, 985), but not with Oliver (B., 1929, 413). This corresponds to an increase of 12% on the permanently sol. N produced; the proportion of formol-N is slightly raised. The protein degradation is seriously inhibited by

mashing in brass beakers, and to a smaller extent by Ni. F. E. DAY.

**Principles of the malting process.** A. R. LING (J. Inst. Brew., 1931, 37, 424—433).—The structural changes which take place in barley during ripening and which lead to the production of mealiness are reviewed. The changes underlying the malting operations of steeping, flooring, and kilning of barley are also discussed. C. RANKEN.

**Determination of bitter principles of hops.** H. WILDNER (Woch. Brau., 1931, 48, 221—225, 231—235, 241—244, 260—263).—The methods for total resin proposed by Winge and Jensen (B., 1915, 43), Siller (B., 1909, 1056), and Wöllmer (B., 1929, 413), and those of Stádník (Böhm. Bierbr., 1927, No. 29 *et seq.*) and Hastings and Walker (B., 1929, 533) are applied to a varied selection of hop samples. Siller's and Wöllmer's methods give results in close agreement, the former usually slightly the higher. Stádník's method includes a small amount of wax etc., and the results are somewhat higher, whilst those of the Hastings-Walker method are 7.7—15% higher still. The Winge-Jensen method gives low results. For soft resins the Lintner-Adler (B., 1912, 1003), the same using hexane in place of light petroleum, Seibriger (B., 1913, 442), Stádník volumetric, and Siller, Wöllmer, Hastings-Walker, and Stádník gravimetric methods are compared. Seibriger's cold extraction with light petroleum gives low, and with  $CCl_4$  high, results as compared with Lintner and Adler's, in which the use of hexane is preferred to that of light petroleum except for old hops. The gravimetric methods of Siller, Wöllmer, and Stádník agree within the limits of experimental error, with a tendency to be higher in the order stated, and the Hastings-Walker method gives still higher results. The results of the first three methods are 0.1—1.7% higher than those of the Lintner-Adler, and, in general, agree with those of the hexane extraction, except that this tends to give low results with old samples. The tendency of the gravimetric results to be relatively high is attributed partly to small amounts of neutral material in the resin and partly to oxidation during drying. As regards humulone, both the Stádník iodometric and the Hastings-Walker Pb precipitation methods give higher results than the Siller and Wöllmer methods when applied to old samples, though they agree with fresh hops. The difference may be due to impurities in the humulone fraction on the one hand, or to imperfect precipitation of the Pb salt from extracts of old samples on the other, but the pre-extraction with phosphate buffer,  $p_H$  6.4, claimed by Windisch, Kolbach, and Winter (B., 1929, 412) to avoid this was not employed in the single comparison of these authors' method. F. E. DAY.

**Colorimetric method for the determination of the preservative value of hops.** J. M. FRENCH (J. Inst. Brew., 1931, 37, 436—439).—The MeOH used in the Guthrie and Philip method (B., 1930, 1086) for determining the preservative value of hops can be replaced by various grades of EtOH, provided the same alcohol is used both for the determination and for the manufacture of the standard solutions of  $\alpha$ -resins and U salt. 10 g. of minced hops are extracted with 100 c.c.

of the selected alcohol (98% EtOH or redistilled methylated spirit) for 10 min. in a corked bottle, and 0.5 c.c. of the extract after mixing with 10–15 c.c. of the alcohol and 7 c.c. of a 0.7% solution of  $\text{UO}_2(\text{NO}_3)_2$  in the same alcohol is made up to 50 c.c. with the alcohol in a Nessler tube and the colour compared with that of the standard solution similarly treated. As the colour is unstable, the determination should be made rapidly, and the solution of the U salt should be kept in the dark or in amber bottles.

C. RANKEN.

**Effect of high electric potentials on brewers' yeast.** N. FLORESCO and A. RAFALESCO-FLORESCO (Bul. Fac. Stiinte Cernauti, 1927, 1, 384–391; Chem. Zentr., 1931, i, 2352).—The capacity for resisting the action of p.d. of 2–320 volts depends on the age, which can thus be determined.

A. A. ELDRIDGE.

**Hemicelluloses of brewers' grains.** I. A. PREECE (J. Inst. Brew., 1931, 37, 409–413).—Two of the hemicelluloses extracted from the spent grains from malt are urono-xylans, whilst a third is a urono-araban. In no case does the amount of uronic anhydride exceed 9% of the hemicellulose. These hemicelluloses agree closely in composition and properties with those obtained from woods.

C. RANKEN.

**Determination of colour of wort and beer.** A. JANKE and A. SZILVINYI (Woch. Brau., 1931, 48, 281–295).—Comparison with I, dye, or mineral solutions, or with glass slides is subject to inaccuracy owing to differences in spectral composition of the light transmitted. I solutions are less objectionable than the other standards tested, but are unstable. A table and graphs of the readings given by dilutions of 0.1N-I solution containing 0.14–2.0 c.c./100 c.c. in the Zeiss-Pulfrich step-photometer are given, from which it is claimed that very accurate colour determinations may be made. The violet screen S43 is used, which was found unsuitable by Meštán (B., 1928, 725), who, however, employed a longer comparison cell.

F. E. DAY.

**Fermentation in open and closed vessels.** G. VIERECK (Woch. Brau., 1931, 48, 303–306).—In agreement with the results of Windisch (B., 1928, 685; 1929, 69), fermentation in closed vessels improves the appearance and fermentative power of the yeast. The closed fermentations generally proceeded rather more rapidly than those in open vessels, but no differences could be detected in the finished beers.

F. E. DAY.

**The cider industry.** G. WARCOLLIER (Chim. et Ind., 1931, 26, 15–32).—The natural sweetness of cider is due to a slow, incomplete fermentation of a thoroughly defecated juice with a low content of N, and to the action of tannin on the yeast. Juice from apples grown in flinty soils gives good defecation, whilst lime and argillaceous soils yield a rapidly fermenting juice and less stable ciders. Sweet cider can be prepared by arresting the fermentation by centrifuging the fermenting juice under pressure of  $\text{CO}_2$ , and thus decreasing the content of N and withdrawing the yeast. Similar results follow by exclusion of the air from the fermenters and chilling to 2–3°. For addition to cider, juice is conc. in vac. at 45–50° until the content of sugar is 700 g. per litre. Chilling will not raise the concen-

tration of sugar above 450 g. per litre. The juice remains stable provided the content of free  $\text{SO}_2$  is 200 mg. per litre, but 150 mg. per litre are sufficient if the juice is previously centrifuged. Unripe apples should not be used, as the enzyme which coagulates the pectic matter is inhibited by the high acidity of the juice, and the greater amount of starch left in the pulp lowers the content of EtOH in the cider. Cider brandies made by distilling naturally sweet ciders contain an abnormally high content of ethers and have a low volatile acidity.

C. RANKEN.

**Analysis of mixed alcohols.**—See III.

## PATENTS.

**Brewing of beer.** A. E. BERRY (B.P. 353,214, 28.6.30).—As a substitute for the use of patent or crystal malt in the mash tun, pale wort is boiled under pressure after the addition of 0.05–0.1% of  $\text{NH}_3$  or of  $\text{NH}_4$  salts.

C. RANKEN.

**Manufacture of vinegar.** H. FRINGS (B.P. 352,740, 14.7.30. Ger., 6.5.30).—By means of valves in the cooling-water supply and in the mash-outflow pipe, the rate and temp. of infusion are automatically co-ordinated, so that the temp. in individual zones of the filling material in the generator remains const. at an optimum temp.

C. RANKEN.

**Roasting malt etc.**—See I. **Treatment of sugar solutions.** Monosaccharide material.—See XVII.

## XIX.—FOODS.

**Vitamins. IV. Vitamin[-C] contents of common German vegetables.** F. V. VON HAHN (Z. Unters. Lebensm., 1931, 61, 545–610; cf. B., 1931, 740).—Full data and growth curves are given, and the samples are classified as follows: (1) Extremely rich in vitamin: raw white, red, and green cabbages, kohlrabi, and cauliflower; raw and cooked Brussels sprouts. (2) Rich in vitamin: raw swedes, turnips, peas, asparagus, leeks, horseradish, and green beans; cooked green and red cabbages and kohlrabi. (3) Containing vitamin: raw carrots; fresh pickled cabbage, spinach, head-lettuce, yellow beans, and comfrey; cooked turnips, peas, and yellow beans. (4) Poor in vitamin: cooked carrots, green beans, horseradish, and asparagus; raw endive, parsley, radish, pumpkin, and cucumber. (5) Almost free from vitamin: raw and cooked mushrooms and red beetroots; raw chives; old cucumber and radish; cooked pickled cucumber, spinach, comfrey, celery, cucumber, and pumpkin.

J. GRANT.

**Corrosion of metals by milk and its relation to the oxidised flavours of milk.** E. S. GUTHRIE, C. L. ROADHOUSE, and G. A. RICHARDSON (Hilgardia, 1931, 5, 425–453).—Cr-Ni alloys, pure Al, glass enamel and carefully Sn-plated metals showed little corrosion by sweet milk and imparted no flavour to the milk. Pure Ni was very unsatisfactory, and plated Cu or Cu alloys were rendered unsuitable through the rapid mechanical wearing of the plating. Aeration of milk during pasteurisation increased the tendency for the development of oxidised flavours.

A. G. POLLARD.

**Swelling of canned prunes.** E. M. MRAK and P. H. RICHERT (California Agric. Exp. Sta. Bull., 1931, No. 508, 24 pp.).—The effects of various types of

containers and of methods of procedure are examined. Sun-dried and dehydrated prunes after canning swelled at similar rates. The rate of swelling was increased by increasing the time or temp. of blanching and by pressure-cooking, and decreased by raising the concentration of the syrup ( $> 30\%$  syrup causes shrivelling). Decreasing the  $p_H$  of the syrup decreased swelling but not corrosion. A. G. POLLARD.

**Occurrence of naringin in marmalade made from S. African grape fruit.** B. SEGAL and T. DE KIEWIET (J. S. Afr. Chem. Inst., 1931, 14, 43—46).—During the storage of S. African grape-fruit marmalade white spots developed; these consisted of clusters of white radiating needles of naringenin, which disappeared when the jars were immersed in boiling  $H_2O$  and did not reappear on cooling. Naringin is shown to be a glucoside which on hydrolysis yields naringenin, which appears to be 2:4:6-trihydroxyphenyl 4-hydroxystyryl ketone. A. R. POWELL.

**Use of ethylene oxide (T-gas) in the destruction of pests in the presence of foods.** T. SUDENDORF and E. KRÖGER (Chem.-Ztg., 1931, 55, 549—550, 570—572).—From tests on numerous foodstuffs it is established that the destruction of pests (in rooms or containers used for the storage of foodstuffs) by means of ethylene oxide in concentrations up to 320 g. per cu. m. has no adverse effect on the quality or flavour of the foods provided that the space treated is thoroughly aerated after treatment. A. R. POWELL.

**Grain diseases.**—See XVI.

#### PATENTS.

**Manufacture of products having a saline taste suitable for food purposes.** DEUTS. GOLD- U. SILBERSCHNEIDENSTALT, VORM. ROESSLER (B.P. 352,673, 4.6.30. Ger., 11.6.29).—To obviate stale after-taste, Na glutamate is treated with an org. acid (formic or adipic) to form a stable double salt of pleasant taste.

E. B. HUGHES.

**Drying of tea. Roasting grain etc.**—See I.  
**Porcelain for cooking.**—See VIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Influence of variations of the concentration of alcohol in tinctures.** I. M. PERINO (Boll. Chim. Farm., 1931, 70, 489—492).—When extracted with EtOH of 35, 62.5, and 90% concentrations, *Digitalis purpurea* yields tinctures of approx. the same digitoxin contents. The use of 35—40% EtOH solution is, however, to be preferred, as the tincture obtained (1) withstands dilution without becoming turbid better than that given by the more conc. EtOH and is thus more suitable for pharmaceutical preparations, and (2) contains less dry residue which is inert or disturbing. T. H. POPE.

**Investigation and evaluation of chlorophyll and chlorophyll preparations.** (A) ZICKGRAF, (B) U. DREHMANN (Arch. Pharm., 1931, 269, 427—428, 428—429).—(A) Polemical against Drehmann (B., 1931, 223). The luminescence test should not be used to evaluate the preps. owing their therapeutic activity to chloro-

phyllin. (B) A reply. The therapeutic activity of chlorophyllin in man is unproved. R. K. CALLOW.

**Ipecacuanha preparations and their stability.** E. H. MADSEN (Pharm. Ztg., 1931, 76, 901—902).—Preparations of ipecacuanha ( $p_H$  3.6) made by double infusion in presence of a suitable amount of HCl (25 c.c.  $N-HCl$  per 50 g. drug) contain 90% of the total alkaloids, are quite stable, and retained their total activity after 3 years (cf. B., 1931, 780). E. H. SHARPLES.

**Analysis, preparation, and composition of hydrargyrum salicylicum.** F. VIEBÖCK and C. BRECHER (Arch. Pharm., 1931, 269, 398—416).—Na or K hydroxymercurisalicylates are formed by treating hydrargyrum salicylicum (*o*- and *p*-anhydrohydroxymercurisalicylic acids) with NaOH or KOH, but the isolated cryst. products are anhydrides of uncertain composition. The method of determining Hg in hydrargyrum salicylicum which depends on the replacement of Hg by I on treatment with I solution is interfered with by side-reactions (hydrolytic demercuration:  $R \cdot HgI + H_2O = RH + IHgOH$ , and nuclear iodination) which may be avoided by methods in which excess of KI is used. Total Hg or organically combined Hg alone may be determined. Na or K hydroxymercurisalicylates are not alkaline to thymolphthalein, and hydrargyrum salicylicum dissolved in excess of alkali hydroxide may be titrated with  $H_2SO_4$  either to the thymolphthalein end-point or, better, until precipitation begins. In a second method,  $Na_2S_2O_3$  is added and the alkali liberated by the reaction  $R \cdot HgOH + Na_2S_2O_3 \rightarrow R \cdot HgS \cdot SO_3Na + NaOH$  is titrated. Thirdly, salicylsalicylic acid can be determined by dissolving the substance in 0.01N-NaOH, adding KI, and titrating with acid, using phenolphthalein; excess of acid is then added, the solution refluxed until hydrolytic demercuration is complete, and the excess of acid titrated. The presence of Hg salts is detected by precipitation of Hg, when an alkaline solution of  $N_2H_4$  or of  $CH_2O$  and KBr is added to the solid substance. Analyses have been made of hydrargyrum salicylicum prepared by nine methods, and the amounts of salicylsalicylic acid and dimercursalicylsalicylic acid present as impurities, as shown by the differences in titration figures under different conditions, have been estimated. The presence of Hg salicylate and phenylmercurisalicylate is also indicated in some cases. R. K. CALLOW.

**Determination of the alkaloid content of alkaloid salts. Rapid method for the determination of alkaloids in medicinal preparations.** E. SCHULEK and F. SZEGHÖ (Pharm. Zentr., 1931, 72, 497—506).—To an amount of the substance containing 0.03—0.04 g. of the alkaloid base are added 2 c.c. of  $H_2O$ , 1—2 drops of 10% HCl, and 70 c.c. of  $CHCl_3$ . After shaking well, 3—4 drops of 10% aq.  $NH_3$  are added, the mixture is vigorously shaken, and about 4 g. of  $Na_2SO_4$  are then added in small quantities at a time. The  $CHCl_3$  is filtered off, the residue washed 3 times with 8 c.c. of  $CHCl_3$  each time, and about 90% of the combined  $CHCl_3$  extract is distilled off, 10 c.c. of 0.02N- $H_2SO_4$  are added to the residue, and the remainder of the  $CHCl_3$  is removed on the water-bath. The excess of acid is then titrated with 0.02N-NaOH. Values identical with those given

by the usual extraction methods are obtained and the method is also successfully adapted to the determination of alkaloids in presence of other substances.

E. H. SHARPLES.

**Determination of morphine in opium.** N. RUSTING (Pharm. Weekblad, 1931, 68, 767—774).—The brown coloration observed during the extraction, which persists in the separated morphine and renders titration difficult, is attributed to atm. oxidation of an unstable alkaloid generally present. Addition of  $MnCl_2$  during extraction simplifies the manipulation. The ethereal solution is titrated with  $NaOH$ , using phenolphthalein; if methyl-red be used, results are uniformly higher, probably by reason of Ca meconate present.

S. I. LEVY.

**Microtitrimetric determination of nicotine in tobacco smoke.** L. BARTA and E. TOOLE (Z. angew. Chem., 1931, 44, 682—683).—The smoke from 0.5—1 g. of tobacco is drawn over glass-wool impregnated with dil.  $H_2SO_4$ . The wool is washed successively with hot  $EtOH$  and  $H_2O$  and the washings are made alkaline and distilled with steam. Nicotine in the distillate is precipitated with picric acid. The dipicrate is distilled with steam from alkaline solution and nicotine is determined in the distillate by titration with 0.01N-HCl in presence of methyl-red.

H. WREN.

**Influence of comminution of drugs in determination of essential oils.** L. KOFLER and F. KRÄMER (Arch. Pharm., 1931, 269, 416—422).—Higher values for the content of essential oil are sometimes obtained when drugs are not powdered before steam-distillation. This occurs particularly with labiate and composite plants in which the oil is in glandular hairs or on the surface. The degree of comminution should be specified in each case. Dafert's method is the best, but NaCl should always be added to the distillate before measuring the oil layer.

R. K. CALLOW.

**Essential oil from the fruits of *Phellodendron Japonicum*, Maxim.** I. K. BRAND and G. WESTERBURG (Arch. Pharm., 1931, 269, 369—383).—The imperfectly ripe fruit yields on steam-distillation 1.1% of an essential oil, from which, by fractional distillation, myrcene may be separated. It has been characterised by conversion into *cis*-4-*isohexenyl*- $\Delta^4$ -tetrahydrophthalic anhydride by treatment with maleic anhydride (Diels and Alder, A., 1929, 819). The amount of condensation product formed directly from the crude oil corresponds to the presence of 78.4% of myrcene.

R. K. CALLOW.

**Use of the analytical quartz lamp for the examination of perfumes.** E. EKMAN and A. SANAYSH-LAYEVA (Riechstoffind., 1930 5, 196—197; Chem. Zentr., 1931, i, 2403).—The fluorescence in ultra-violet light of the following is described: geranyl acetate, benzyl acetate, Et benzoate, *iso*amyl acetate, and benzyl salicylate.

A. A. ELDRIDGE.

**Fe in pharmaceutical preps.**—See XXIII.

#### PATENTS.

**Preparations for immunising against infectious diseases.** E. LOEWENSTEIN (B.P. 352,210, 17.6.30. Austr., 19.6.29. Addn. to B.P. 314,854; B., 1930, 640).

—Salves, protective against diseases other than diphtheria, are prepared by adaptations of the method described previously; absorption of such salves is increased either by increasing the dispersive power or adding substances which irritate the skin.

E. H. SHARPLES.

**Antiseptic and disinfectant solutions.** PEPSODENT Co. (B.P. 352,397, 2.4.30. U.S., 3.4.29).—Dil. chlorothymol solution (0.01—0.5%) is germicidal if acidified to  $pH$  4.5 or less with an org. acid. The solvents used are  $H_2O$ -miscible monohydric alcohols, the harsh flavour of which is reduced by polyhydric alcohols or sugar products in the case of mouth-wash solutions.

E. B. HUGHES.

**Manufacture of  $\beta$ -hydroxy-derivatives of alkylpyridines.** I. G. FARBENIND. A.-G. (B.P. 348,988, 18.9.30. Ger., 21.10.29. Addn. to B.P. 335,818; B., 1931, 13).—The process of the prior patent is extended to other homologous pyridines. 2-Hydroxy-4-methylpyridine, m.p. 118—120°, b.p. 285—290° (slight decomp.), and 2-hydroxy-6-ethylpyridine, m.p. 170—173°, are obtained from the corresponding sulphonc acids.

C. HOLLINS.

**Manufacture of derivatives of 1-benzyl-3-methylisoquinoline.** G. B. ELLIS. From E. MERCK (B.P. 348,956, 15.7.30).—The Bischler-Napieralski method is applied to the synthesis of papaverine-like compounds containing a 3-Me group. The amide, m.p. 146°, from homopiperonylic acid and  $\beta$ -(3:4-methylenedioxyphenyl)isopropylamine is heated in PhMe with  $POCl_3$  to give a dihydroisoquinoline, m.p. 130° [hydrochloride, m.p. 237° (decomp.)], which is dehydrogenated with Pd-black to give 6:7-methylenedioxy-1-(3':4'-methylenedioxybenzyl)-3-methylisoquinoline, m.p. 141° [hydrochloride, m.p. 254° (decomp.)]. The corresponding homoveratric amide, m.p. 123°, and  $\alpha$ -phenyl-*n*-butyramide, m.p. 138°, give isoquinolines, m.p. 125° [hydrochloride, m.p. 220° (decomp.)] and 143° (hydrochloride, m.p. 80°), respectively. 3-Methylpapaverine, m.p. 136° [hydrochloride, m.p. 234° (decomp.)], is similarly obtained from the  $\beta$ -3:4-dimethoxyphenylisopropylamide of homoveratric acid, and analogous isoquinolines, m.p. 109° and 168—169°, respectively, from the corresponding amides of phenylacetic and homopiperonylic acids.

C. HOLLINS.

**Manufacture of *o*-nitroarylarsinic acids.** I. G. FARBENIND. A.-G. (B.P. 349,037, 19.2.30. Ger., 19.2.29).—An *o*-nitroarylamine (e.g., 2:4-dinitroaniline, 7-nitro-6-amino- and 6-nitro-7-amino-benzomorpholones, *o*-nitroaniline, 3-nitro-*p*-phenetidine, 3-nitro-*p*-tolyl  $\beta$ -hydroxyethyl ether, 3-nitro-*p*-aminoacetanilide, 5-chloro-2:4-dinitroaniline, 2:5-dinitro-4-aminophenylacetic acid, 5:6- and 6:5-nitroaminobenzoxazolones) is diazotised in presence of  $As_2O_3$  or an arsenite in absence of a reducing agent. Preferably an accelerating agent (Cu, kieselguhr, animal charcoal) and/or NaOAc or other sol. org. salt are/is added.

C. HOLLINS.

**Manufacture of easily soluble [ $\beta$ -hydroxyethylamine] salts of 3-acetamido-4-hydroxybenzene-arsinic acid.** I. G. FARBENIND. A.-G. (B.P. 348,921, 13.6.30. Ger., 14.6.29).—The arsinic acid is pasted with aq. aminoethyl alcohol, dissolved in  $H_2O$ , and



EtOH is added; the salt which crystallises out is more sol. and better tolerated than are other salts. C. HOLLINS.

**Cigarette paper.**—See V. Substances containing S.—See VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Wave-length and colour balance in screen plates.** T. T. BAKER (Brit. J. Phot., 1931, 78, Col. Suppl., 25, 29—30).—Gamma infinity is reached in min. development time when the gelatin: Ag ratio in an emulsion is as low as is practicable. Change in gradation due to wave-length is then obviated. J. LEWKOWITSCH.

**Antifogging agents in [photographic] developers.** II. A. P. H. TRIVELLI and E. C. JENSEN (J. Franklin Inst., 1931, 212, 155—169; cf. B., 1930, 1005).—Of many compounds tried, only cysteine hydrochloride and thiobarbituric acid showed antifogging properties. Their action is less than that of 6-nitrobenzimidazole, and produces greater density depression. Photomicrographs of the action of these compounds, KBr, and thioacetanilide are given. J. LEWKOWITSCH.

**Relative masses of photo-silver and sensitivity specks in the photographic latent image.** S. E. SHEPPARD (Phot. J., 1931, 71, 313—317).—In emulsions digested with gelatin containing labile S, the average amount of  $\text{Ag}_2\text{S}$ , per g. AgBr, is  $5 \times 10^{-4}$  g. The no. of photo-Ag atoms per g. AgBr is too small to agree with "crit. size" theories of latent image nuclei. The theory that the essential process is the orientation of Ag atoms to initiate a Ag space lattice is supported. J. LEWKOWITSCH.

### PATENTS.

**Protective paper strips for photographic roll films.** I. G. FARBERIND. A.-G. (B.P. 353,324, 29.9.30. Ger., 16.12.29).—Paper sized with montan wax is quite pliable and has no tendency to uncoil quickly after winding. J. LEWKOWITSCH.

**Manufacture of transparent non-inflammable film.** I. KAWAI (B.P. 343,702, 21.1.30).—An aq. solution of agar-agar is treated with  $\text{CH}_2\text{O}$  (40%), boiled, diluted with  $\text{H}_2\text{O}$ , and set to a hard jelly. This is shredded, washed free from excess  $\text{CH}_2\text{O}$ , remelted, filtered, and used to prepare the film. J. W. GLASSETT.

**Photographic emulsion for electronic discharge.** A. G. COOLEY (U.S.P. 1,791,053, 3.2.31. Appl., 7.6.28).—Finely-divided metallic particles (e.g., Mg) are incorporated in the emulsion used for recording corona discharge in the process of U.S.P. 1,702,595, thereby increasing the potential gradient through the emulsion and diffusing the discharge. J. LEWKOWITSCH.

**Imparting printing capability to photographic images in emulsions which are excessively poor in silver.** F. LIERC (B.P. 345,311, 18.9.29).—The Ag image is converted into a mordant compound, e.g., AgI or  $\text{Cu}_2\text{I}_2$ , and treated with a solution of a phenosafranine alone or admixed with a yellow dye such as thioflavine-yellow. The dye can be fixed by a lake-forming substance, and the mordant compound blackened with  $\text{Na}_2\text{S}$ . J. W. GLASSETT.

**Colour photography. [Multicolour screens on film.]** J. N. GOLDSMITH, T. T. BAKER, C. BONAMICO, and SPICERS, LTD. (B.P. 352,949, 14.4.30).—The film is impregnated with one component of a dye, e.g.,  $\beta$ -naphthol or Naphthol AS in alcoholic NaOH or KOH. Red lines are produced by ruling with a solution of a diazo compound, e.g., diazotised  $\alpha$ -naphthylamine or Fast Scarlet Salt GG, and blue lines, crossing these, with, e.g., diazotised dianisidine or Variamine Blue Salt B. A third (yellow) dye, e.g., Auramine, may be applied. No overlap colours are formed, as the impregnated solution is exhausted where the previous diazo compound is ruled on. Pretreatment of the film for dyeing is unnecessary. J. LEWKOWITSCH.

**Colour photography.** J. N. GOLDSMITH, T. T. BAKER, C. BONAMICO, and SPICERS, LTD. (B.P. 339,238, 1. and 14.8.29).—In the preparation of multicolour screens the dyes are confined to the surface layers of the film by using (a) a non-penetrative solvent, (b) a viscous solution of the dye, e.g., in castor oil or gum arabic, (c) a suspension of the dye, or (d) by rubbing or spreading a powdered dye on the surface of the film or forcing a finely-divided dyed substance in contact with the film. J. W. GLASSETT.

**Colour photography.** I. G. FARBERIND. A.-G. (B.P. 345,575, 4.6.30. Ger., 4.6.29).—The alkali salts of the sulphuric leuco-esters of indigosol-golden-yellow ICK, indigosol-blue OCB, and indigosol-pink IR extra are recommended for the preparation of yellow, blue, and red images, respectively, by direct exposure. A suitable support is sensitised with the leuco-ester, exposed under the appropriate component negative, and the unchanged ester removed by a suitable solvent. The process is repeated on the same or on separate supports for the other colours. A substance having an oxidising action when exposed to light, e.g., inorg. nitro-compounds, is added as a sensitiser. J. W. GLASSETT.

**Light-sensitive paper.** J. TACKELS (B.P. 348,281, 13.3.30).—The paper support is treated with dil.  $\text{H}_2\text{SO}_4$  and then coated with a Ag halide emulsion containing 0.2—6% of fatty or oily substances. Examples are: poly-alcohols (e.g., glycerin), lanoline, vaseline, or hydrocarbon derivatives (liquid paraffin). J. W. GLASSETT.

**Manufacture of photographically sensitive materials.** GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 344,354, 29.11.29).—Plates of Cu or its alloy are sensitised by bathing in a 10% aq. solution of  $\text{CuCl}_2$  followed by rinsing in  $\text{H}_2\text{O}$  and in EtOH. The image is fixed by immersion in a dil. solution of a neutral salt, e.g., NaCl or KBr. J. W. GLASSETT.

**Manufacture of [light]-sensitive diazotype layers.** N. V. CHEM. FABRIEK L. VAN DER GRINTEN, Assees. of F. VAN DER GRINTEN (B.P. 344,721, 14.5.30. Holl., 15.5.29).—Difficultly sol. diazo compounds such as the diazonium salts of chlorostannic acid are used in the sensitive layer, which also contains (a) acid-reacting substances and hydroxylamine to lower the coupling activity, (b) neutral salts which reduce the spreading of the image, and (c)  $\text{NH}_4$  salts which buffer the alkali of the developer. The support is also treated with acid and salt solutions, and, after coating, the paper is subjected to intense drying. J. W. GLASSETT.

**Manufacture of photographic diazotype prints.** M., G., H., and A. RENKER (DÜRENER FABR. PRÄPARIER-TER PAPIERE RENKER & Co.) (B.P. 353,486, 27.2.30. Ger., 22.3.29).—Ureas (*e.g.*, thiourea) or  $\text{NH}_4$  salts of org. acids are added to the diazo compound in the paper, to prevent discoloration. Shrinkage of the paper is avoided if excess developer is rapidly applied and immediately removed by squeegee rollers.  $\text{Na}_2\text{S}_2\text{O}_3$  may be added to the developer. J. LEWKOWITSCH.

**Fixing of light-sensitive layers containing basic dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 348,102, 13.8.29).—After exposure, the sensitive layer is treated with a 12% aq. solution of Na fluoborate followed by a bath of 1% concentration, whereby the sensitiser is removed but the dye is converted into the insol. fluoborate. J. W. GLASSETT.

**Preparation of photo-sensitive layers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,511, 25.4.30).—Photographic emulsions made with vegetable colloids, particularly polyglycuronic  $\text{NH}_4$  salt, are softened by incorporation of soaps or sulphonic derivatives of fatty compounds. The tendency to cloudiness is reduced by addition of aromatic sulphonic salts, especially the Na salt of mono- or di-butyl-naphthalene-sulphonic acid. J. LEWKOWITSCH.

**Production of photomechanical printing surfaces.** H. T. P. GEE. From MEISENBACH RIFFARTH & Co., A.-G. (B.P. 348,162, 7.11.29).—After development, the traces of unexposed chrome colloid remaining on the plate are removed by applying a mixture of a dehydrating agent and  $\text{HNO}_3$ . J. W. GLASSETT.

**Photomechanical printing surfaces.** PINCHOT-NEUMANN HOLDING CORP. (B.P. 349,933, 27.2.30. U.S., 27.7.29).—Gelatin printing surfaces are applied to metal plates by means of a transfer paper consisting of a paper support coated with a  $\text{H}_2\text{O}$ -sol. layer of sugar and starch, and a superimposed layer of gelatin, which may be sensitised. J. W. GLASSETT.

**Production of photomechanical printing plates for relief printing.** N. LUDVIGSEN (B.P. 345,255, 13.12.29. Fr., 19.12.28).—A negative resist screen is applied direct to the metal plate and a layer of sensitised colloid superimposed upon it. J. W. GLASSETT.

**Photogravure processes.** S. SAWARAGI (B.P. 346,483, 22.1.30).—The Cu plate is coated (as a substratum) with a mixture containing beer 100 c.c.,  $\text{Na}_2\text{SiO}_3$  (1:1) 120 c.c., egg albumin 100 g., water 100 c.c., and while wet the sensitised gelatin solution is poured on as the plate is heated and rotated. J. W. GLASSETT.

**Device for ascertaining the gradation of graduated series of blackenings in photographic layers.** I. G. FARBENIND. A.-G. (B.P. 353,641, 19.6.30. Ger., 20.12.29).

**Production [with copying apparatus] of prints from photographic negatives.** DÜRKOPFERWERKE A.-G. (B.P. 353,964, 12.5.30. Ger., 21.2.30).

## XXII.—EXPLOSIVES; MATCHES.

**Detonating ability and explosive power of chlorate mixtures.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930,

8, 115—118; Chem. Zentr., 1931, i, 2426).—With addition of liquid hydrocarbons detonation proceeds more readily than when solid hydrocarbons are used. Detonating ability rapidly diminishes with increase in hydrocarbon content, but markedly increases on addition of nitrobenzene or nitrotoluene. Mixtures with carbohydrates, soot, or metals are less shattering. Mixtures of  $\text{KClO}_3$  with up to 3% of sawdust are not explosive; with at least 10% shattering explosives are produced.

A. A. ELDRIDGE.

**Pentaerythrityl tetranitrate and glyceryl nitrate.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 121; Chem. Zentr., 1931, i, 2426).—A mixture (80:20) is pulverulent, but cakes on slight pressure; with more than 20% of glyceryl nitrate, or at a higher temp., the oil separates in droplets. The explosive properties are recorded.

A. A. ELDRIDGE.

**Sources of error in the determination of the chemical stability of cellulose nitrate and smokeless powders by Bergmann and Junk's method.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 28—31; Chem. Zentr., 1931, i, 2427).—Deviations are attributed to separation of  $\text{H}_2\text{O}$  which hydrolyses the cellulose nitrate. The small separation of NO from powders containing  $\text{NHPh}_2$  at high temp. is ascribed to the fact that at low temp.  $\text{NHPh}_2$  is deposited on the cooler particles as a fused sublimate, and that at high temp. this remains gaseous and combines with liberated NO.

A. A. ELDRIDGE.

**Determination of chemical stability of smokeless powder and explosives by determination of hydrogen-ion concentration.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 26—28; Chem. Zentr., 1931, i, 2426—2427).—The test is of value only when the  $\text{CaCO}_3$  content is not too high (0.5—2%). Addition of amyl alcohol,  $\text{NH}_2\text{Ph}$ , and inorg. salts of strong acids is without effect, but the presence of dichromate leads to incorrect results. The method may be applied to celluloid, the presence of  $\text{ZnO}$  and  $\text{PbCrO}_4$  not affecting the results. The specimens are heated to decomp., and extracted with dil. aq.  $\text{HNO}_3$  ( $p_{\text{H}}$  5.5). With cellulose nitrate or glyceryl nitrate powders heated at moderately high const. temp. the  $p_{\text{H}}$  val. of the aq. extract at the beginning of heating usually falls considerably. The decomp. velocity diminishes with stable powders after about 2 hr., after which the decomp. usually proceeds continuously. After a  $p_{\text{H}}$  val. of about 2.5 has been reached, the evolution of acid vapours again increases until  $p_{\text{H}}$  1.3 is reached, when the quantity of  $\text{HNO}_3$  liberated again diminishes with simultaneous formation of slightly dissociated acids, *e.g.*, oxalic and mucic.

A. A. ELDRIDGE.

**Explosive properties of trinitronaphthalene.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 119—121; Chem. Zentr., 1931, i, 2426).—The behaviour of technical material, m.p. 145—155° (N 14—14.2%), granulated or compressed, is recorded.

A. A. ELDRIDGE.

**Guncottons.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 22—24; Chem.

Zentr., 1931, i, 2412).—The degree of degradation of the denitrated cellulose of 12 samples of cellulose nitrate was determined by means of the Cu no., Ba(OH)<sub>2</sub> resistance, and solubility in 5% NaOH. A. A. ELDRIDGE.

**Significance of composition of nitroglycerin waste acids in the rational manufacture of nitroglycerin.** G. WALLERIUS (Proc. World Eng. Cong., 1929, 31, 75—76).—The yield of nitroglycerin is a function of the wt. of waste acid and the solubility of nitroglycerin therein. Hence for any given nitrating acid mixture there is an optimum waste-acid composition. The solubility of nitroglycerin in waste acids containing 10—16% HNO<sub>3</sub> with 12—20% H<sub>2</sub>O content was determined, it being known that 13% H<sub>2</sub>O is the low limit for H<sub>2</sub>O content and 17% HNO<sub>3</sub> the high limit for HNO<sub>3</sub> content. On the basis of these results the ratio of glycerin to mixed acid and the corresponding concentration of HNO<sub>3</sub> in the latter for varying H<sub>2</sub>O content are calc. C. IRWIN.

**Explosive fertilisers.**—See XVI.

#### PATENTS.

**Nitrocellulose. Pyroxylin sheets.**—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Rapid determination of the active content of insecticides.** VI. **Titrimetric determination of arsenic.** A. TERÉNYI and J. PÁSKUJ (Z. anal. Chem., 1931, 84, 416—441).—As may be quantitatively distilled from a conc. HCl solution; KBr does not aid the distillation of As<sup>+++</sup> or As<sup>++++</sup>, but it takes part in the reduction of the latter by FeSO<sub>4</sub>. In 19—28% HCl solution KBr and, particularly, H<sub>2</sub>SO<sub>4</sub> aid the distillation, and the H<sub>2</sub>SO<sub>4</sub>, in addition, accelerates the reduction of As<sup>++++</sup>. Details are given of the determination of As in both easily and difficultly decomposable insecticides; for the latter, 4—5 hr. are required, and a micro-method, requiring only about 1.5 hr., is recommended. H. F. GILLBE.

**Manipulation of  $p_H$  [of water] at Springfield, Ill.** C. H. SPAULDING (J. Amer. Water Works' Assoc., 1931, 23, 1190—1201).—By adding Ca(OH)<sub>2</sub> at a varying instead of a uniform rate an increased reduction of 10 p.p.m. of total hardness is obtained, probably due to precipitation of Mg during periods of high  $p_H$ . Scrubbed and filtered flue gas is used for recarbonation, but deposits on the filter sand have been observed (attributed by Fleming in the discussion on the paper to an insufficient settling period between carbonation and filtration). Sterilisation produced by lime treatment is thought to be due more to mechanical removal by the bulky floc obtained than to any direct toxic action. C. JEPSON.

**Silicic acid in mineral water analyses.** F. HUNDESHAGEN and F. W. SIEBER (Z. angew. Chem., 1931, 44, 683—685).—Sources of error are discussed. H. WREN.

**Colorimetric determination of iron in drinking water and [pharmaceutical] iron preparations.** K. SCHERINGA (Pharm. Weekblad, 1931, 68, 735—738).—The precautions to be taken when the thiocyanate method is employed in presence of org. and other materials are described. S. I. LEVY.

**Destruction of pests.**—See XIX.

#### PATENTS.

**Sterilisation of liquids.** G. A. KRAUSE (B.P. 353,409 and Addn. B.P. 353,686, [A] 21.2.30, [B] 19.7.30. Ger., [A] 1.3.29).—(A) Very low concentrations of salts of oligodynamically active metals (not exceeding 2 mg./litre) can be utilised for the sterilisation of H<sub>2</sub>O on the large scale if the time of exposure is sufficiently long and contact with baser metals avoided. (B) Very sparingly sol. salts, e.g., AgBr, are substituted for freely sol. ones, thus avoiding possibility of errors, e.g., overdosing, and manipulative troubles without sacrificing sterilising efficiency. The valuable metals may be recovered after use by deposition on a collector of a suitable base metal, e.g., Fe or Al. C. JEPSON.

**Vermin-destroying compositions.** DEUTS. GOLD-Ü. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 353,354, 14.11.30. Ger., 14.11.29).—Low-boiling, gaseous or liquid vermin-destroying substances, e.g., ethylene oxide, CNCl, or HCN, are mixed with solid CO<sub>2</sub>. Alternatively, the substances may be cooled to the solid state before mixing, or they may be solidified after mixing with liquid CO<sub>2</sub>. W. J. WRIGHT.

**Disinfection, and destruction of insect pests.** A. CARPMAEL. FROM I. G. FARBERIND. A.-G. (B.P. 349,004, 12.11.29, 10.12.29, and 24.6.30).—Dihydroxydiaryl sulphides or polysulphides or their alkali salts are used in bactericidal, fungicidal, and insecticidal preparations. Examples are: 5 : 5'-dichloro-2 : 2'-dihydroxydiphenyl sulphide with soft soap for use against animal pests, or in water against mildew or moths in fabrics, or with talcum etc. for face powder or against *B. pyocyaneus*; the 5 : 5'-dibromo-compound with talcum against mildew on roses, or with talcum and a wetting agent for disinfecting seed grain, or in aq. NaOH for treating anthrax-infected skins or dead wood, or in aq. NH<sub>3</sub> for addition to albumin solutions; 4 : 4'-dihydroxydi-*o*-tolyl sulphide with soft soap; 4 : 4'-dihydroxydiphenyl sulphide with glycerin, wool fat, and petroleum jelly as a cosmetic against hyperidrosis, or the 2 : 2'-diarsinic acid with talcum against mildew on roses; 4 : 4'-dihydroxy-2 : 2'-dimethyl-5 : 5'-diisopropylidiphenyl sulphide in H<sub>2</sub>O against staphylococci; 3 : 3'-dibromo-4 : 4'-dihydroxydi-*m*-tolyl sulphide with yellow wax in disinfectant polishes. C. HOLLINS.

**Treatment of [coke-oven] effluents.** W. W. GROVES. FROM KLÄR & ENTPHENOLUNGSGES. M.B.H. (B.P. 353,625, 10.6.30).—The effluents after dephenolating by solvent extraction are aerated by compressed air, cascades, etc. at 50—60°, whereby thiosulphates, pyridines, etc. are oxidised or volatilised. The waste air is discharged through the chimney stack. C. JEPSON.

**Incinerator structures.** H. KLEIN (B.P. 353,623, 6.6.30).

**Water-softening systems.** FLORIDA WATER SOFTENER, INC., Assees. of W. E. DUNBAR (B.P. 353,543, 1.5.30. U.S., 8.5.29).

**Depuration of waste water containing cyanide.**—See VII. **Electrolysis of aq. solutions.**—See XI. **Disinfectant.**—See XX.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

OCT. 9 and 16, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Acid-resisting materials for chemical and building industries.** P. N. GREGORIEV and I. I. SILVESTROVICH (J. Appl. Chem., Russia, 1930, 3, 1155—1158).— $\text{Na}_2\text{SiF}_6$  is recommended as a binder for materials containing silicates. CHEMICAL ABSTRACTS.

**High-pressure boiler plants [at Mannheim].** MARGUERRE (Engineering, 1931, 132, 267—271).—In boilers working at 1422 lb./sq. in. pressure, failures occurred owing to tubes in which the circulation of  $\text{H}_2\text{O}$  was doubtful, being exposed to too high a temp. Traces of  $\text{O}_2$  in the feed water hastened the failure. The trouble was overcome by increasing the section of the down tubes in order to withdraw the tubes with doubtful circulation into zones of cooler gas, by making alterations in the drums, so that the tubes acted as either downtakers or risers and by adding  $\text{Na}_2\text{SO}_3$  to the feed water to reduce the  $\text{O}_2$  content to 0.01 p.p.m., and  $\text{Na}_3\text{PO}_4$  to give a soda alkalinity of 100 p.p.m. In the first section of the superheater, of mild steel, the steam is heated to  $400^\circ$ ; in the second section, of 3% Ni steel or, later, Cr-Mo steel, to  $470^\circ$ .  $\text{H}_2\text{O}$  thermostatically controlled is injected into the latter section at max. loads. When starting up,  $\text{H}_2\text{O}$  is circulated through the superheater and boiler until 427—711 lb./sq. in. pressure is reached, the circulation is then stopped, and the  $\text{H}_2\text{O}$  blown from the superheater. A constant-pressure steam accumulator is in operation. Feed pumps, valves, and pipe joints suitable for high-pressure plant are described. D. K. MOORE.

**Volumetric determination of sulphate [in boiler feed-water].** D. NORTHALL-LAURIE (Analyst, 1931, 56, 526—527).—The  $\text{SO}_4^{--}$  may be determined approx. by shaking with a suspension of  $\text{BaCO}_3$ , filtering, and titrating the increase in alkalinity. T. McLACHLAN.

**Efficiency of the Eirich rapid mixer.** L. ZAKARIAS (Chem.-Ztg., 1931, 55, 597).—The Eirich rapid mixer (B., 1930, 931) was found to require only 5—10 min. to render a mixture of polysaccharide paste and soda perfectly homogeneous, against 1 hr. for a kneading machine of the usual type. C. IRWIN.

**Extraction apparatus for rubber, cellulose, fats, etc.** W. H. STEVENS (Analyst, 1931, 56, 528—529).—A modification of the apparatus of Beadle and Stevens (A., 1913, ii, 444) is described, in which a small, wide-necked, conical flask is fitted with an internal, removable, spiral condenser supporting the extraction thimble, thereby enabling the extract to be weighed in its original container. T. McLACHLAN.

**Filtration and electro-ultrafiltration.** K. SILBEREISEN (Woch. Brau., 1931, 48, 381—385).—A short bibliography and summary of laboratory methods. F. E. DAY.

**Moisture content of saturated gases and the quantities of water which separate on cooling such gases.** F. HURDELBRINK (Gas- u. Wasserfach, 1931, 74, 685—686).—The vol., moisture content, and heat content of a saturated gas are tabulated as functions of the temp. over the range  $-10^\circ$  to  $80^\circ$  in steps of  $10^\circ$ , in such a manner as to facilitate the calculation of the quantity of  $\text{H}_2\text{O}$  or heat given up on cooling a gas over any temp. interval. A. B. MANNING.

**High-pressure compressors.**—See VII. **Porcelain grinding balls etc.**—See VIII. **Microturbidimeter.** **Tensile-testing machine.**—See XIV.

See also A., Sept., 1026, **Calibrating low-temp. and Beckmann thermometers.** **Thermostats.**

### PATENTS.

**Counterflow furnace.** V. A. HAIN, Assr. to G. J. HAGAN Co. (U.S.P. 1,792,423, 10.2.31. Appl., 16.4.28).—In an elongated furnace in which the incoming goods are preheated by the outgoing goods moving in an opposite direction on a parallel track, the transfer of heat is aided by transverse circulation of the atm. produced by fans and guided by a horizontal partition between the rows of articles. B. M. VENABLES.

**Reversing regenerative furnaces.** A. V. KEMP (B.P. 354,399, 26.11.30).—Two streams of air and one of gas are admitted at different angles and levels to a mixing chamber, whence the mixture passes to the furnace under an adjustable arch which is raised when the furnace is reversed to afford a larger passage for products of combustion. B. M. VENABLES.

**Retort.** W. F. RIEGER, Assr. to STOKER MATIC CORP. (U.S.P. 1,790,840, 3.2.31. Appl., 13.8.28).—A retort for a boiler furnace is provided with a fuel compartment which is surrounded by air under forced draught and, again round the upper part, by a water-cooled passage. The air enters the fuel sideways. B. M. VENABLES.

**Dryer.** J. B. and J. J. BERRIGAN, Assrs. to PRESS & DRIER Co. (U.S.P. 1,792,064, 10.2.31. Appl., 14.6.29. Cf. U.S.P. 1,770,727; B., 1931, 278).—Material, e.g., thickened sewage, is passed through narrow spaces between highly-heated plates by means of pump pistons to which are attached blades extending into the spaces. B. M. VENABLES.

**Heat exchangers for heating air and water by furnace gases from steam generators.** D. ADAMSON & Co., LTD., and G. KERFOOT (B.P. 353,087,

\* The remainder of this set of Abstracts will appear in next week's issue.

25.4.30).—The heat exchanger consists of several parallel passages with tubes inside alternate ones. Hot gas flows through the passages containing the tubes, thereby heating the water flowing both through the tube and the passage walls, and these heat the air flowing through the adjacent passages. D. K. MOORE.

**Heat-transferring apparatus, particularly applicable to heating air.** A. E. WHITE, From DOHERTY RES. Co. (B.P. 351,950, 1.3.30).—The apparatus is suitable for heating a fluid under pressure, *e.g.*, compressed air, by means of another fluid such as exhaust gases from an engine. The tubes for the fluid under pressure are coiled so that the turns lie alternately in two parallel planes with the openings of the loops in one plane opposite the tubes of the loops in the other plane. Groups of tubes are placed in recesses formed in a mass of refractory material and forming the passages for the outer fluid. B. M. VENABLES.

**Fire-extinguishing material.** D. J. BLOCK, Assr. to DU-GAS FIRE EXTINGUISHER CORP. (U.S.P. 1,793,420, 17.2.31. Appl., 22.9.28).—The material comprises a mixture of 97% of  $\text{NaHCO}_3$ , 1.5% of Mg stearate, 1% of  $\text{MgCO}_3$ , and 0.5% of  $\text{Ca}_3\text{P}_2\text{O}_8$ . The addition of these salts to the  $\text{NaHCO}_3$  prevents caking under humid conditions. A. R. POWELL.

**Absorbent for refrigerants [ammonia].** R. M. BUFFINGTON, Assr. to FRIGIDAIRE CORP. (U.S.P. 1,792,628, 17.2.31. Appl., 27.6.29).—The absorbent comprises anhyd.  $\text{SrCl}_2$  mixed with 5% of  $\text{NH}_4\text{NO}_3$ . A. R. POWELL.

**Crusher.** E. H. FRICKEY (U.S.P. 1,791,669, 10.2.31. Appl., 5.10.28).—In a crusher of the single-roll or hammer type, adapted to treat clayey or sticky material, with the fixed (adjustable) breaker plate comparatively short, a tangential continuation is formed by a plate conveyor or feeder running downwards, and in the other direction adjoining the crusher plate are grid bars arranged on a circle. B. M. VENABLES.

**Crushing machinery.** H. F. CAMPBELL, Assr. to PENNSYLVANIA CRUSHER Co. (U.S.P. 1,791,712, 10.2.31. Appl., 17.1.30).—A hammer crusher is adapted to treat clayey or sticky material by the provision of one or more scraper bars reciprocating over the breaker plate; the bars are carried by chains at their ends and the chains are continued as racks which are reciprocated by pinions. B. M. VENABLES.

**Cone crusher.** E. B. SYMONS, Assr. to NORDBERG MANUFG. Co. (U.S.P. 1,791,584, 10.2.31. Appl., 9.12.29).—A method of excluding dust from the spherical bearing of a conical gyratory crusher by means of an air current and wiping members is described. B. M. VENABLES.

**Crushing apparatus.** N. D. LEVIN, Assr. to JEFFREY MANUFG. Co. (U.S.P. 1,787,828, 6.1.31. Appl., 1.6.27).—A form of disintegrator in which rigid hammers are supported between discs is described. B. M. VENABLES.

**Crushing machinery.** G. W. BORTON, Assr. to PENNSYLVANIA CRUSHER Co. (U.S.P. 1,789,539, 20.1.31. Appl., 12.12.29).—The breaking surface of a hammer crusher is provided with scraping blades to prevent accumulation of pasty material. B. M. VENABLES.

**Hammer for hammer mills.** C. E. SPITZER (U.S.P. 1,791,007, 3.2.31. Appl., 13.9.28).—The hammers comprise flat pieces of metal with grooves on the edges which are inclined in opposite directions on opposite edges. B. M. VENABLES.

**Mill for grinding and mixing ores, colours, chemicals, cements, etc.** T. BREAKELL (B.P. 353,042, 5.4.30).—The mill consists of a mortar with rotating mullers, each muller also rotating about its vertical axis. D. K. MOORE.

**Mixing machine.** W. F. DEHUFF, Assr. to GLEN MIXER Co. (U.S.P. 1,792,363, 10.2.31. Appl., 11.12.28).—A form of joint enabling a beater to be quickly attached to its shaft is described. B. M. VENABLES.

**Mixing of substances.** J. G. JACKSON (B.P. 353,958, 7.5.30).—Fluid or semi-fluid substances are delivered in concentric streams by separate helical conveyors and are further mixed between rotating discs or cones. B. M. VENABLES.

**Mixing machines for rubber and such like mixes.** C. F. SCHNUCK and F. H. BANBURY (B.P. 354,585, 9.9.30. U.S., 23.5.30).—The charging ram of a mixer is provided with a sloping upper face and with pipes for cooling agent for the purpose described in B.P. 352,803 (B., 1931, 867). B. M. VENABLES.

**Vertical batch mixer.** A. SCHUCHARDT (U.S.P. 1,791,169, 3.2.31. Appl., 22.11.28).—A mixing machine for dry powders comprises a cylindrical vessel having a lower part tapering to a discharge neck. An axial worm is rotated in a direction to lift the material and when mixing makes a close fit in the neck; for discharge, the whole worm is raised clear of the neck. B. M. VENABLES.

**Devices for blending, mixing, agitating, and aerating liquids and kindred uses.** W. H. CARTER-LEE (B.P. 354,403, 13.5.30).—The apparatus comprises a perforated, hand-operated piston working in a cylinder which is provided with a quickly detachable end; the latter is deeply grooved and the piston shaped to suit so as to eliminate dead material at the end of the stroke. B. M. VENABLES.

**Centrifugal dust separator.** T. FRÖHLICH A.-G. (B.P. 354,608, 1.10.30. Ger., 1.10.29).—The inlet of a cyclone separator is divided either vertically or horizontally into several passages the walls of which all terminate on the separator casing at the same radius from the outlet tube. Means such as a flap shutter are provided to cut off some of the passages at times of reduced load so as to maintain the velocity of the air. B. M. VENABLES.

**[Centrifugal] separator.** P. B. SHEE, Assr. to SEARS, ROEBUCK & Co. (U.S.P. 1,791,250, 3.2.31. Appl., 5.4.30).—A method of securing the bowl and other parts on the driving element is described. B. M. VENABLES.

**Driving mechanism of centrifuges.** AKTIEB. SEPARATOR (B.P. 354,613, 10.10.30. Swed., 12.10.29).—A system of driving through clutches avoiding belt slip is described: the worm-wheel (which drives a worm on the separator shaft) forms the female part of a friction clutch for use when accelerating, and in the driving

pulley is embodied a ratchet clutch which permits the centrifuge to overrun should the source of power slow up.  
B. M. VENABLES.

**Continuous separation of granular and pulverulent material.** J. C. PEARSON and F. A. HITCHCOCK (U.S.P. 1,791,426, 3.2.31. Appl., 8.9.28).—The material is fed in at one end of the lower part of a casing of which the upper part leads to a bag filter or other means of collecting dust. The lower part of the casing is V-shaped and the material is stirred up by air jets in or near the point of the V, the coarse material being gradually worked to an outlet at the end remote from the feed. A preferred arrangement comprises an air pipe with downwardly directed jets which is reciprocated through a distance greater than the pitch of a corrugated strip of metal which is laid in the bottom of the V.

B. M. VENABLES.

**Apparatus for separating solids from liquids by filtration.** J. T. SHIMMIN (U.S.P. 1,791,251, 3.2.31. Appl., 14.9.25. Renewed 20.10.30).—The apparatus comprises a sectional drum rotating in a bath of prefilter; the filter medium is on the interior of the drum and the filtering pressure (preferably a vac.) is applied only to sections that have been drowned some time so that first some sand may settle on the filter by gravity alone. The cake is detached by pulsating fluid pressure and removed by a conveyor in the upper part of the drum.

B. M. VENABLES.

**Feeding of wet material.** AFRICAN EXPLOSIVES & INDUSTRIES, LTD., and M. S. SALOMON (B.P. 354,022, 3.7.30. S. Afr., 16.7.29).—Apparatus suitable for wet material that does not flow freely comprises a rotating pan upon the bottom of which the material is spread in a layer of adjustable thickness by means of a rake, a small portion of the layer being discharged every revolution by the automatic opening of a trap door in the bottom.

B. M. VENABLES.

**Processing plant [for canned goods etc.].** CARRIER ENG. CO., LTD., and W. S. YARROW (B.P. 354,123, 23.10.30. Addn. to B.P. 342,722; B., 1931, 422).—A conveyor with outstanding prongs is used to effect the longitudinal transfer of the cans; the conveyor as well as the drum is within the fluid-tight casing, and the locks for entry and exit are embodied in the conveyor sprockets.

B. M. VENABLES.

**Evaporator.** O. H. WURSTER (U.S.P. 1,791,296, 3.2.31. Appl., 28.5.27).—The return pipe from the vapour separator enters the calandria at an angle of about 45° to avoid settling out of crystals, and a deflector is provided to guide the latter to the salt vessels below. Its use for the treatment of crude glycerin is specially mentioned.

B. M. VENABLES.

**Evaporating apparatus.** A. W. BAUMANN (U.S.P. 1,791,262, 3.2.31. Appl., 27.12.27).—A method of distributing steam evenly among several concentric coils each comprising a complete circle is described.

B. M. VENABLES.

**Apparatus for evaporating and concentrating liquids.** M. J. KERMER (U.S.P. 1,791,317, 3.2.31. Appl., 10.12.25).—The liquid is spread in a thin layer upon the interior of a conical bowl which is rotated at

centrifugal speed in exterior contact with steam supplied through slit-like apertures in a radial direction.

B. M. VENABLES.

**Refrigerating processes and apparatus for separation of vapours from gases in solid form.** M. FRÄNKEL (B.P. 353,014, 14.4.30. Ger., 16.4.29).—Flue gas at 5–15 atm. (gauge) pressure is cooled to <100° in one of a pair of regenerators filled with strips of metal, and previously cooled by exhaust gas, and water vapour is deposited. It is then expanded, doing external work, and the CO<sub>2</sub> is solidified. The expanded gas is filtered from CO<sub>2</sub> snow and passed through the second regenerator in the reverse direction, cooling it and vaporising the previously deposited H<sub>2</sub>O. Every 3–5 min. the operation of the regenerators is reversed. The process is applicable to other mixtures of gases.

D. K. MOORE.

**Air - filtering apparatus.** F. C. SMITH (B.P. 348,447, 2.7.30).—Within a single casing are arranged a motor-driven fan, inlet and outlet conduits, and a filtering medium comprising cellulose wadding between the conduits.

B. M. VENABLES.

**Tubular filters for gases.** E. W. STRAUS-SCHARINA (B.P. 349,331, 12.6.30. Ger., 12.6.29).—The filter bags of a group are constructed in lengths which progressively increase away from the outlet and, if desired, also from the inlet, whereby better distribution and freer exit of the gas is obtained.

B. M. VENABLES.

**Tubular filters for gases.** MASCHINENFABR. BETH A.-G. (B.P. 348,424, 7.6.30. Ger., 7.6.29. Addn. to B.P. 281,994; B., 1928, 353).—Cross-connexions and valves are provided so that one auxiliary filter at a time may be cleaned by countercurrent of air, and the air from it passed through the other auxiliary filter.

B. M. VENABLES.

**Centrifugal apparatus for separation and collection of dust from gases.** VISCO ENG. CO., LTD., and F. C. SMITH (B.P. 348,124, 8.11.29).—The apparatus comprises a volute-shaped passage, in which the dust is separated by centrifugal force, followed, if desired, by a filter. The volute first contracts in cross-section and then expands, and its outer wall is formed with louvre-like openings.

B. M. VENABLES.

**Filtering of air and other gases.** HALL & KAY, LTD., S. HALL, and P. KAY (B.P. 352,960, 14.1.30).—The filter medium consists of wood or papers coated with viscous oil or bird lime. [Stat. ref.]

D. K. MOORE.

**Revivifying spent absorbent materials.** (A, B) R. C. PALMER and (A) J. L. BURDA (U.S.P. 1,794,538—9, 3.3.31. Appl., [A] 25.11.29, [B] 2.1.30).—(A) H<sub>2</sub>O is removed from fuller's earth by heating the material with petroleum naphtha at 130–150° under pressure, then releasing the pressure so that part of the naphtha and all the H<sub>2</sub>O are vaporised. (B) Fuller's earth that has been used as a filter for rosin is washed with a mixture of petroleum naphtha and COMe<sub>2</sub>, EtOH, MeOH, EtOAc, BzOH, furfuryl alcohol, furfuraldehyde, or other org. solvent, which is at least partly miscible with the naphtha and with H<sub>2</sub>O.

A. R. POWELL.

**Absorbent for gas filters particularly for breathing purposes.** G. K. E. H. STAMPE, Assr. to

O. H. DRÄGER (U.S.P. 1,792,939, 17.2.31. Appl., 6.9.29. Ger., 29.9.28).—An absorbent filter for  $\text{NH}_3$  and  $\text{H}_2\text{S}$  comprises activated charcoal impregnated with 20% of  $\text{Pb}(\text{NO}_3)_2$  and, after drying, mixed with 40% of  $\text{Cu}_2\text{Cl}_2$ . A. R. POWELL.

Apparatus for measuring the temperature of gases [hotter than their surroundings]. R. E. WILSON (U.S.P. 1,791,020, 3.2.31. Appl., 5.5.22).—A thermometer is shielded from loss of heat by radiation to cold flue walls by a device composed of thin, polished, sheet metal, which is non-tarnishing, preferably has a low coeff. of radiation, and two concentric walls, the outer one exposed on both sides to the stream of gas and the inner forming a pocket for the bulb or thermocouple. If the thermocouple is suitable, the inner bulb may also be opened to the flow of gas, in which case a separate shield is provided to prevent radiation endways. B. M. VENABLES.

Material adapted to bearings. C. F. NOFTZGER (U.S.P. 1,791,834, 10.2.31. Appl., 14.2.27).—A closely folded schist consisting of small scales of sericite containing about 95% of earthy oxides is found at Buckeye, Ariz. Claim is made for the use of this (or similar) material for bearings in the proportion of 60–80% of the schist to 40–20% of binding materials, the latter consisting preferably of an infusible and insol. condensation product of  $\text{PhOH}$  and  $\text{CH}_2\text{O}$ . B. M. VENABLES.

Method and apparatus for determining viscosity. S. T. RODGERS (U.S.P. 1,790,948, 3.2.31. Appl., 12.4.26).—The time taken by a buoyant body to rise through a column of the liquid under test is determined. The buoyant body is constructed of, or contains, magnetic material, and is held in the lower starting position by a magnet. B. M. VENABLES.

Spectroscopes with dispersion prism. C. ZEISS (B.P. 354,659, 1.12.30. Ger., 17.12.29).—The instrument is arranged to give an image of a wave-length scale adjacent to or overlapping the dispersed image of the slit. B. M. VENABLES.

Atomisers, vaporisers, liquid fuel burners, etc. A. MAGOWAN (B.P. 354,304, 3.3.30).

Absorption refrigeration. J. C. BERTSCH (B.P. 354,868, 22.5.30).

Refrigeration apparatus. SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 354,720, 10.3.30. U.S., 14.3.29).

Reversible refrigerating apparatus employing a solid absorbent or an adsorbent. J. O. BOVING (B.P. 354,726, 12.5.30).

[Automatic control valves for] straining or filtering devices for gas. II. J. YATES, M. HOWLETT & Co., LTD., and J. DOLPHIN (B.P. 354,079, 4.9.30).

[Portable] apparatus for demonstrating the sound-absorbing properties of various materials. C. F. BURGESS LABORATORIES, INC., Assees. of C. F. BURGESS (B.P. 354,431, 24.5.30. U.S., 19.6.29).

Vacuum distillation.—See II. S from gases.—See VII. Furnaces for enamel ware.—See VIII. Light-metal pistons.—See X.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Constitution of coal and its classification. A. ECCLES and A. MCCULLOCH (Fuel, 1931, 10, 308–319; cf. B., 1931, 426).—The quantities of  $\text{HCl}$  evolved when a number of coals of varying rank were treated with  $\text{Cl}_2$  under standard experimental conditions have been determined. When these quantities, referred to pure coal, are plotted against % C in the coal a diagram is obtained similar to that on which Seyler has based his method of classifying coals, and in which % H is plotted against % C. The coal ulmins are regarded as consisting of a benzenoid nucleus around which are spaced peripheral groups of a straight-chain character; these groups react with  $\text{Cl}_2$  with the evolution of  $\text{HCl}$ , the quantity of the latter produced being a measure of the number and size of such groups. A certain correlation has been observed between the quantity of  $\text{HCl}$  evolved and the volatile matter content of the original coal, determined at 925°. The correlation between the  $\text{HCl}$  evolved and the volatile matter content of the chlorinated coal, determined at 525°, was less marked.

A. B. MANNING.

Ball structure in Indian coals. K. L. BHOLA and M. A. MAJEED (Fuel, 1931, 10, 330–331).—The mode of occurrence of ball coals in Indian seams leads to the conclusion that they are due to jointing which has been caused by a peculiar effect of pressure and friction. A. B. MANNING.

Behaviour of solid fuels during oxidation. IV. Influence of increasing the oxygen temperature on the ignition and combustion characteristics of and the rate of heat loss from solid fuels. B. MOORE (Fuel, 1931, 10, 293–296; cf. B., 1931, 659).—In studying the oxidation of fuels by the method previously described a rise in the  $\text{O}_2$  temp. was found to increase the rate of combustion and heat evolution during the pre-ignition period, but did not raise the mean or max. temp. attained by the combustion. With a higher  $\text{O}_2$  temp. the rate of combustion of cokes decreased soon after ignition and remained practically uniform for a relatively long period. Neither the rate of heat loss from the fuel during combustion nor the combustible capacity was greatly affected by raising the  $\text{O}_2$  temp. above the ignition temp.

A. B. MANNING.

Tar firing of retorts. W. E. YOUNG (Gas J., 1931, 195, 431).—The tar is led by gravity from a storage tank through a filter and fed into the producer, partly filled (75%) with bricks by an adapted "Buffalo" steam injector (nozzle reduced to  $\frac{1}{8}$  in. diam. and the Venturi tube increased to  $\frac{3}{8}$  in. diam.). 100 gals. of tar fire seven retorts for 24 hr. and save 1 ton of coke. The quantity of air admitted with the tar may be regulated by analysis of the flue gases. A. H. EDWARDS.

Porosity of lump coke. W. T. K. BRAUNHOLTZ and G. TIPLADY (Fuel, 1931, 10, 328–329).—The coke is dried and weighed in air ( $W$ ). The accessible pores are then filled with  $\text{H}_2\text{O}$  and the saturated coke is reweighed under  $\text{H}_2\text{O}$  ( $W_w$ ) and again, after drainage of the superficial  $\text{H}_2\text{O}$ , in air ( $W_a$ ). The apparent sp. gr. is given by  $W/(W_a - W_w)$ , and the apparent porosity by  $100(W_a - W)/(W_a - W_w)$ . The coke



may be saturated either by immersing it in  $H_2O$  and repeatedly evacuating and releasing the pressure in the vessel, or by boiling the coke in  $H_2O$  and allowing this to cool with the coke completely submerged. The second method is no less accurate than the first and is easier to manipulate. Either is preferable to the paraffin wax method in that the coke after drying remains uncontaminated.

A. B. MANNING.

**Influence of dry and wet cleaning on coke properties and on gas and by-product yields.** A. C. FIELDNER (U.S. Bur. Mines Rep. Invest. 3114, 1931; Fuel, 1931, 10, 320—327).—As a result of tests with two Pittsburgh coals and an Alabama coal it was found that (a) within the moisture limits of the coals tested (4.2% max.) there was no difference in the effects of dry or wet cleaning, respectively, on the coke, gas, or by-products, (b) the coke from the cleaned coal was only slightly superior in resistance to breakage to that from the uncleaned coal, (c) the yields of gas, tar, and light oil, and the heating value of the gas, were greater from the cleaned coal than from the uncleaned, even after allowing for the difference in ash and moisture content, and (d) the yields of  $(NH_4)_2SO_4$  in the laboratory  $SiO_2$ -tube tests, but not in the Fe-retort tests, were slightly higher from the washed coal. It is believed that the  $SiO_2$ -tube results are more indicative of the results to be expected in practice because of the known catalytic effect of hot Fe on the decomp. of  $NH_3$ . A high % of mineral matter in the coal appears to promote cracking of the tar.

A. B. MANNING.

**Nature and properties of certain hydrocarbons in coal gas and their effect on meter leathers.** R. S. ANDREWS (Gas J., 1931, 193, 158—159, 212—213; Gas World, 1931, 95, 130—132).—Of 1000 meters examined after prolonged use, only 42% had good leather diaphragms; 29% of the others were rotted, 12% were dry, and 12% had resin deposited on them. The oil in the leather dressing scrubs the light oil from the gas, becoming less viscous and draining from the leather. This is left quite dry, and is susceptible to the rotting effect of the moisture in the gas. The resins are formed by the polymerisation of the unsaturated hydrocarbons, accelerated by  $O_2$  and other substances. Meter troubles are accentuated by vertical-retort gas and carburetted water-gas because of their higher light oil and unsaturated hydrocarbon content.

A. KEY.

**Electrical formation of hydrocarbons from water-gas.** F. FISCHER and K. PETERS (Brennstoff-Chem., 1931, 12, 268—273).—The apparatus consisted of a discharge tube, a condenser immersed in liquid air or other cooling agent, and a Hg-vapour pump, connected in series so that the reaction gas could be circulated continuously through them. Fresh gas was supplied through a valve at a rate sufficient to maintain a const. pressure, e.g., 10 mm., in the apparatus. From the reaction products, corresponding in composition to the "electrical equilibrium" set up in the discharge tube, the condensable constituents were continuously separated. A relatively light electrical discharge was used. From mixtures of CO and  $H_2$ , using liquid air as the cooling agent,  $CO_2$ ,  $H_2O$ , and various hydrocarbons were formed, in which  $C_2H_2$  and  $C_2H_4$  predom-

inated. If the products were condensed at somewhat higher temps. they contained only higher hydrocarbons. The intermediate formation of  $CH_4$  was observed. Traces of  $CH_2O$  and MeOH were formed, and if the original gas contained  $N_2$  it was converted almost completely into  $NH_3$  and HCN.

A. B. MANNING.

**Sp.-gr. basis for determining the heating value of refinery gas.** F. W. ISLES (Chem. Met. Eng., 1931, 38, 475—476).—The calorific val. of refinery gas may be derived from the equation: B.Th.U./cu. ft. =  $(1555 \times \text{sp. gr.}) + 112$ , where sp. gr. (air = 1) is that of the  $H_2S$ - and air-free gas.

D. K. MOORE.

**Detonation, spark-plug position, and engine speed.** R. O. KING and H. MOSS (Engineering, 1931, 132, 177—180).—Experiments have been carried out on an engine fitted with 4 spark plugs so that the direction of flame travel in the cylinder head could be either to or from the hot (exhaust) side to the cooler (inlet) side or towards the centre of combustion space. The petrols used were a commercial aviation spirit (A) containing 57, 35, and 8%, respectively, of paraffins, naphthenes, and aromatics, and (B) a straight-run Borneo spirit capable of being used at a higher compression ratio than A, and blends made from these petrols. With the flame movement towards the exhaust valves the highest useful compression ratio (H.U.C.R.) was 0.23—0.3 lower at all engine speeds with petrol A alone or mixed with benzol or "ethyl fluid." Increased engine speed gives a higher H.U.C.R., the increased turbulence more than counterbalancing the effect of higher engine temp., whilst the time for org. peroxides to form is also reduced. Increase in engine temp. due to an induction temp. of  $50^\circ$  reduces the effect due to increased engine speed, especially in the case of benzol mixtures. The relationships between detonation and engine speed are of interest with supercharged engines where the induction temp. may reach  $50^\circ$ . Fuels enriched with benzol for these engines may lose the beneficial effect on detonation if the flame movement is towards the exhaust valves. Fuels improved by ethyl fluid are not adversely affected by increase in induction temp. or engine speed. Increased engine speed was found not to affect so favourably an increase in H.U.C.R. with petrol B as with petrol A. The increase in H.U.C.R. with speed is found to be greatest with paraffins; with pure naphthene (cyclohexane) it is negative.

T. A. SMITH.

**Ignition of firedamp.**—See XXII.

See also A., Sept., 1015, Explosibility of  $C_2H_2$  mixtures. Direct oxidation of hydrocarbons by air. 1017, Catalysts for production of  $H_2$  from water-gas. 1027, Determining sp. gr. of semi-solids. 1030, Natural Italian gases. 1035, Cracking of olefines. 1083, Carcinogenic potency of mineral oils.

#### PATENTS.

**Firing of gas and like retorts.** S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION CO., LTD. (B.P. 350,960, 11.3.30).—The heating gases are passed through the flues of a series of retorts in succession, the required gas temp. being maintained by boosting the gases, e.g., by burning additional fuel and adding the products of combustion to them, or by passing them through tubes in a

heating chamber, after their passage through each retort. If necessary, the gases after boosting may be reduced in vol. of flow at a predetermined stage in the series by passing them through two or more retorts in parallel. The method of heating is particularly suitable for retorts for the low-temp. carbonisation of coal, such as those described in B.P. 244,505 (B., 1926, 146).

A. B. MANNING.

**Carbonisation of fuel.** J. WEISS (B.P. 350,920, 10.1.30).—The fuel, preferably bituminous coal, is carbonised in the form of briquettes in an apparatus comprising a battery of carbonising chambers having heating flues in their walls and connexions whereby the heating gases may be passed around any desired chamber or group of chambers, while simultaneously distillation gases and/or steam may be circulated through the chambers. Each chamber can be independently charged and discharged. The flow of the heating gases is so directed that the carbonisation is carried out in stages, e.g., a preheating stage to about 350°, a tar-evolving stage, 350—500°, a gas-evolving stage, 500—600° or above, and a cooling stage, the battery of chambers at any given moment being divided into sets each containing material at one of these successive stages.

A. B. MANNING.

**Production of coke from carbonaceous fuels.** G. HILGER (B.P. 353,139, 16.5.30).—Inferior fuel is mixed with well-coking fuel dust in a perforated Fe receptacle which may have a central, vertical, perforated shaft, or spikes may be put through the mass horizontally; the whole is then compressed. When spikes are used these are now removed and the receptacle is placed in a retort. The channels made by the spikes and the central shaft carry away the gas produced. When the receptacle with the central shaft is used the charge may be compressed during carbonisation, the ram forming a hood to carry away the gas. The coke is removed in the receptacle.

D. K. MOORE.

**Decomposition of gaseous hydrocarbons to obtain hydrogen and a high-grade carbon black.** SOC. D'ÉTUDES ET RÉALISATION DITE "ÉREAL" (B.P. 353,100, 29.4.30. Belg., 30.4.29).—Gaseous hydrocarbons, e.g., coke-oven gases substantially deprived of their H<sub>2</sub>, are forced under pressure and/or suction through an incandescent fuel mass maintained at 1000—1200° in a chamber, so that the separated C is carried away with the gaseous products outside the chamber. The thickness of the fuel mass should be 1—2 m., and is so adjusted as to give an emergence temp. of the converted products from the fuel mass not above 600—700°.

H. S. GARLICK.

**Production of combustible gas.** A. C. BECKER and W. BERTELSMANN (B.P. 353,034, 19.2.30. Addn. to B.P. 335,228; B., 1931, 10).—The gas is mixed with other gases prior to the removal of the poisonous gas, so that the purified gas has the same properties as regards sp. gr., heating val., and speed of ignition as the original gas.

D. K. MOORE.

**Treatment of tars [for roads].** THERMAL INDUSTRIAL & CHEM. (T.I.C.) RES. CO., LTD., and C. O. CONDRUP (B.P. 351,023, 19.3.30).—Coal tars are distilled in two separate operations so as to produce (1) a dehydrated

and lightly "topped" tar, and (2) a heavy residue tar or pitch, and these products are blended in proportions giving a mixture complying with the British Standard Specifications and having other desirable characteristics, in particular a relatively high content of oils evaporable about 200° to ensure quick drying. A. B. MANNING.

**Operation of oil stills.** B. BROIDO, Assr. to SUPER-HEATER Co. (U.S.P. 1,794,439, 3.3.31. Appl., 31.5.23).—The oil is forced through a coil in one section in contact with the hottest furnace gas, and then through a coil in the other section countercurrent to the gas. The heating of the cold oil with the hottest gas rapidly reduces the viscosity, decreases the power required to pump the oil through the still, and reduces the deposition of C. A valve enables the gas produced in the first section to by-pass the second.

D. K. MOORE.

**Cracking of oil.** R. C. OSTERSTROM and C. R. WAGNER (B.P. 353,230, 8.7.30).—Oil is vaporised, and the vapours are dried without cracking by passage through a superheating zone comprising tubes of relatively great cross-section as compared with those used in the vaporising and cracking zones. The dried vapours are rapidly passed in a series of parallel streams of restricted cross-sectional area through a highly heated zone to raise them quickly to above 538°, and are then merged in a single stream continuously moving at a lower velocity, sufficient heat being supplied to maintain it at the cracking temp. for a desired length of time.

H. S. GARLICK.

**[Cracking] treatment of hydrocarbons.** L. KIRSCHBRAUN, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,791,566, 10.2.31. Appl., 1.9.20).—The proper degree of cracking may be effected and deposition of C in the heating tubes prevented by thermostatically controlling to  $\pm 1^\circ$  the temp. of the oil as it flows from the tubes into an enlarged vapour chamber.

D. K. MOORE.

**Cracking of hydrocarbons.** E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,789,072, 13.1.31. Appl., 4.1.28).—The vapours from a pressure-cracking still are passed to a fractionating column in the lower portion of which they are brought in contact with raw cracking stock, which is thus preheated. In the upper portion of the column the vapours are treated with lighter refluxing material, which is almost completely vaporised in the column. The unvaporised portion and preheated raw stock are returned to the pressure still.

T. A. SMITH.

**Conversion [cracking] of petroleum oils.** PETROLEUM CONVERSION CORP. (B.P. 354,496, 25.6.30. U.S., 12.11.29).—Petroleum oil vapours superheated to 480° are heated to 510—540° by mixing with hot gas, whereby cracking takes place. The deposition of C in the superheater is prevented by the high velocity of the vapours.

D. K. MOORE.

**Cracking of hydrocarbon oils.** A. E. WHITE. From TEXAS Co. (B.P. 353,276, 12.4.30).—Hydrocarbon oil is passed through a pressure-cracking still and expanded into a still operating under reduced pressure. The evolved vapours are passed to a fractionating tower, from which an overhead distillate is taken constituting the gasoline fraction. A heavy condensate is with-

drawn from the bottom of the tower and utilised as a cycle charging stock for further cracking. A middle fraction is withdrawn from an intermediate portion of the tower and may be used to dilute the heavy still residue for use as fuel oil.

H. S. GARLICK.

**Production of gasoline.** D. G. BRANDT, Assr. to DOHERTY RES. Co. (U.S.P. 1,791,113, 3.2.31. Appl., 25.2.26).—Vapours from a cracking unit are passed through a series of three condensing zones (A—C) under approx. 50 lb./sq. in. pressure, and temps. of approx. 127° in A, 70° in B, and atm. in C. The condensates from zones A and B are introduced into the upper and lower portions respectively of a mixing and stabilising tower in which is maintained a mixed condensate having a mean b.p. above that of the condensate obtained in C, which is introduced into the mid-portion of the tower. Any uncondensed vapours are passed into contact with the condensate from A.

H. S. GARLICK.

**Distillation of [hydrocarbon] oil.** J. E. BELL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,795,070, 3.3.31. Appl., 1.11.24).—Oil and the vapours formed from it during preheating by the vapours from the fractionating column are introduced into the column at such points that they are in equilibrium with the reflux and vapour, respectively, at the points of entry. Light oil, which may be the condensate from the preheater, is introduced at the top of the column to assist in controlling the reflux.

D. K. MOORE.

**Distillation [of hydrocarbon oils].** R. D. HUNNE-MAN, F. M. ROGERS, and R. E. WILSON, Assrs. to STANDARD OIL Co. (U.S.P. 1,791,209, 3.2.31. Appl., 1.4.25).—Oil is heated to 350—400°, intimately mixed with steam in the proportions of 2—10 lb. per gal. of distillate produced, and passed through an unheated conduit, where thermal equilibrium is established, at a velocity sufficient to prevent separation. It is then discharged into the vapour space of an enlarged chamber, where prompt separation of the vapours and unvaporised liquid is effected, the vapours being retained in the chamber for 1 sec. or less, an abs. pressure of 25—75 mm. being maintained throughout.

H. S. GARLICK.

**Distillation of hydrocarbon oils.** G. CASH, Assr. to STANDARD OIL Co. (U.S.P. 1,788,932, 13.1.31. Appl., 30.12.26).—Oil is heated in a coil and expanded into a drum from which vapours are removed, preferably under vac. The unvolatilised oil from the drum is pumped to the top of a fractionating column in which a further amount of oil is volatilised by means of steam. The vapours and steam from the column are added to the oil stream in the heating coil before this oil enters the expansion chamber. The method is suitable for lubricating oil stocks.

T. A. SMITH.

**Distilling hydrocarbons.** E. PIRON (U.S.P. 1,794,542, 3.3.31. Appl., 20.1.27).—Hydrocarbons are fed on to hot blocks forming a chain moved by sprocket wheels. Immediate carbonisation occurs and a further quantity is fed on to the coke and is carbonised also, thereby preventing undue cracking of the volatile products. On the under part of the travel the coke is scraped off and the blocks are heated.

D. K. MOORE.

**Use of entrainment separators in vacuum distillation [of petroleum oils].** A. C. SPENCER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,791,940, 10.2.31. Appl., 1.10.26).—The separator consists of a cylindrical vessel with a frusto-conical wire screen near the bottom. The incoming gas and liquid are carried by a central vertical pipe down on to the screen. A pipe from the bottom carries away the liquid, and one from near the top the gas.

D. K. MOORE.

**Refining of oil ["sour" gasoline].** A. P. BJERREGAARD (U.S.P. 1,791,521, 10.2.31. Appl., 26.8.25).—Motor spirit may be rendered non-reactive to the Na plumbite test and darkening and deposition of gum on exposure to light prevented by adding 0.1% by vol. of MeOH and then treating with an alkali or alkaline-earth hydroxide.

D. K. MOORE.

**Refining of hydrocarbon oils.** RICHFIELD OIL Co. OF CALIFORNIA (B.P. 353,055, 15.4.30. U.S., 8.5.29).—Crude gasoline vapours enter a scrubbing tower, and rise countercurrent to a stream of an aq. (50—80%) solution of a salt (or salts) of one or more of the metals Zn, Cd, and Hg; preferably a solution of a Zn salt containing 0.5—3% of Zn(OH)<sub>2</sub> is used. Both aq. solution and vapours are maintained at 200—260°, and vapours leaving the upper end of the tower pass to a suitable condenser. To prevent concentration, H<sub>2</sub>O or steam is continuously introduced into the tower.

H. S. GARLICK.

**Refining of hydrocarbons.** SINCLAIR REFINING Co., Asses of (A) F. A. APGAR, (B) E. C. HERTHEL (U.S.P. 1,795,067 and 1,795,124, 3.3.31. Appl., 14.9.28).—(A) Cracked hydrocarbon vapours are scrubbed from tar by the oil to be cracked, partly condensed, and passed through an adsorptive catalyst, e.g., fuller's earth, in which the unsaturated hydrocarbons are polymerised. At the same time part of the condensate is passed through the catalyst to wash out the polymerides and prolong its life. The liquid and vapour mixture is passed into a fractionating column the vapours from which are condensed to motor spirit, whilst the condensate is used to scrub the cracked vapours. (B) Vapours containing 50—75% of motor-spirit vapour are partly condensed and passed, without separation of the liquid, through the catalyst.

D. K. MOORE.

**Treating of [hydrocarbon] oils.** J. C. BLACK (B.P. 353,148, 20.5.30).—Hydrocarbon oil is heated by waste gases to distil off a portion and form a condensate, a quantity of which is then cracked in a heated coil, while a further quantity of the condensate is cooled to considerably below the cracking temp. and is introduced into the cracking coil in the region of the hottest point, in which the rate of cracking is unduly rapid, and is itself cracked thereby. The heated products from the cracking coil are directed into a reaction chamber and a further portion of the condensate is introduced into the final passes near the end of the coil in order to prevent excessive decomp. of the treated oil and deposition of C in such portions of the coil and in the transfer line leading to the reaction chamber, and also to control the temp. in the reaction chamber. Vapours from the reaction chamber pass to an evaporator wherein gasoline and a condensate are formed, the

latter being returned together with the condensate produced in the pretreatment to the cracking stage.

H. S. GARLICK.

**Treatment of petroleum.** W. F. BLEECKER (U.S.P. 1,794,668, 3.3.31. Appl., 1.5.28).—In a divided cell under 10 lb. per sq. in. pressure brine or dil.  $\text{H}_2\text{SO}_4$  is electrolysed and oil is forced continuously over the liquid in the anode chamber. The corrosive compounds, *e.g.*,  $\text{H}_2\text{S}$ , are oxidised by the nascent Cl or O.

D. K. MOORE.

**Purification of petroleum oils.** K. T. STEIK, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,791,941, 10.2.31. Appl., 30.8.28).—Petroleum oils may be more effectively purified by treatment with a liquid mixture of  $\text{SO}_2$  and  $\text{SO}_3$  at  $-10^\circ$  than with  $\text{H}_2\text{SO}_4$  or oleum.

D. K. MOORE.

**Treatment of [removal of wax from] petroleum oils.** H. B. SETZLER and M. C. McDONALD, Assrs. to NAT. REFINING Co. (U.S.P. 1,791,329, 3.2.31. Appl., 27.10.21).—Petroleum oils are cooled to  $0-5^\circ$  and agitated while continually chilling with conc.  $\text{H}_2\text{SO}_4$  to ppt. sludge and amorphous wax; the oil and semi-solid constituents are then separated.

H. S. GARLICK.

**Preparation of [lower] mercaptans [from petroleum naphtha etc.].** G. L. WENDT, Assr. to STANDARD OIL Co. (U.S.P. 1,791,179, 3.2.31. Appl., 12.11.25).—A solution of lower mercaptans is prepared by washing petroleum naphtha with aq. NaOH, adding thereto a solution of a Ca compound, *e.g.*,  $\text{Ca}(\text{SH})_2$  derived from the washing of hydrocarbon gases with a CaO suspension, and separating the ppt.

H. S. GARLICK.

**Absorption of gas and/or vapour in oil.** STANDARD OIL DEVELOPMENT Co., Assecs. of H. J. NICHOLS, JUN., and P. E. KÜHL (B.P. 353,079, 23.4.30. U.S., 2.5.29).—In a customary absorption system, automatically operating controlling means are provided, whereby the flow of gas and/or vapours to the absorption vessel regulates the flow of oil thereto, and also the flow of oil from the separating means regulates the flow of charged oil and steam thereto.

H. S. GARLICK.

**Production of high-quality lubricating oils.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 354,441, 28.5.30).—When paraffin wax is cracked at  $440-500^\circ$  in the presence of a dehydrogenating catalyst, *e.g.*, Cu, a liquid with I val. above 200 is obtained. The liquid is condensed at up to  $150^\circ$ , with or without the addition of cyclic hydrocarbons, *e.g.*,  $\text{C}_{10}\text{H}_8$  or cyclohexane, in the presence of  $\text{AlCl}_3$  to yield lubricating oils.

D. K. MOORE.

**Production of refined lubricating oils from heavy hydrocarbon oils by treatment with hydrogen at high temperatures and pressures.** STANDARD OIL DEVELOPMENT Co., Assecs. of P. L. YOUNG (B.P. 354,478, 18.6.30. U.S., 22.7.29).—Heavy oils are maintained at  $370-460^\circ$  in  $\text{H}_2$  at 200 atm. pressure in the presence or absence of a catalyst, *e.g.*, oxides of Cr and Mo alone or mixed with oxides or salts of metals of groups VI and VIII. Light oils distill over. The remainder is passed through a pressure-reduction valve, when part vaporises, to fractionating columns. The vapours are condensed and the unvaporised liquid,

with the catalyst in suspension, is returned to the pressure vessel. The process is continuous.

D. K. MOORE.

(A) **Gelatinisation or solidification of mineral oils.** (B) **Lubricants.** W. W. TRIGGS. From M. MORGAN (B.P. 351,041—2, 18.3.30).—(A) 6—9% of a salt of a soap-forming fatty acid is added to a mineral oil and the mixture is heated to  $177^\circ$  or above. When the spume has subsided the mass is stirred and allowed to cool. The product forms a lubricant varying from a jelly-like to a soap-like consistency with the proportion of metal soap used. Other solid lubricants such as graphite may be added if desired. (B) A solidified or thickened lubricant which will flow when a certain desired temp. is exceeded, or when subjected to friction, is obtained by mixing a lubricant produced as described in (A) with another solidified vegetable oil lubricant, or with a castor oil lubricant prepared as in B.P. 349,684 (B., 1931, 728). [Stat. refs.]

A. B. MANNING.

**Lubricant oil.** F. W. SULLIVAN, JUN., Assr. to STANDARD OIL Co. (U.S.P. 1,789,026, 13.1.31. Appl., 12.11.26).—The pour point of viscous lubricating oils is lowered by the addition of the Zn or Mg salt of the acids obtained by oxidising paraffin wax; 50 g. of the salt are added per litre of oil.

T. A. SMITH.

**Lubricant.** G. D. PETERS & Co., LTD. From J. A. BURTON (B.P. 353,295, 26.8.30).—A rust remover and penetrating lubricant for leaf springs etc. consists of a light petroleum lubricating oil, *e.g.*, spindle oil, dispersed graphite, one or more chlorinated hydrocarbons, *e.g.*,  $\text{CCl}_4$  or  $\text{PhCl}$ , and a hydrocarbon oil, *e.g.*, kerosene.

D. K. MOORE.

**Reclamation of [lubricating] oils.** L. D. GRISBAUM, Assr. to RAILWAY SERVICE & SUPPLY CORP. (U.S.P. 1,791,474, 3.2.31. Appl., 12.12.25).—Emulsified and contaminated journal box lubricating oil is mixed with aq. NaOH sufficiently conc. to break the emulsion, and heated under pressure. The oil is then washed by projecting  $\text{H}_2\text{O}$  on to its surface, separated from contaminations, and passed through a supply of  $\text{H}_2\text{O}$ .

H. S. GARLICK.

**[Anti-knock] fuels for internal-combustion engines.** L. ROSENSTEIN (B.P. 349,475, 18.11.29).—Organo-metallic cyanides, cyanates, thiocyanates, etc. (*e.g.*,  $\text{Pr}^{\text{B}}$ , Et, and amyl ferrocyanides and corresponding Co, Tl, Pb, Ni, Os, Mo, Cr, U, V, and W compounds), also org.-base metalocyanides (*e.g.*, aniline ferrocyanide), org. selenocyanates (*e.g.*, Ph selenocyanate), and tellurocyanates, are used as antidetonants. Solvents must usually be added to the fuel to dissolve the agent. [Stat. ref.]

C. HOLLINS.

**Operating [discharging] coke-oven plant.** A. I. DAVIES, H. J. THURLOW, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 354,317, 7.5.30).

**Machines for screening or grading of coke, coal, etc.** B. W. BROADHEAD (B.P. 354,709, 8.4.30).

**Liquid seal for gas offtakes of carbonising retorts or chambers.** GAS CHAMBERS & COKE OVENS, LTD., and N. J. BOWATER (B.P. 354,621, 21.10.30).

**Oil burners.** GILBERT & BARKER MANUFG. Co. (B.P. 355,117, 31.10.30. U.S., 18.11.29).

**Liquid-fuel burners.** G. E. W. DAWSON (B.P. 354,333, 10.2., 17.10., and 6.11.30).

**Reduction of phenols.**—See III. **Treatment of fibres.** Coated sheets.—See V. **Dyeing of acetate silk.** **Treatment of textiles.**—See VI. **Materials for roads etc.**—See IX. **Pickling of Fe and steel.** **Concentrating ores.** **Cr-plated surfaces.**—See X. **Mineral oil-sol. castor oil.**—See XII. **Coatings.** **Polish.**—See XIII. **Meat cure.**—See XIX.

### III.—ORGANIC INTERMEDIATES.

**Manufacture of nitroamines.** H. BLUM, and Soc. ALSACIENNE DE PROD. CHIM. (Sealed Note No. 2622, 20.6.25. Bull. Soc. Ind. Mulhouse, 1931, 97, 379—380). Report by M. BATTEGAY (*Ibid.*, 380—381).—Hydrolysis of nitroarylsulphonamides for the production of nitroamines (cf. G.P. 157,859, 164,130, 163,516, and 166,600) is effected with  $\text{ClSO}_3\text{H}$  instead of  $\text{H}_2\text{SO}_4$ ; *p*-toluenesulphonyl chloride is thereby formed as a by-product and is recoverable. BATTEGAY draws attention to a similar suggestion of Schuloff and others (A., 1929, 1056). A. J. HALL.

**Hydrocarbons from water-gas.**—See II. **Fatty acids.**—See XII. **EtOH.**—See XVIII.

See also A., Sept., 1017, **Hydrogenation catalyst.** 1034, **Formic acid from hydrolysis of cellulose acetate.** 1041, **Org. solvents.** 1042, **Prep. of glycine.** 1047, **Prep. of methylene sulphate.** 1048, **Prep. of  $\beta$ -orcinol.** 1058,  $\text{C}_{14}\text{H}_{10}$  derivatives. 1062, **Amino-disulphonates of anthraquinone.** 1063, **Benzanthraquinones.** 1064, **Reduction products of naphthacenequinone.** *lin*-Benzanthraquinone series. 1079, **Determination of citrates, salicylates, and benzoates.**

#### PATENTS.

**Manufacture of acetaldehyde from acetylene.** I. G. FARBENIND. A.-G. (B.P. 349,022, 15.2.30. Ger., 15.2.29).—High-percentage  $\text{C}_2\text{H}_2$  is passed at 0.8—1.5 atm. into the acid Hg solution above  $85^\circ$  in absence of org. solvent. C. HOLLINS.

**Production of *as*-dichloroethylene.** A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 349,872, 17.9.30).— $\alpha\alpha$ - or  $\alpha\alpha\beta$ -Trichloroethane is stirred with excess of milk of lime and slowly heated to  $70$ — $80^\circ$ . The yield of  $\alpha\beta$ -dichloroethylene on distillation is 90%. C. HOLLINS.

**Manufacture of vinyl chloride [from ethylene dichloride].** I. G. FARBENIND. A.-G. (B.P. 349,263, 1.5.30. Ger., 1.5.29).— $\text{MeOH}$  is used as solvent for the  $\text{NaOH}$ . C. HOLLINS.

**Production of 1 : 1 : 2-trichloroethane.** I. G. FARBENIND. A.-G. (B.P. 349,097, 21.2.30. Ger., 11.3.29).—The prep. of vinyl chloride and its conversion into  $\alpha\alpha\beta$ -trichloroethylene are effected without isolation of the former by treating the mixed gases from  $\text{C}_2\text{H}_2$  and  $\text{HCl}$  with the calc. amount of  $\text{Cl}_2$  or slightly more. Waste gases are returned to the vinyl chloride producer. C. HOLLINS.

**Preparation of ketones [from aliphatic acids].** Soc. DES BREVETS ETRANGERS LEFRANC & Cie. (B.P. 346,822, 16.1.30. Fr., 24.1.29).— $\text{CaO}$  or  $\text{Ca(OH)}_2$  is

added to  $\text{Ca}$  salts of aliphatic acids above  $\text{C}_2$ , together with sand, whereby the decomp. temp. is lowered to about  $300^\circ$ . C. HOLLINS.

**Manufacture of polymerisation products of diolefines.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,802, 28.2.30).—The polymerisation of butadiene etc. by  $\text{Na}$  etc. proceeds smoothly in presence of up to 1% of org. halogen compounds, *e.g.*, ethylene dichloride, vinyl chloride,  $\text{CH}_2\text{PhCl}$ , 1:2-dibromocyclohexane, 3-chloroquinoline, 1-chloro- or *o*-bromonaphthalene, 3-chloro-2-hydroxyindane. C. HOLLINS.

**Manufacture of substituted phenolcarboxylic [salicylic] acids.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 346,938, 12.3. and 25.11.30).—2:5-Dichlorophenol is carboxylated with  $\text{KOH}$  and  $\text{CO}_2$  at  $140$ — $150^\circ/40$  atm. to give 3:6-dichlorosalicylic acid, m.p.  $187^\circ$ ; 4-chloro-*o*-cresotic acid, m.p.  $155^\circ$ , from 4-chloro-*o*-cresol, and 4-chloro-*m*-2-cresotic acid, m.p.  $174^\circ$ , from 4-chloro-*m*-cresol, are similarly prepared. C. HOLLINS.

**Manufacture of mixed fatty aromatic ketones.** SCHIERING-KAHLBAUM A.-G. (B.P. 347,543, 4.7.30. Ger., 9.7.29).—An aromatic dicarboxylic acid or anhydride is passed with an aliphatic acid in vapour form over  $\text{ThO}_2$ ,  $\text{MnO}$ ,  $\text{FeO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaCO}_3$ , or other oxide or carbonate of alkaline-earth, rare-earth, or heavy metals. Phthalic anhydride and  $\text{AcOH}$  over  $\text{MnO}$  at  $350$ — $400^\circ$  give acetophenone and  $\text{COMe}_2$ . C. HOLLINS.

**Manufacture of water-soluble [non-resinous] condensation products derived from formaldehyde with aliphatic aldehydes and/or ketones.** I. G. FARBENIND. A.-G. (B.P. 349,556, 28.2.30. Ger., 2.3.29).—The products are heated in vac. below resinifying temp. to give colourless, transparent, viscous products useful as substitutes for glycerin, as textile finishing agents, and as dyeing or printing assistants. C. HOLLINS.

**Manufacture of ethers from alcohols.** H. D. ELKINGTON. From N. V. BATAAFSCHE PETROLEUM MAATS. (B.P. 350,010, 5.3.30. Addn. to B.P. 332,756; B., 1930, 981).—In the process of the prior patent,  $\text{FeCl}_2$ ,  $\text{CuSO}_4$ ,  $\text{SnCl}_2$ ,  $\text{MnCl}_2$ ,  $\text{AlCl}_3$ , potash and chrome alums, and  $\text{Cr}_2(\text{SO}_4)_3$  are used as catalysts at  $200$ — $300^\circ$ . The last three are most effective in the prep. of  $\text{Bu}_2\text{O}$ . C. HOLLINS.

**[Manufacture of] wetting, penetrating, foaming, and dispersing agents.** H. T. BÖHME A.-G. (B.P. 350,080, 350,432, and 350,595, [A] 20.3.30. Ger., 27.4.29, [B] 4.3.30. Ger., 6.3.29. Addn. to B.P. 318,610; B., 1931, 290, and [C] 18.3.30. Ger., 20.3.29).—(A) Org. bases, especially pyridine bases, are condensed with a sulphonated aliphatic alcohol above  $\text{C}_5$ ; *e.g.*, lauryl or stearyl alcohol is sulphonated with  $\text{ClSO}_3\text{H}$  and treated with pyridine or pyridine bases. (B) Sulphonated lauryl or myristyl alcohol is much more effective than are the other sulphonated alcohols used in the process of the parent patent. (C) Alkyl or cycloalkyl esters of polynuclear sulphaaromatic carboxylic acids are added to liquid or plastic compositions such as dye powders, preparations for foam-dyeing, fur-dyeing, cosmetics, etc. C. HOLLINS.

**Reduction of phenols.** F. UHDE (B.P. 349,575, 25.2.30. Ger., 6.3.29. Addn. to B.P. 339,317; B.,

1931, 190).—Phenols are heated with  $H_2O$  and spongy Fe in a closed vessel to about  $400^\circ/200$  atm. Pure PhOH gives a lubricant, b.p. above  $360^\circ$ , with much gaseous hydrocarbon, the formation of which is prevented by admixture of tar oils, *e.g.*, creosote oils from brown coal.

C. HOLLINS.

**Manufacture of compounds [resins etc.] from indene and phenols.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,934, 28.2.30. Addn. to B.P. 297,075; B., 1928, 845).—In the process of the prior patent, other inorg. acids or salts, *e.g.*,  $H_2SO_4$ , are used in place of halogen acids. Indene is thus condensed with resorcinol, PhOH, and 1:7-dihydroxy-naphthalene.

C. HOLLINS.

**Manufacture of derivatives of *m*-hydroxydiphenylamine.** W. W. GROVES. From I. G. FARBENIND. A.-G. (P.P. 349,677, 12.3.30).—2-Amino-*p*-cresol is condensed with an arylamine, free from  $NO_2$ ,  $SO_3H$ , and  $CO_2H$  groups, in presence of HCl. Products from aniline (b.p.  $255^\circ/20$  mm.), *p*-toluidine, and *p*-chloroaniline are described.

C. HOLLINS.

**Production of tetrazoles.** KNOLL A.-G. CHEM. FABR. (B.P. 349,682, 14.3.30. Ger., 14.3.29).—Iminoethers, especially cyclic iminoethers, are treated with hydrazoic acid or its salts in absence of catalysts. Examples are 4:5-pentamethylenetetrazole from  $[CH_2]_5 < \overset{N}{\underset{\cdot}{C}} \cdot OMe$ , and 5-phenyltetrazole, m.p.  $215^\circ$  (decomp.), from benzimino-ethyl ether.

C. HOLLINS.

**Production of esters [of sulphonated carboxylic acids. Wetting agents].** II. T. BÖHME A.-G. (B.P. 350,425, 5.2.30. Ger., 6.2.29).—Aralkyl or aryl esters of aliphatic carboxylic acids above  $C_8$ , or alkyl, aralkyl, or aryl esters of non-hydroxylated cyclic carboxylic acids, are sulphonated in presence of a lower fatty acid or anhydride or chloride, and/or with oleum,  $ClSO_3H$ , or  $BrSO_3H$ . In the process of B.P. 315,832 (B., 1930, 1058) oleum etc. is used in the case of alkyl esters of aliphatic carboxylic acids above  $C_8$ . Acetylsulphuric acid may also be employed. Examples of starting materials are:  $Bu^a$  or  $Bu^b$  and methylcyclohexyl ricinoleates, benzyl oleate,  $Pr^b$   $\beta$ -naphthoate, and  $Bu^a$  hydroxystearate. Sulphonation may precede esterification.

C. HOLLINS.

**Manufacture of 3'-amino-6'-halogenbenzyl-*o*-benzoic [6-halogeno-3-aminodiphenylmethane-2', carboxylic] acids and substitution products and derivatives thereof.** NEWPORT CO. (B.P. 347,171, 16.12.29. U.S., 2.1.29. Addn. to B.P. 314,804; B., 1931, 290).—A 3-amino- or 3-acylaminodiphenylmethane-2'-carboxylic acid, carrying, if desired, Me, MeO, EtO, halogen,  $CO_2H$ ,  $SO_3H$ , or other substituent in position 4, is chlorinated or brominated in the 6-position by halogenation, preferably in a solvent (AcOH). The following diphenylmethane-2'-carboxylic acids are described: 6-bromo-3-amino-4-methoxy- (m.p.  $189-190^\circ$ ; Ac derivative, m.p.  $228-229^\circ$ ), 4:6-dichloro-3-amino- (m.p.  $164-165^\circ$ ; Ac derivative, m.p.  $246-247^\circ$ ), 6-chloro-3-amino-4-methoxy- (m.p.  $190^\circ$ ; Ac derivative, m.p.  $226^\circ$ ; phthaloyl derivative), 6-chloro-3-acetamido-4-ethoxy- (m.p.  $219-220^\circ$ ), 6-bromo-3-amino-4-methyl- (m.p.  $193-194^\circ$ ; Ac derivative, m.p.  $246^\circ$ ), 6-chloro-3-

amino-4-methyl- (m.p.  $196-197^\circ$ ; Ac derivative, m.p.  $256^\circ$ ; *p*-toluenesulphonyl and Bz derivatives).

C. HOLLINS.

**Manufacture of 1-amino-4-halogen-[4-halogeno-1-amino]-9-anthrone and substitution products and derivatives thereof.** NEWPORT CO. (B.P. 347,195, 16.12.29. U.S., 2.1.29. Addn. to B.P. 340,519; B., 1931, 336).—The acylated products of B.P. 347,171 (preceding) are cyclised to anthrones, and, if desired, oxidised to anthraquinones. 4-Chloro-1-acetamido-2-methoxy-9-anthrone (m.p.  $170^\circ$ ) and -anthraquinone (m.p.  $242-243^\circ$ ), the 4-bromo-compounds (m.p.  $153-154^\circ$  and  $205-206^\circ$ , respectively), 4-bromo-1-acetamido-2-methyl-9-anthrone [m.p.  $171^\circ$  (decomp.)] and -anthraquinone (m.p.  $212-123^\circ$ ), the 4-chloro-compounds (m.p.  $175^\circ$  and  $203-204^\circ$ , respectively), and 2:4-dichloro-1-acetamido-9-anthrone (m.p.  $208^\circ$ ), are described.

C. HOLLINS.

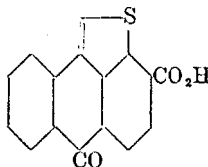
**Manufacture of *o*-aminocarboxylic esters of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 349,795, 13.6.30. Ger., 14.6.29. Addn. to B.P. 267,164 and 314,028; B., 1928, 361; 1930, 809).—Anthraquinone-1:2-isooxazoles react with aliphatic or aromatic alcohols in absence of alkali to give 1-aminoanthraquinone-2-carboxylic esters. The Me (m.p.  $227-228^\circ$ ), and  $CH_2Ph$  (m.p.  $182^\circ$ ) esters, and amyl 5:8-dichloro-1-aminoanthraquinone-2-carboxylate, m.p.  $124^\circ$  (from 3:6-dichloro-2-*p*-toluoylbenzoic acid, m.p.  $162^\circ$ , cyclised to 5:8-dichloro-2-methylantraquinone, m.p.  $244^\circ$ , nitrated to the 1- $NO_2$ -compound, m.p.  $242^\circ$ , and thence converted by oleum into the isooxazole), are described.

C. HOLLINS.

**Manufacture of 1-hydroxy- and 1-alkoxy-anthraquinone-3-carboxylic acids and derivatives thereof.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,635, 4.2.30).—2-Alkoxybenzophenone-2':4-dicarboxylic acids are cyclised in  $H_2SO_4$  or oleum, preferably in presence of  $H_3BO_3$ ; the alkoxy group is frequently lost during cyclisation. The prep. of 1-hydroxy- (m.p.  $282-284^\circ$ ), 4-chloro-1-hydroxy- (m.p.  $198-199^\circ$ ), 1:4-dihydroxy- (m.p.  $249-250^\circ$ ; from the 5-chloro- or 5-hydroxy-dicarboxylic acid), and 4-hydroxy-1-methoxy- (m.p.  $215-216^\circ$ ) -anthraquinone-3-carboxylic acids is described.

C. HOLLINS.

**Manufacture of conversion products of 1:2-anthraquinonethioglycollic-carboxylic acid [2-carboxyanthraquinone-1-thioglycollic acid].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,080, 20.2.30).—2-Carboxyanthraquinone-1-thioglycollic acid, heated with  $Ac_2O$  etc., gives a mixture of thienanthrone-3-carboxylic acid (annexed formula), m.p. above  $300^\circ$ , and the *O*-acetate, m.p.  $248^\circ$ , of 6:7-phthaloylthioindoxyl, separable by means of NaOH or a solvent such as PhMe or PhCl. Shorter reaction produces the intermediate thienanthrone-1:3-dicarboxylic acid, which loses the 1- $CO_2H$  group when heated in  $C_6H_5Cl_3$ . The *O*-acetate is converted by alcoholic KOH into 6:7-phthaloylthioindoxyl. The formation of the thioindoxyl or its acetate is favoured by the addition of alkali, metal oxide, or a weak acid



salt, or by using an alkali salt of the thioglycollic acid. In the absence of  $\text{Ac}_2\text{O}$  the aq. alkali salt yields mainly the thienanthrone-3-carboxylic acid, or, in higher concentrations, the 1:3-dicarboxylic acid (dichloride, m.p. 206–212°). C. HOLLINS.

**Lower mercaptans.**—See II. **Catalysts for org. reactions.**—See VII.

#### IV.—DYESTUFFS.

**[Manufacture of] yellow and reddish-brown direct dyes.** SOC. CHEM. IND. IN BASLE (Sealed Note No. 1051, 7.9.98. Bull. Soc. Ind. Mulhouse, 1931, 97, 375–376). Report by M. BATTEGAY (*Ibid.*, 377–378).—Direct dyes fast to light,  $\text{Cl}_2$ , air, and acids are obtained by condensation in the presence of NaOH of certain monoazo compounds resulting from coupling diazotised sulphanilic acid,  $\alpha\text{-C}_{10}\text{H}_7\text{-NH}_2$ , or  $m\text{-C}_6\text{H}_4(\text{NH}_2)_2$  with *p*-nitrotoluenesulphonic acid or its oxidation products such as dinitrobenzyl- and dinitrostilbene-disulphonic acids. Suitable monoazo compounds may also be obtained by coupling diazotised metanilic, toluidine-sulphonic,  $\beta$ -naphthylaminesulphonic, and aminobenzoic acids with  $\alpha\text{-C}_{10}\text{H}_7\text{-NH}_2$ . Condensation of *m*-nitroaniline with dinitrostilbenedisulphonic acid yields a dye fast to  $\text{Cl}_2$  and having the shade of Chloramine Yellow. BATTEGAY reports that the manufacture of the last-named dye is described in G.P. 101,760, and that dyes obtained by condensation of dinitrostilbene- and dinitrobenzyl-disulphonic acids with monoazo dyes are described in G.P. 204,212. A. J. HALL.

See also A., Sept., 1041, **Solvent action in dyeing.** 1046, **Diphenyl derivatives.** 1052,  **$\text{C}_{10}\text{H}_7$ -green.** 1056, **4:4'-Dihydroxydiphenyl-3:3'-dialdehyde and its derivatives.** 1066, **Colouring matter of paprika.** **Vegetable dyes.** 1075, **Condensed thiazine- and thiazole-thionaphthen derivatives.** 1092, **Colouring matter of *Penicillio*psis.** **Citromycetin and citrinin.**

#### PATENTS.

**Manufacture of metalliferous dyes.** SOC. CHEM. IND. IN BASLE (B.P. 349,304, 26.5.30. Switz., 25.5.29).—An azo dye of the type *o*-aminophenol or *o*-aminobenzoic acid  $\rightarrow$  *N*-substituted J-acid is treated in alkali with a metal compound and a nitrosating agent in either order. Examples are: 5-nitro-*o*-aminophenol  $\rightarrow$  phenyl-J-acid, chromed and nitrosated (steel-blue on cotton, viscose, wool, or silk), or nitrosated (blue-violet by after-coppering); 4-nitro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  di-J-acid, chromed and nitrosated (grey-blue); *o*-aminophenol-4-sulphonamide  $\rightarrow$  di-J-acid, coppered and nitrosated (red-violet). C. HOLLINS.

**Manufacture of wool dyes [of the anthraquinone series].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,126, 19.2.30).—4-Halogeno-1-aminoanthraquinone-2-sulphonic acids are condensed with aromatic diamine-monosulphonic acids, having at least 1 primary  $\text{NH}_2$  group, in aq. solution in presence of  $\text{Na}_2\text{CO}_3$  etc. and a Cu catalyst. C. HOLLINS.

**Manufacture of [acid] dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 349,325, 6.6.30. Ger., 7.6.29).—Amino or alkylamino-dianthraquinonyl

amines are sulphonated with oleum in presence of  $\text{H}_3\text{BO}_3$  and a Hg catalyst, *e.g.*, at 120–150°, to give acid wool dyes more sol. than those of B.P. 201,575 (B., 1924, 861). C. HOLLINS.

**Manufacture of fast vat dyes [of the benz- and thien-anthrone series].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,036, 19.2. and 30.4.30).—Cyclic di- or poly-ketones having at least 1 free  $\text{NH}_2$  group are condensed with a benz- or thien-anthrone-carboxyl chloride, preferably in a diluent ( $\text{PhCl}$ ,  $\text{C}_6\text{H}_4\text{Cl}_2$ ,  $\text{PhNO}_2$ ) in absence of acid-binding agents. Examples are: thienanthrone-3-carboxyl derivatives of 1-aminoanthraquinone (yellow vat dye), 1-amino-4- and -5-benzamidoanthraquinone (orange, golden-yellow), aminoanthanthrone (red-brown), aminopyranthrone (brown), aminodibenzanthrone (green, not becoming black with  $\text{Cl}_2$ ), 5-amino-1:1'-dianthraquinonylamine (green), aminodibenzpyrenequinone (yellow-brown); thienanthrone-4-carboxylic derivative of 1-aminoanthraquinone (yellow); thienanthrone-5-carboxylic derivative of the carbazole from 5-amino-4'-benzamido-1:1'-dianthraquinonylamine (brown); thienanthrone-1:3-dicarboxylic derivatives of 1-amino-5-benzamidoanthraquinone; benzanthrone-4-carboxylic derivatives of 1-amino-5-benzamidoanthraquinone (yellow), 1:5-diaminoanthraquinone (green-yellow), 4:4'-diamino-1:1'-dianthraquinonylamine, carbazolised (olive), aminodibenzpyrenequinone (brown-yellow), and aminopyranthrone (yellow-brown); benzanthrone-3-carboxylic derivative of 1-amino-5-benzamidoanthraquinone (yellow). Thienanthrone-4-carboxylic acid (chloride, m.p. 256°) is obtained from 1-aminoanthraquinone-3-carboxylic acid by way of the 1-thioglycollic acid (*cf.* B.P. 349,080; B., 1931, 918). C. HOLLINS.

**[Manufacture of vat] dyes [of the dibenzanthrone series].** R. S. BARNES, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 350,030, 1.3.30).—Dibenzanthrone or its halogenated derivatives is treated with  $\text{K}_2\text{Cr}_2\text{O}_7$  or other strong oxidant in presence of  $\text{HNO}_3$  to give grey to black vat dyes. C. HOLLINS.

**Manufacture of high-molecular pyridino-compounds [vat dyes].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 349,059, 15.11.29).—Polycyclic amines are converted by Skraup reaction into quinolines, which may, if desired, be further condensed, halogenated, or nitrated. Examples are quinolines from: 9-amino-benzanthrone (isomeric products, m.p. 250° and above 300°, respectively, green-yellow on acetate silk; violet-blue vat dye by alkaline fusion); 11-aminobenzanthrone (alkaline fusion, navy-blue vat dye); amino-*allo*-*ms*-naphthadanthrone (m.p. 350–352°, red; hexachlorinated, red-orange; dibrominated, orange; tetrabrominated, blue-red; oxidised to the anthanthrone, orange); diamino-compound (blue-red); aminopyranthrone (m.p. above 300°, blue-red; dibrominated, Bordeaux; nitrated, blue-grey changing to brown with  $\text{Cl}_2$ ); diaminoanthanthrone (yellow, golden-orange when purified from  $\text{H}_2\text{SO}_4$ ; chlorinated, brilliant red-orange); amino-1:2:6:7-dibenzpyrene-3:8-quinone (orange-yellow; brominated, orange-yellow); diamino-compound (brown-yellow); 2:3-dichloro-7-amino-8:9-phthaloylacridone (weak violet); aminoanthanthrone



(golden-orange; dibrominated, orange-yellow; nitrated); bromoamino-compound (orange). C. HOLLINS.

**Manufacture of [indigoid] vat dyes.** I. G. FARBENIND. A.-G. (B.P. 349,361, 16.7.30. Ger., 16.7.29).—A 1-hydroxy-4-alkoxy-9-anthrone (cf. B.P. 340,639; B., 1931, 336), which may contain substituents in positions 5—8, is condensed with isatin  $\alpha$ -anil (etc). Examples are products from 1-hydroxy-4-methoxy-9-anthrone, m.p. 156—157° (green-blue), the 4-ethoxy-compound, m.p. 145—146° (green-blue), and 5:8-dichloro-1-hydroxy-4-methoxy-9-anthrone (green). C. HOLLINS.

**Manufacture of black [tetrakis]azo dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,600, 24.2.30).—A 1:8-aminonaphthol-4-mono- or -4:6- or -3:6-di-sulphonic acid is coupled with 2 mols. of a diazotised nitro- or acylamino-arylamine carrying substituents suitable for coppering (OH, CO<sub>2</sub>H, or OR *ortho* to either N), the NO<sub>2</sub> is reduced or the NHAc hydrolysed, and the tetrazotised product is coupled with 2 mols. of resorcinol, *m*-phenylenediamine, *m*-aminophenol, or derivatives of these. The dyes are coppered on the fibre. Examples are: 5-nitro-*o*-anisidine  $\rightarrow$  (acid) 1:8:4:6-aminonaphtholdisulphonic acid (alk.)  $\leftarrow$  5-nitro-*o*-anisidine, reduced,  $\rightarrow$  2 mols. of *m*-aminophenol or *m*-phenylenediamine; 6-nitroresidine similarly, with resorcinol as end-component; *p*-nitroaniline  $\rightarrow$  (acid) 1:8:4:6-acid (alk.)  $\leftarrow$  5-nitro-*o*-anisidine, reduced  $\rightarrow$  2 mols. of *m*-phenylenediamine; *p*-aminoacetanilide  $\rightarrow$  (acid) H-acid (alk.)  $\leftarrow$  6-nitroresidine, hydrolysed, reduced,  $\rightarrow$  2 mols. of resorcinol; 5-aminoacet-*o*-anisidine  $\rightarrow$  (acid) 1:8:4:6-acid (alk.)  $\leftarrow$  *p*-aminoacetanilide or 4-chloro-5-aminoacet-*o*-anisidine, hydrolysed,  $\rightarrow$  2 mols. of *m*-phenylenediamine; 5-aminoacet-*o*-anisidine  $\rightarrow$  (acid) S-acid (alk.)  $\leftarrow$  5-aminoacet-*o*-anisidine, hydrolysed,  $\rightarrow$  2 mols. of resorcinol; *p*-nitroaniline  $\rightarrow$  (neutral) 1:8:4:6-acid (alk.)  $\leftarrow$  5-nitro-*o*-aminophenol or 5-nitroanthranilic acid, reduced,  $\rightarrow$  2 mols. of *m*-phenylenediamine.

C. HOLLINS.

Wetting etc. agents.—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Swelling of cellulose and its affinity relations with aqueous solutions. IV. Preferential absorption of barium hydroxide. V. Absorption of copper from dilute cuprammonium hydroxide, both characteristic properties of cellulose and an indication of previous mercerisation or other swelling treatment. Catalytic method for microdetermination of copper. S. M. NEALE (J. Text. Inst., 1931, 22, T 349—356, T 357—364; cf. B., 1931, 835).—IV. The absorption of Ba(OH)<sub>2</sub> affords similar information about the state of activation of cellulose to that given by the absorption of NaOH (*loc. cit.*). The "baryta absorption ratio" relative to the amount of Ba(OH)<sub>2</sub> absorbed by plain scoured cotton from a solution of the same end concentration at the same temp. is almost indistinguishable from the NaOH absorption ratio for samples of cotton of widely different pretreatment, and, like the latter, is independent of the reagent concentration. These facts increase the significance of the alkali absorption ratios as measures of the relative amount

of accessible salt-forming OH rendered available when cellulose is swollen, washed, and dried. Whilst the ratio may be used as a check on the regularity of a standard mercerised product, it cannot be relied on as a direct measure of any quality of textile value.

V. [With T. BROWSETT and F. D. FARROW.] The absorption of Cu from dil. cuprammonium hydroxide (too dil. to swell cellulose appreciably), either in the presence or absence of NaOH, is dependent on the pretreatment of the cellulose in much the same way as are the NaOH and Ba(OH)<sub>2</sub> absorptions, but though the approx. identity of the Cu, NaOH, and Ba(OH)<sub>2</sub> absorption ratios is a noteworthy fact, it does not form a basis for any satisfactory speculations. The method used for determining Cu is a development of that employed by Baines (B., 1929, 1050) and depends on the fact that the reaction between Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> is accelerated by the presence of traces of Cu. B. P. RIDGE.

**Extraction apparatus for cellulose.**—See I. **Saccharification of cellulose.**—See XVII.

See also A., Sept., 1041, **Acetylation of cellulose**. 1081, **S in South African wools**.

## PATENTS.

**Retting of fibres.** ROHM & HAAS Co., Assecs. of C. S. HOLLANDER and P. H. D. PLAINE (B.P. 353,868, 28.3.30. U.S., 17.5.29).—The raw material, *e.g.*, flax, flax straw, jute, etc., is boiled for 1—4 hr. with 10—25 times its wt. of an alkaline liquor containing, *e.g.*, 5—10% (on the wt. of fibre) each of NaOH, soap, and a buffer (Na<sub>2</sub>SO<sub>4</sub>), and is then washed and treated for 24—48 hr. at, *e.g.*, 30° in an enzyme bath prepared from fungi of the *Aspergillus*, *Penicillium*, *Mucor*, or *Rhizopus* groups or bacteria of the *subtilis-mesentericus*, *carotovorus*, or like groups. The best results are obtained when the bath is maintained at pH 7, and an activator is present, *e.g.*, 1% of Na<sub>2</sub>SO<sub>4</sub> on the wt. of fibre. PhMe may be added to inhibit bacterial growth. D. J. NORMAN.

**Manufacture of material from difficultly-tractable animal fibres.** H. M. PIKE (B.P. 353,429, 15.4.30).—The tendency for the fibres to become displaced and form free ends in yarns and fabrics made from fibres such as horsehair, mohair, goat hair, and the lower grades of wool, is obviated by coating or impregnating such materials with a cellulose composition which contains preferably a cellulose ester (*e.g.*, cellulose acetate) and a plasticiser. A suitable composition consists of EtOH 20 pts., C<sub>6</sub>H<sub>6</sub> 24 pts., COMe<sub>2</sub> 45 pts., cellulose acetate 5 pts., glyceryl triacetate, 5.2 pts., and Ph<sub>3</sub>PO<sub>4</sub> 0.8 pt. A. J. HALL.

**Treatment of vegetable fibres.** L. MELLERSH-JACKSON. From TWITCHELL PROCESS Co. (B.P. 354,303, 3.3.30).—The addition of 0.1—2.0% of mineral oil sulphonates ("mahogany" sulphonates) to the alkali solution used in boiling out textile fibres considerably reduces the boiling time so that satisfactory results may be obtained by a continuous process working at atm. pressure. D. J. NORMAN.

**Drying of bodies, particularly breadths or strips of fibrous material impregnated or coated with a solution of synthetic resin in spirit [and recovery**

of the volatile solvent therefrom]. R., M., and E. SCHRÖDER, S. R., and S. LEVIS (JAROSLAW'S ERSTE GLIMMERWAREN-FABR. IN BERLIN) (B.P. 354,099, 17.9.30. Ger., 19.9.29).—The material passes successively through 2 or more drying chambers, the bulk of the solvent being removed and recovered in the first chamber. The drying medium, *e.g.*, air (optionally heated), is repeatedly circulated through the first chamber by means of an external pump until a relatively high concentration of solvent vapour is present. At this point some of the solvent-laden air is continuously drawn from the system for solvent recovery, and replaced by an equal quantity of fresh air or air which has been circulating in the second dryer. D. J. NORMAN.

**Producing at the cooking of cellulose and especially of sulphite-cellulose a waste liquor containing a high percentage of organic matter.** G. HAGLUND, ASSR. to PATENTAKTIEB. GRÖNDAL-RAMÉN (U.S.P. 1,791,476, 3.2.31. Appl., 1.3.30. Swed., 10.4.29).—The chips are first impregnated under pressure with fresh liquor and then cooked in a mixture of fresh and waste liquors. D. J. NORMAN.

**Production of artificial filaments, threads, ribbons, etc. by dry-spinning processes.** H. DREYFUS (B.P. 353,956, 6.5.30. Addn. to B.P. 312,203; B., 1929, 594).—In the manufacture of filaments of low denier and high extensibility by the process of the prior patent, improved results are obtained if the ratio of non-solvent liquid to high-boiling solvent is between 3:1 and 1:3 (preferably between 2:1 and 1:2) by wt. The total quantity of non-solvent liquid in the solvent mixture should preferably be < 12%, *e.g.*, about 6–8% by wt. A suitable solvent mixture contains, by wt., 80–84 pts. of COMe<sub>2</sub>, 9–7 pts. of H<sub>2</sub>O, and 11–9 pts. of diacetone alcohol, Et phthalate, or the like. D. J. NORMAN.

**Luminous [artificial silk] filament.** H. A. GARDNER (U.S.P. 1,791,199, 3.2.31. Appl., 29.12.27).—About 5% of a base material, *e.g.*, TiO<sub>2</sub>, which has been treated with a small quantity of a radioactive or phosphorescent substance, *e.g.*, RaBr<sub>2</sub>, CaS, BaS, or chlorophene, is added to the spinning solution. D. J. NORMAN.

**Manufacture of cellulose derivatives.** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 353,930, 23.4.30).—Cellulose derivatives of the type described in B.P. 264,261 and 268,505 (B., 1927, 552, 473), which whilst retaining their fibrous structure show a pronounced affinity for acid azo dyes, are obtained by treating the xanthated and mildly oxidised fibre with an aliphatic diamine, *e.g.*, piperazine, ethylenediamine, diethylethylenediamine, etc. Alternatively, the xanthated fibre may be treated with reagents which induce linking of the cellulose xanthate mols. either directly or through an intermediate group, *e.g.*, S<sub>2</sub>Cl<sub>2</sub>, COCl<sub>2</sub>, CSCl<sub>2</sub>, AcCl, chlorocarbonic esters, or the like, the cellulose xanthate derivative thus obtained being in turn treated with an aliphatic diamine. D. J. NORMAN.

**Solution of cellulose derivatives.** J. G. DAVIDSON, ASSR. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,791,301, 3.2.31. Appl., 19.5.26. Renewed 12.8.29).—Compounds of the type C<sub>n</sub>H<sub>2n</sub>(R)·OR', where *n* is > 1,

R' is an alkyl or aryl group, and R an acid group, *e.g.*, glycol monoethyl ether acetate, OEt·CH<sub>2</sub>·CH<sub>2</sub>·OAc, b.p. 153°, are suitable solvents for use in the manufacture of cellulose ester compositions. D. J. NORMAN.

**Manufacture of cellulose esters.** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 353,978, 20.5.30).—Cellulosic material, *e.g.*, cotton, regenerated cellulose, or partly esterified or etherified cellulose, is esterified without loss of fibrous structure or degradation of the cellulose mol. by impregnating the material with KOAc or other K salt of a weak acid, drying, and heating with an org. acid anhydride, optionally with addition of esterification accelerators, *e.g.*, a *tert.* organic base, or inert diluents, but without addition of acid catalysts. Thus when viscose silk is immersed for 1 hr. in 60% aq. KOAc, centrifuged, dried at about 70°, and boiled for about 1 hr. with a 4:1 (by vol.) C<sub>6</sub>H<sub>6</sub>·Ac<sub>2</sub>O mixture the products contain 1–2 Ac groups per mol. of C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>, show the dyeing properties of acetate silk, are insol. in org. solvents, have a high wet strength, and may be obtained either as matte woolly fibres or as highly lustrous smooth fibres. Pattern effects are obtained on fabrics by printing with KOAc-gum mixtures and heating the dried material at 100° for about ½ hr. with a 20% PhMe solution of Ac<sub>2</sub>O. D. J. NORMAN.

**Manufacture of [cellulose ester or ether] artificial threads, filaments, and the like.** COURTAULDS, LTD., and C. DIAMOND (B.P. 353,597, 26.5.30).—In spinning by the downward dry-spinning process, the whole of the evaporative atm. is introduced into the spinning cell in the neighbourhood and on one side of the extrusion nozzle and is then withdrawn from the cell on the opposite side of the nozzle wholly at the level of the nozzle, or partly at the level and partly below the level of the nozzle; the whole of the evaporative atm. is thus drawn across the extrusive nozzle. A. J. HALL.

**Fibre-liberating process [for woods of high resin content].** G. A. RICHTER and M. O. SCHUR, ASSRS. to BROWN Co. (U.S.P. 1,790,838, 3.2.31. Appl., 27.6.27).—A soft pulp of good quality is obtained by the alkaline digestion of, *e.g.*, jack pine if 0.3–0.4% of soap is added to the digestion liquor to disperse the difficultly saponifiable resins. Other dispersing agents, *e.g.*, Na<sub>3</sub>PO<sub>4</sub>, Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, tale, colloidal clay, may also be added. D. J. NORMAN.

**Washing of pulp.** O. MANTIUS (U.S.P. 1,790,714, 3.2.31. Appl., 6.4.28).—The pulp is pressed to remove as much liquor as possible and is then intimately mixed in a shredder with a small quantity of washing liquor which in turn is removed by pressure. This cycle of operations is repeated any desired number of times. Suitable apparatus for continuous countercurrent washing along these lines is described. D. J. NORMAN.

**Manufacture of pulp web of high absorptivity.** G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,790,839, 3.2.31. Appl., 1.6.28).—Up to 12% of mechanically gelatinised cellulose is mixed with unbeaten or but lightly beaten long-fibred pulp, preferably of high α-cellulose content, and the resulting sheet is dried without application of pressure. The addition of gelatinised cellulose gives increased bursting, breaking, and tearing

strength without substantially reducing the absorbency of the product. D. J. NORMAN.

**Felted cellulose fibre product.** G. L. SCHWARTZ, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,791,248, 3.2.31. Appl., 22.9.28).—A preformed fibrous sheet, *e.g.*, made from kraft pulp and preferably unsized, is run through a bath of 8–15% NaOH sol. at  $< 5^\circ$ , *e.g.*, a 10–11% NaOH solution at  $-5^\circ$  (time of treatment 1–30 sec.), squeezed, and rapidly heated by blowing steam at more than 10 lb. pressure on to the sheet while held under pressure against a rotating drum by a wire cloth. The sheet is then preferably passed through a 15–18% NaOH solution at 80–104° (time of treatment 2–20 sec.), after which it is washed and dried in the usual way. The resulting paper is flexible and shows a considerably increased tearing and tensile strength, whilst the tensile strength in the wet state is raised from 2% to about 20% of the dry strength.

D. J. NORMAN.

**Manufacture of pulp board.** H. L. BECHER (B.P. 354,001, 13.6.30. U.S., 13.7.29).—Beaten pulp, *e.g.*, ground wood, chemical pulp, straw, etc., optionally in admixture with binders, *e.g.*, pitch, asphalt, or gilsonite, is run into a mould, pressed, and heated, first without substantial pressure during the removal of interstitial  $H_2O$  (*viz.*, to an air-dry condition), and then under heavy pressure during the removal of colloiddally retained moisture (bone-dry). The pressure is then released without cooling the platens.

D. J. NORMAN.

**Manufacture of plastic articles [paste-board packing material] from artificial wood pulp.** K. KÜRSCHNER (B.P. 348,791, 24.3.30. Ger., 4.2.30. Addn. to B.P. 347,807; B., 1931, 671).—Long-fibre cellulosic material, *e.g.*, wood pulp, after or during a preliminary or final moulding process forming part of treatment ordinarily used in the manufacture of paper, is impregnated with a dissolved compound of cellulose which is subsequently transformed into hydrocellulose either immediately after the impregnation or after the completion of the moulding process.

F. R. ENNOS.

**Coated material in sheets.** DURASTIC BITUMINOUS PRODUCTS, LTD., and W. B. THOMPSON (B.P. 353,910, 29.4.30).—Fibrous material (preferably 40% hair and 60% jute) is impregnated at 93° with a mixture of pitch and tar (10% of tar) and coated on one side with a substance which will adhere to metal or other surfaces on application of heat, *e.g.*, bitumen, pitch, resins, etc.

D. J. NORMAN.

**Regeneration of sulphides.**—See VII. **Plastic materials.** **Fibrous compositions.**—See XIII. **Leather for spinning-rollers.**—See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Use of glucose in printing dyes by reduction methods.** A. SCHEUNERT and N. VOSNESSENSKI (Sealed Note No. 1993, 22.4.10. Bull. Soc. Ind. Mulhouse, 1931, 97, 378). Report by M. BATTEGAY (*Ibid.*, 379).—The method described previously in Note 1990 (B., 1920, 745 A) for producing vat discharges on an indigo ground may be used for obtaining vat effects on a white ground. A satisfactory printing paste contains glucose 300 pts.,

Indanthrene Blue paste 250 pts., and thickening 450 pts. BATTEGAY reports favourably on the process.

A. J. HALL.

**Simultaneous weighting with production of clear shades on a deep ground colour of wool fabrics.** AMOS & CIE., and E. KOECHLIN (Sealed Note No. 2773, 12.12.29. Bull. Soc. Ind. Mulhouse, 1931, 97, 372–373). Report by J. NIEDERHAUSER and E. DUHEM (*Ibid.*, 373–374).—The loss of wt. experienced in milling (felting) wool materials is counterbalanced (sometimes an increase of wt. is obtained) by treatment for  $\frac{1}{2}$  hr. at 90° in a bath containing clipped rabbit fur and 0.5%  $H_2SO_4$ ; the wool simultaneously acquires from the fur a clear grey shade not otherwise obtainable except by previous scouring. NIEDERHAUSER and DUHEM refer to previous similar processes in which wool is used, but in an alkaline bath and for the purpose of weighting only.

A. J. HALL.

**Washing of piece goods [stained with river water].** O. METZGER and L. M. GRANDERYE (Sealed Note No. 2225, 24.2.13. Bull. Soc. Ind. Mulhouse, 1931, 97, 371). Report by M. BADER (*Ibid.*, 372).—Fabrics stained pink, salmon, and cream by washing with impure river water could not be satisfactorily "cleared" by chlorination while passing over Cu cylinders, but they responded to treatment with a solution containing 10–20 g. of  $NaHSO_3$  per litre; this solution had no deleterious action on the cylinders. BADER confirms the novelty of the process.

A. J. HALL.

**Swelling of cellulose.**—See V.

See also A., Sept., 1041, **Solvent action in dyeing etc.**

## PATENTS.

**Bleaching [with peroxides] of textile materials of vegetable origin.** H. O. KAUFFMANN, and ELEKTROCHEM. WERKE MÜNCHEN A.-G. (B.P. 352,690, 18.6.30).—Vegetable fibres are bleached by subjection to a pressure boil with an alkali followed by an open boil with  $H_2O_2$ , the latter treatment being preferably effected in an Fe or other vessel coated as described in B.P. 350,726 (B., 1931, 801).

A. J. HALL.

**Dyeing of acetate silk.** L. MELLERSH-JACKSON. From TWITCHELL PROCESS Co. (B.P. 354,326, 3.2.30).—The mineral oil ("mahogany") sulphonates obtained in the refining of mineral oils with oleum or  $SO_3$  (*cf.* B.P. 127,159; B., 1919, 493 A) are used after purification as dispersing agents for cellulose acetate dyes. Both types of "mahogany" sulphonates may be used, *viz.*, those recovered from the acid sludge, and, more particularly, from the oil layer. Dispersion of these sulphonates in  $H_2O$  is facilitated by the addition of small quantities of low-viscosity mineral oils, aromatic, hydrogenated, or chlorinated solvents. [Stat. ref.] D. J. NORMAN.

**Application of disazo dyes to the dyeing of regenerated cellulose in even shades.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 349,960, 3.2.30).—2-Nitro- or 2:2'-dinitro-benzidine is tetrazotised and coupled with 2 mols. of 1:5:7-aminonaphthol-sulphonic acid or an *N*-substituted derivative, or with 1 mol. of such acid and 1 mol. of a phenol, naphthol, or pyrazolone, or sulphonic or carboxylic derivatives of

these; *e.g.*, 2-nitrobenzidine  $\rightarrow$  salicylic acid and 1:5:7-acid gives a level violet-brown on viscose silk.

C. HOLLINS.

**Production of colorations or coloured effects on cellulose derivative materials.** BRIT. CELANESE, LTD. (B.P. 349,683, 15.3.30. U.S., 15.3.29).—Volatile colouring matters are applied as vapour, *e.g.*, in steam, or by means of transfers, or are locally removed by volatilisation. Suitable dyes are: *p*-nitroaniline  $\rightarrow$  *p*-xylydine (orange); 4-chloro-2-nitro-4'-methyldiphenylamine (golden-yellow); 5-nitro-*o*-anisidine  $\rightarrow$  dimethyl- or diethyl-aniline (red).

C. HOLLINS.

**Dyeing and printing of fibrous materials of cellulose esters.** DURAND & HUGUENIN A.-G. (B.P. 352,808, 15.9.30. Ger., 14.9.29).—Cellulose acetate etc. is coloured by means of the leuco-compounds of gallo-cyanine dyes, since these have a greater affinity for the artificial silk than when in the fully oxidised form.

A. J. HALL.

**Finishing of [viscose] artificial silk spun on bobbins.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 353,747, 3.9.30. Ger., 3.9.29).—The freshly-spun silk is washed, desulphurised, bleached, and otherwise purified on the bobbins, and the bobbin lap is then removed in a moist condition, dried on any suitable support, but while free to shrink in length, and then unwound.

A. J. HALL.

**Mercerisation of cotton fabrics.** A. NITSCHÉ (B.P. 354,312, 5.5.30. Switz., 5.3.30).—The liquor (*e.g.*, of *d* 1.091—1.116) obtained by the counter-current washing of mercerised fabric is cooled in a heat exchanger and used for impregnating (also on the countercurrent principle) fabric prior to mercerisation. When the fabric to be mercerised is initially wet, the diluted liquor (*e.g.*, of *d* 1.045—1.059) is filtered and returned to the washing system, but when the fabric is dry the liquor is filtered and discharged to the lye recuperating tanks. Suitable apparatus is described.

D. J. NORMAN.

**Waterproofing of textile materials and the like.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,443, 29.5.30).—Yarns, leather, paper, etc. are waterproofed by superficially impregnating them with salts of multivalent metals with  $H_2O$ -sol. acid sulphuric esters of aliphatic, cycloaliphatic, or mixed aliphatic-aromatic compounds which contain at least 10 C atoms and at least one ethylenic linking or OH group, or both, or at least one group (CHO, CO, Cl, etc.) which is readily converted into a double linking or OH group, or with metal salts of true sulphonic acids of saturated or unsaturated aliphatic or cycloaliphatic compounds containing at least  $C_{10}$  in the mol. Suitable compounds include sulphopalmitic acid, cetyl hydrogen sulphate, and palmitbutylanilidesulphonic acid. *E.g.*, acetate silk is impregnated at 40—50° with a 0.5% solution of Na sulphopalmitate, squeezed, and transferred to a bath of  $Al(OAc)_3$  (*d* 1.014). After removing the excess of liquor the fabric is finally dried.

D. J. NORMAN.

**Effecting the shrinking of woollen fabrics.** TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 354,540, 31.7.30).—A continuous process is described, the material being first wetted and then

passed in festoons successively through drying, cooling, and conditioning chambers.

D. J. NORMAN.

**Conditioning of wool or the like.** A. H. GILL (U.S.P. 1,791,057, 3.2.31. Appl., 31.7.25).—1—2% of a mono-, di-, or poly-hydric phenol (*e.g.*, quinol) is added to emulsions of oils containing unsaturated constituents, as used for lubricating wool materials preparatory to spinning and related operations, since such compounds prevent atm. oxidation and spontaneous combustion.

A. J. HALL.

**Preparations for use in the treatment of textile materials with aqueous liquids.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,300, 6.2.30).—The  $H_2O$ -sol. acid phosphoric esters of saturated or unsaturated, simple or substituted aliphatic alcohols ( $C_8$  or higher) or salts of these, *e.g.*, the Na or triethanolamine salt of cetyl hydrogen phosphate or of the acid phosphoric esters prepared from the unsaturated alcohols resulting from the destructive oxidation of paraffin wax by means of air, are used in quantities of about 2—6% on the wt. of fibre for dressing fabrics of all kinds, for scouring and washing wool, as levelling agents in dyeing, or in the production of emulsions for oiling textiles.

D. J. NORMAN.

**Treatment of materials containing organic derivatives of cellulose [to increase their scroop].** BRIT. CELANESE, LTD. (B.P. 354,200, 2.5.30. U.S., 14.5.29).—The material, *e.g.*, taffeta, is treated with a solution or suspension of a Na, K,  $NH_4$ , Ba, or Ca salt of tartaric, citric, oxalic, or other polybasic aliphatic acid, optionally in admixture with oil emulsions, and after removing the excess the material is dried in a stretched condition without washing.

**Treatment of textile yarns or threads [on spools and yard beams] with fluids.** W. H. FRANCKE (B.P. 354,437, 27.5.30. Ger., 27.5.29).

**Products from  $CH_2O$  and aldehydes etc.** Wett-ing etc. agents.—See III. Cellulose esters.—See V. Degreasing of solvents.—See XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Manufacture of nitric acid by oxidation of ammonia.** G. B. TAYLOR, T. H. CHILTON, and S. L. HANDFORTH (Ind. Eng. Chem., 1931, 23, 860—865).—The low-pressure plant erected by Du Pont Co. at their Repauno Works (N.J.) comprised five converters with cylindrical, Pt-gauze catalysts and had a capacity of 25 tons  $HNO_3$  per day. Subsequently a high-pressure plant of 8 units with a total capacity of 80 tons  $HNO_3$  per day was constructed. An illustrated description is given. Cost of power is higher in the high-pressure than in the atm. process, but initial cost is about half, and there is an increase of 8—10% in acid concentration for a yield of 90% or above, when the pressure is 7.8 atm. Loss of Pt-Rh gauzes is about half that of the Pt gauzes.

W. J. WRIGHT.

**High-pressure compressors for the ammonia synthesis.** SCHNEIDER (Brennstoff-Chem., 1931, 12, 273—276).—Details are given of the compressors built

by the Demag for the Ruhrchemie A.-G. Each compresses 6000 cu. m. (referred to atm. pressure) of mixed gas from 10 atm. to 900 atm. pressure per hr.

A. B. MANNING.

**Producing dry salt [ammonium sulphate] by heat of reaction.** G. FAUSER (Chem. Met. Eng., 1931, 38, 456—457).—Gaseous  $\text{NH}_3$  is introduced into the bottom, and a spray of 65.5%  $\text{H}_2\text{SO}_4$  into the top, of a reaction chamber. The heat of reaction evaporates the  $\text{H}_2\text{O}$  and dry  $(\text{NH}_4)_2\text{SO}_4$  is removed by a conveyor. The outlet gas from the chamber passes up a tower down which the  $\text{H}_2\text{SO}_4$  flows and  $\text{NH}_3$  is absorbed. Control is effected by condensing a little of the exit gas from the chamber; the condensate should contain not less than 12—15%  $\text{NH}_3$ .

D. K. MOORE.

**Removing acidic gases by reaction with organic bases.** R. R. BOTTOMS (Chem. Met. Eng., 1931, 38, 465—467).—Natural gas containing 15%  $\text{CO}_2$ , 8% He, and 77%  $\text{N}_2$  is scrubbed with 50% aq. diethanolamine at 40° and the  $\text{CO}_2$  content is reduced to 0.01%. The aq. diethanolamine carbonate and bicarbonate is heated to boiling in a reactivating column and kettle, whereby  $\text{CO}_2$  is driven off, and, after cooling, the solution is re-used. The heat of absorption is 650 B.Th.U. per lb. of  $\text{CO}_2$  absorbed. Triethanolamine may also be used. Both ethanolamines may be used in a similar way to absorb  $\text{H}_2\text{S}$ .

D. K. MOORE.

**Acid-resisting materials.**—See I. Andalusite as refractory.—See VIII. Determining “free cyanide” in plating solutions.—See X.

See also A., Sept., 1013, Electrolysis of NaCl and KCl solutions. 1017, Catalysts for production of  $\text{H}_2$  from water-gas. 1020, Prep. of  $\text{Hg}_2\text{I}_2$  and of salts of Ce group rare earths. 1021, Prep. of monosilicic acid. Mo-blue. 1022, Prep. of polyhalides. Working up I residues. 1043, Org. Au compounds. Mg org. halides.

#### PATENTS.

**Production of hydrocyanic acid.** IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, H. A. T. MILLS, W. B. FLETCHER, and J. McAULAY (B.P. 353,407, 21.2.30. Addn. to B.P. 335,947; B., 1930, 1150).—The interaction between gaseous hydrocarbons and  $\text{NH}_3$  is carried out at 1150° or above in presence of  $\text{N}_2$ , in addition to or instead of  $\text{H}_2$ , so as to avoid deposition of C.

W. J. WRIGHT.

**Ammonia-soda process.** UNION CHIM. BELGE SOC. ANON. (B.P. 354,313, 5.5.30. Belg., 7.5.29).— $\text{NH}_4\text{Cl}$  is recovered from the mother-liquors from the manufacture of  $\text{NaHCO}_3$  by saturating the solution with  $\text{NH}_3$  and  $\text{CO}_2$ . The filtrate from the  $\text{NH}_4\text{Cl}$  is heated to eliminate excess of  $\text{NH}_3$  and  $\text{CO}_2$ , to prevent contamination of the next batch of  $\text{NaHCO}_3$  with  $\text{NH}_4\text{HCO}_3$ .

A. R. POWELL.

**Purifying the nitrogen-hydrogen mixture in the synthesis of ammonia.** L. C. JONES, Assr. to CHEM. ENG. CORP. (U.S.P. 1,794,903, 3.3.31. Appl., 2.4.27).—The gas mixture, after compression, is treated with  $\text{CO}_2$  in amount not exceeding 50% of the  $\text{NH}_3$  present, whereby  $(\text{NH}_4)_2\text{CO}_3$  is formed, condensation of which removes moisture and suspended matter from the gases before they pass over the catalyst.

A. R. POWELL.

**Production of potash [potassium carbonate] and soda.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 354,451, 4.6.30. Ger., 13.7.29).—The  $\text{NH}_3$ -soda mother-liquor obtained in the process is treated with  $\text{SrSO}_4$  (or  $\text{BaSO}_4$ ),  $\text{SrCO}_3$  is removed, and the mother-liquor is treated with sylvinit and  $\text{NH}_3$ , yielding a residue of glaserite and KCl which, after removal, is treated with cold  $\text{H}_2\text{O}$  to yield  $\text{K}_2\text{SO}_4$  and NaCl solution; the solution, after admixture with the previous liquor, is worked up to  $\text{Na}_2\text{CO}_3$  by the  $\text{NH}_3$ -soda process, and the  $\text{K}_2\text{SO}_4$  is treated with the  $\text{SrCO}_3$  to yield  $\text{K}_2\text{CO}_3$  solution and  $\text{SrSO}_4$  for re-use in the process.

L. A. COLES.

**Manufacture of hydrogen peroxide.** H. E. G. ROWLEY. From J. D. RIEDEL-E. DE HAËN A.-G. (B.P. 354,520, 12.7.30).—A solution containing 20% of  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  and 10% of  $\text{H}_2\text{SO}_4$  is atomised by superheated steam at 220° at the point at which it enters the lower end of a distillation column maintained under pressure (40 mm.). The vapours pass through the column, which is packed with refractory material and steam-heated, and thence into a receiver in which the  $\text{H}_2\text{O}_2$  is collected under reduced pressure.

A. R. POWELL.

**Manufacture of sodium nitrate.** A. MENTZEL (B.P. 353,733, 22.8.30. Ger., 31.8.29. Addn. to B.P. 347,426; B., 1931, 630).—The NaOH obtained in the prior process is subjected to further treatment with C and  $\text{N}_2$  at high temps. and then to oxidation, so as to produce  $\text{NaNO}_3$ , the liberated  $\text{NH}_3$  being returned to the  $\text{NH}_3$ -soda process and recovered as  $\text{NH}_4\text{Cl}$ . Any N oxides formed are treated with NaOH so as to give a further amount of  $\text{NaNO}_3$ .

W. J. WRIGHT.

**Regeneration of sulphides of the alkali or alkaline-earth metals.** COURTAULDS, LTD., and H. J. HEGAN (B.P. 354,607, 27.9.30).— $\text{Na}_2\text{S}$  solutions etc. which have been used, e.g., for extracting S from artificial silk, are submitted to electrolytic reduction in the cathode compartment of a diaphragm cell.

L. A. COLES.

**Precipitation of metals [lead, zinc, etc.] from solutions as sulphides.** N. C. CHRISTENSEN (U.S.P. 1,793,906, 24.2.31. Appl., 3.5.26).—Pb, Zn, Fe, and Mn may be precipitated consecutively from brine or other leaching liquors by saturating the solution with  $\text{H}_2\text{S}$  and fractionally adding further quantities of  $\text{H}_2\text{S}$  together with almost sufficient powdered  $\text{CaCO}_3$  to neutralise the acid formed in the reaction. The PbS first formed is purified from ZnS by agitating it with a further quantity of the original solution, the ZnS is freed from co-precipitated FeS by stirring it with a quantity of the solution from which Pb has been removed, and so on.

A. R. POWELL.

**Industrial treatment of leucite, or of aluminous potassic and sodic silicates, or of natural and artificial alums, for the purpose of obtaining pure alumina, compounds of potassium, or sodium and silica.** G. GALLO (B.P. 354,255, 1.5.30).—Solutions of alums containing an excess of  $\text{Na}_2\text{SO}_4$  or  $\text{K}_2\text{SO}_4$ , which may be prepared by the decomp. of leucite etc. with  $\text{H}_2\text{SO}_4$  and subsequent removal of  $\text{SiO}_2$ , are electrolysed, e.g., in a group of cells having two compartments separated by porous diaphragms and so operated that the

anodic and cathodic solutions circulate in opposite directions; the spent anode solution is used for the treatment of fresh material, and the cathode solution, which contains  $\text{NaAlO}_2$  or  $\text{KAlO}_2$ , is treated, after filtration, with  $\text{CO}_2$  to yield  $\text{Al(OH)}_3$  and  $\text{Na}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$ .

L. A. COLES.

**Production of metallic cyanates.** J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 354,604, 25.9.30).—Solutions of  $\text{NH}_4\text{CNO}$  in monohydric aliphatic alcohols or in liquid  $\text{NH}_3$  are treated with oxides or hydroxides of the alkali or alkaline-earth metals. A. R. POWELL.

**Flotation concentration of phosphate-bearing material.** W. TROTTER and E. W. WILKINSON, ASSS. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,795,100, 3.3.31. Appl., 24.1.28).—After desliming, the ore pulp is agitated with an oleate soap and petroleum oil, whereby the phosphate minerals float as a froth relatively free from silicate gangue.

A. R. POWELL.

**Manufacture of colloidal metallic [lead] phosphate.** F. E. BISCHOFF, ASSR. to H. J. ULLMANN (U.S.P. 1,794,510, 3.3.31. Appl., 23.8.27).—A 5% solution of  $\text{PbCl}_2$  in a 4% gelatin solution at  $95^\circ$  is treated drop by drop with the theoretical quantity of a dil. solution of  $\text{Na}_3\text{PO}_4$  until the  $p_{\text{H}}$  of the mixture reaches 7.4. The resulting colloidal solution of  $\text{Pb}_3\text{P}_2\text{O}_8$  is used in the form of a 0.4% solution of Pb for intravenous injection for the treatment of malignant tumours, being without toxic action on the blood.

A. R. POWELL.

**Catalytic preparations [platinised magnesium sulphate] and their applications.** IMPERIAL CHEM. INDUSTRIES, LTD., From E. I. DU PONT DE NEMOURS & Co. (B.P. 354,500, 27.6.30).— $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  is heated with 1%  $\text{Fe}_2(\text{SO}_4)_3$ ,  $\text{Th}(\text{SO}_4)_2$ , or  $\text{Ce}_2(\text{SO}_4)_3$  until a dry granular mass is obtained. This is broken up into pellets and platinised with the equivalent of 0.3% Pt as  $\text{H}_2\text{PtCl}_6$ . The catalyst has a high conversion activity in the oxidation of  $\text{SO}_2$  to  $\text{SO}_3$  and a longer life than the usual catalysts.

A. R. POWELL.

**Manufacture of catalytic masses and their employment in organic reactions.** BRIT. CELANESE, LTD., H. F. OXLEY, W. H. GROOMBRIDGE, and E. B. THOMAS (B.P. 354,351, 2.5.30).—Catalytically active elements are precipitated as oxides, hydroxides, or carbonates in the form of a highly gelatinous mass which is washed free from sol. salts and ground in a paint mill to give a thick viscous paste. This is extruded into rods or pressed into tablets which are allowed to dry in a current of cold dry air, whereby they become very hard.  $\text{ZnCO}_3$ , from  $\text{Zn(NO}_3)_2$  and  $\text{Na}_2\text{CO}_3$ , prepared as above, is an active catalyst for the production of oxygenated org. compounds from CO and  $\text{H}_2$ , and a mixture of 2 mols. of  $\text{ZnCO}_3$  with 1 mol of  $\text{MgCO}_3$  is suitable for the production of ketones from aliphatic alcohols.

A. R. POWELL.

**Supplying artificially-ionised oxygen for ventilation etc.** F. E. HARTMAN, ASSR. to F. H. and W. R. MONTGOMERY (U.S.P. 1,793,799, 24.2.31. Appl., 2.8.26).—Air containing ionised O and  $\text{O}_3$ , the former largely predominating, is obtained by subjecting air to an electronic discharge from a valve tube, the glass wall of which serves as a dielectric, so as to induce a unidirectional

corona discharge on or about the valve tube. The potential employed is sufficient to continue the electronic discharge into the free air beyond the field of the corona discharge.

A. R. POWELL.

**Recovery of sulphur from sulphide minerals.** PATENTAKTIEBOLAGET GRÖNDAL-RAMÉN, and N. E. LENANDER (B.P. 352,477, 5.4.30).—Cupriferous pyrite is smelted with coke in a blast furnace under such conditions as to produce a rich Cu matte and flue gases practically free from  $\text{O}_2$ . The gases pass through a dust-settling chamber, then through an electrostatic purifier, and finally at  $350\text{--}400^\circ$  through a catalyst chamber containing partly dehydrated bauxite, whereby the  $\text{SO}_2$  reacts with the COS,  $\text{CS}_2$ , and  $\text{H}_2\text{S}$  present to form  $\text{H}_2\text{O}$ ,  $\text{CO}_2$ , and S. The remaining  $\text{SO}_2$  is removed by addition of CO and passage of the mixture through a second, similar, catalyst chamber. S is recovered by cooling the gases to  $120\text{--}150^\circ$  in a condenser, preferably in the form of a water-tube boiler to utilise the heat. Removal of the last traces of S is effected in a second electrostatic apparatus.

A. R. POWELL.

**Collection of sulphur [from gases containing it].** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 354,207, 3.5.30).—The gases, after subjection, if necessary, to a preliminary electrostatic cleaning at a temp. above the b.p. of S, are submitted to electrostatic treatment at  $115\text{--}150^\circ$  to cause precipitation of the particles of liquid S.

L. A. COLES.

**Liquefaction of chlorine.** KREBS & Co. G.M.B.H. (B.P. 353,704, 29.7.30. Ger., 14.12.29).—A compressed mixture of  $\text{Cl}_2$  and other gases is cooled in a condenser so as to deposit part of the  $\text{Cl}_2$  in a liquid state, the remainder of the mixture being cooled and liquefied in a second condenser by part of the  $\text{Cl}_2$  so obtained, after releasing it from pressure. The cold gases from the second condenser are led to the inlet of the compressor to produce a circulation of  $\text{Cl}_2$ , and after mixing with the other gases serve to cool the compressor.

W. J. WRIGHT.

**Simultaneous production of phosphorus, or phosphoric acid, and alumina.** I. G. FARBENIND. A.-G. (B.P. 353,749, 4.9.30. Ger., 4.9.29).—A mixture of Al phosphate, heavy-metal sulphide ore, and a reducing agent is fused in an electrically-heated furnace at  $1600\text{--}1700^\circ$ , the  $\text{Al}_2\text{O}_3\text{--Al}_2\text{S}_3$  melt being removed and worked up to produce  $\text{Al}_2\text{O}_3$ .

W. J. WRIGHT.

**Fire-extinguishing material.** Absorbent for refrigerants. Gases in solid form. Absorbent for gas filters.—See I.  $\text{H}_2$  and C black.—See II. Fer-tilisers.—See VII. Gases by electrolysis.—See XI. Zn white.—See XIII. Rendering insol. phosphates available.—See XVI.

## VIII.—GLASS; CERAMICS.

**Modern methods in the production of porcelain grinding balls and lining blocks.** R. L. CAWOOD (Ind. Eng. Chem., 1931, 23, 865—867).—An automatic method employed by the Patterson Refractories Company is described. The clay is ground with  $\text{H}_2\text{O}$ , sieved, and treated in a filter press, the 40-lb. press cakes being sent to a tunnel dryer. For porcelain balls, the clay is allowed to age for several weeks. It is then transferred

to a pug mill, the extruded rolls being fed to a ball-spinning machine from which the balls are ejected by air under slight pressure and conveyed to a humidity dryer, where they remain for 48 hr. In making lining blocks, the clay from the tunnel dryer is crushed to  $\frac{3}{4}$ -in. mesh, moistened with  $H_2O$ , and allowed to soak for several days. It is then ground to 20-mesh and pressed under vac. in a block-forming machine. The blocks are dried for 18 hr. in a humidity dryer. Both the porcelain balls and the lining blocks are fired in kilns of the intermittent type, the saggars being made from a mixture of clay and grog. W. J. WRIGHT.

**Possibility of andalusite as a refractory.** F. H. RIDDLE (Trans. Amer. Electrochem. Soc., 1931, 59, 225—240).—When natural Al silicates are fired at a sufficiently high temp., cryst. mullite,  $3Al_2O_3 \cdot 2SiO_2$ , separates from the excess of  $Al_2O_3$  or  $SiO_2$ . Andalusite,  $Al_2O_3 \cdot SiO_2$ , when fired with a min. amount of a plastic aluminous bond and a suitable amount of corundum, yields a refractory which continues to improve when maintained at high temps. The occurrence of andalusite, cyanite, and similar minerals is examined and the properties of the resulting refractories are discussed. H. J. T. ELLINGHAM.

**Methods of testing refractory materials. I. Chemical analysis.** L. BELLADEN and C. F. BECCHI (Annali Chim. Appl., 1931, 21, 307—323).—To decompose the material it is powdered and 1 g. fused in a Ni crucible with NaOH or KOH for 10 min. with raw or 20—30 min. with baked products. The cooled mass is softened with  $H_2O$  in a porcelain dish on the water-bath and dissolved with HCl.  $SiO_2$  is determined as usual by double evaporation, but the silicious residue is heated on the water-bath for 6 hr. The treatment with  $H_2SO_4$  and HF is necessary because part of the Ti becomes insol. while heating the  $SiO_2$ . In order to determine Al, Ti, and Fe, the Ti residue is fused with  $KHSO_4$ , the melt dissolved in dil.  $H_2SO_4$  is added to the filtrate from the  $SiO_2$ , and the bulk made up to 250 c.c. To 100 c.c. are added 3 g. of  $NH_4NO_3$ , the solution is brought to the b.p., and aq.  $NH_3$  added in slight excess. The ppt. is collected, washed for  $1\frac{1}{2}$  hr. with neutral  $NH_4NO_3$  (methyl-red), and strongly ignited for 20 min. On another 100 c.c. portion the Fe is determined by the Zimmermann-Reinhardt method. On the remaining 50 c.c. Ti is determined colorimetrically by Weller's method. The Al is obtained by difference. Ca and Mg are determined in the filtrate from the hydroxides. O. F. LUBATTI.

**Electrical strength of porcelain.**—See XI.

See also A., Sept., 1010, System  $Fe_2O_3-SiO_2$ . 1011, System  $K_2O-CaO-SiO_2$ . 1023, Determining  $H_2O$  and  $CO_2$  in silicates.

#### PATENTS.

**Manufacture of glass yarn.** O. GOSSLER (B.P. 354,763, 10.5.30).—The glass is melted in a crucible constructed of a non-corrosive, heat-resistant alloy of Fe with, e.g., Cr, Ni, W, Mo, or mixtures of these, and serving as an electrical heating resistance when connected with a low-voltage circuit. The crucible

has a perforated bottom and may have perforated bridges etc. inside to act as filters and ribs etc. to increase the heating effect. L. A. COLES.

**Furnaces, in particular for burning enamel ware.** NAAML. VENN. FERRO ENAMELING CO. OF HOLLAND, ASSECS. OF L. W. MANION (B.P. 353,976, 19.5.30. U.S., 20.5.29).—The furnace comprises a tunnel with one closed end, the goods being conveyed in an elongated U-shaped course. The combustion chamber, or series of small chambers, is (are) situated under the furnace at the closed end and is (are) separated from the goods chamber by a roof which is corrugated to give increased radiation; the gases are led away through a long flue under the incoming run of the conveyor. B. M. VENABLES.

**Moulding sand [for cores].** R. CROSS, Assr. to SILICA PRODUCTS CO. (U.S.P. 1,795,011, 3.3.31. Appl., 22.7.29).—A sand mixture for cores comprises 90% of  $SiO_2$  sand, 7.5% of clay, and 2.5% of bentonite, with the addition of 1—2% of Portland cement, MgO, or ground blast-furnace slag to increase the binding power of the bentonite. A. R. POWELL.

**Manufacture of sheet glass.** N. V. MIJ. TOT BEHEER EN EXPLOITATIE VAN OCTROOIEN (B.P. 355,134, 17.11.30. Ger., 28.11.29).

**Electric furnace.**—See XI.

#### IX.—BUILDING MATERIALS.

**Preservation of wood by soluble salts.** WOLMAN and PFLUG (Z. angew. Chem., 1931, 44, 696—698).—Previous work on chemical preservatives for wood is discussed. The advantages of a prep. containing dinitrophenol, F, As, and Cr are described: the mixture forms a non-corrosive solution stable at the b.p.; after absorption by the wood it is converted into insol. compounds, and its fungicidal power is high.

H. F. GILLBE.

**Acid-resisting building materials.**—See I.

See also A., Sept., 1011, System  $CaO-Fe_2O_3$ . 1102, Drying of a softwood.

#### PATENTS.

**Production of artificial [slate] slabs.** F. TRAVNITSCHKE and J. FRANK (B.P. 355,056, 11.9.30).—A paste comprising cement, finely-ground slate freed from fat, long-fibre asbestos, and  $H_2O$  is pressed and cut into slabs which are then stacked, pressed, and dried.

L. A. COLES.

**Materials for use in the construction of roads, roofs, blocks, etc.** C. M. C. HUGHES and C. W. FULTON (B.P. 351,073, 9.5.30).—Tar or similar material (45—55 pts.) is mixed with "metrolfelt" pitch (5—15 pts.), and the product then mixed intimately with a slurry formed from clay (15 pts.) and  $H_2O$  (22 pts.), and, if desired, slaked lime or chalk (3 pts.).

A. B. MANNING.

**Emulsions for treatment of road surfaces and road-making materials.** F. J. E. CHINA and W. A. WHITE (B.P. 351,242, 23.6.30).—Coal-tar emulsions are prepared by using, as emulsifier,  $\frac{3}{4}$ —1½% of soap,



and as a stabilising agent an aq. extract of carrageen moss containing an amount of extractable material equiv. to 0.125–0.25% of the wt. of tar and H<sub>2</sub>O in the emulsion. A. B. MANNING.

Mixing mill.—See I. Tar for roads.—See II. H<sub>2</sub>O-resistant coatings.—See XIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Stein-Brackelsberg rotating furnace fired with pulverised coal for melting cast iron. M. STEIN (Mem. Soc. Ing. Civ. France, 1931, 84, 454–477).—The furnace comprises a long cylinder with ends in the shape of truncated cones, the whole being lined with SiO<sub>2</sub> brick. Firing is started with oil, which is replaced by powdered coal when the temp. reaches 900°. Some examples of the quality of cast Fe produced from the usual charge of pig and scrap are given together with a complete thermal balance. Temps. of 1400–1500° are readily obtained without danger of burning the metal, and owing to the rotation melting is rapid and efficient mixing of the charge is obtained. Clean castings, free from porosity and quite sound, even in very thin parts, are produced owing to the fluidity of the metal at the high temp. of the furnace. Fuel consumption varies from 9.7% for grey castings to 14.3% for malleable ones. A. R. POWELL.

Transformation of mixed gases in the open-hearth furnace. W. HEILIGENSTÄDT (Stahl u. Eisen, 1931, 51, 1045–1052).—The reactions which occur in the gases of open-hearth furnaces fired with a mixture of coke-oven and blast-furnace gases may be followed quantitatively from a knowledge of the equilibria in the reactions:  $\text{CO}_2 + \text{C} \rightleftharpoons 2\text{CO}$  and  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$ . The equilibrium consts. in the case of an open-hearth furnace at the Burlach smelter have been determined and from them the effect of varying the moisture content of the gas feed on the separation of C and on the heat consumption of the furnace has been calc. With gases saturated with H<sub>2</sub>O at 20° the effect is small, but with increasing moisture content the quantity of C which separates becomes progressively smaller, until, with gas saturated at 50°, C is no longer deposited. Simultaneously the heat requirements of the reaction increase so that the preheating temp. under the same conditions is reduced and the deposition of C still more retarded. The temp. relations in the recuperator are discussed with especial reference to the effect of the thickness of the walls on the heat exchange.

A. R. POWELL.

Distribution of the alloying elements between steel bath and slag in the production of steel. G. TAMMANN and W. OELSEN (Arch. Eisenhüttenw., 1931–2, 5, 75–80).—From a survey of the results of other workers it is shown that the distribution of Mn between the slag and the molten steel bath follows the simple law of mass action. With basic slags in which the CaO:SiO<sub>2</sub> ratio is >2, the equilibrium const. is independent of this ratio, but with the more acid slags the const. increases rapidly with the SiO<sub>2</sub> content, the increase being almost linear with 40–65% SiO<sub>2</sub>. The effect of temp. on the const. is small. The Mn distribution

reaches equilibrium values under basic slags only after the P reaction has reached equilibrium. A. R. POWELL.

Physico-chemical principles for determining the relations between steel bath and slag. G. TAMMANN (Arch. Eisenhüttenw., 1931–2, 5, 71–74).—Equations are derived, based on the electrochemical theory of the equilibria between a liquid metal mixture and a liquid electrolytic mixture (slag), by means of which it is possible to calculate the distribution of alloying elements between the metal bath and the slag layer in concentrations at which the laws applicable to dil. solutions apply. A. R. POWELL.

Decarbonisation and desulphurisation of steel and pig iron by means of hydrogen. J. CIOCHINA (Stahl u. Eisen, 1931, 51, 1024–1026).—Removal of C from Fe and steel by heating in H<sub>2</sub> starts at 700° and is rapid above 1000°, the rate of removal increasing with the C content. Desulphurisation of pig Fe in a H<sub>2</sub> atm. commences at about 650°, but is slow below 1100° and complete only at 1400–1450°; at 1480° the rate is much reduced from the max. Passing H<sub>2</sub> through molten pig Fe gives a high-quality cast Fe free from slag and oxide inclusions; under the same conditions steel is converted into Fe of Armco quality (99.75% Fe). The mechanical properties of Fe treated with H<sub>2</sub> are much superior to those of similar metal deoxidised in the usual way. A. R. POWELL.

Field of the  $\delta \rightarrow \alpha$ -transformation in the iron-nickel system. H. BENNEK and P. SCHAFMEISTER (Arch. Eisenhüttenw., 1931–2, 5, 123–125).—Thermal analysis of Fe alloys with < 50% Ni under retarded rates of cooling of 0.17–2° per sec. has confirmed the general shape of the liquidus and solidus curves, and of the  $\delta$ - and  $\alpha$ -fields, as found by Hanson and Freeman (B., 1923, 660 A) and by Kasé (A., 1927, 830). The peritectic horizontal, however, extends from 4 to 12% Ni at 1497°. This is at variance with Vogel's results (A., 1925, ii, 709), but the effects tend to be masked by supercooling. A. R. POWELL.

Effect of the alloying elements, nickel, silicon, aluminium, and phosphorus, on the solubility of carbon in liquid and solid iron. E. SÖHNCHEN and E. PIVOVARSKY (Arch. Eisenhüttenw., 1931–2, 5, 111–121).—The solid solubility of C in Fe increases linearly with the temp. from 0.84% at 733° to 1.85% at the eutectic temp., 1152°; addition of Ni displaces this line progressively towards the Fe side, i.e., Ni reduces the solid solubility of C in Fe almost linearly and depresses also the eutectoid transformation temp. Si reduces the solid solubility of C in Fe and raises the eutectoid temp. to 940° with 5.7% Si. Al has a similar effect to Si. The effects of Si and Ni are additive, at least up to 2% Si and 4% Ni. P up to 0.5% slightly reduces the solid solubility of C in Fe, but more than 0.5% P has no further action. In molten Fe the solubility of C is reduced more by addition of Al than by addition of Si up to 3%, after which Si has the more powerful depressing effect. The hardness of high-C alloys is almost halved by addition of 1% Si; further Si reduces the hardness much more slowly. The hardness curve of highly carburised Fe to which Ni is added shows two maxima at 1% and 10% Ni and flat minima at

6% and 14% Ni. Up to 1% Al halves the hardness of Fe-C alloys; further addition up to 8% has little effect, and after this the hardness rises sharply to its original value with 11% Al. A. R. POWELL.

**Determination of sulphur in cast iron and hæmatite.** G. ZENKER (Arch. Eisenhüttenw., 1931—2, 5, 101—103).—Equally good results are obtained by the  $\text{FeCl}_3\text{--Et}_2\text{O}$ , direct combustion, and evolution methods; the second procedure is recommended as the most rapid. A. R. POWELL.

**Determination of sulphur in alloy steels.** C. HOLTHAUS (Arch. Eisenhüttenw., 1931—2, 5, 95—99).—A crit. investigation of various methods of determining S in alloy steels shows that satisfactory results are obtained only by the method involving removal of  $\text{FeCl}_3$  by  $\text{Et}_2\text{O}$ , and, much more rapidly, by the combustion method. The evolution method is satisfactory only when the steel has a low Ni content or is a plain Co, Cu, Al, Mn, or Cu steel; erratic results are obtained with V steels and entirely erroneous results with steels containing W, Cr, Mo, Ti, or a high Ni content. The I method is tedious and subject to numerous sources of error. A. R. POWELL.

**Determination of small quantities of phosphate by Pouget and Chouchak's method: determination of phosphorus in small samples of steel.** S. G. CLARKE (Analyst, 1931, 56, 518—525).—The method of Pouget and Chouchak (A., 1909, ii, 266), depending on the measurement of opacity caused by the formation of strychnine phosphomolybdate, is recommended. Considerable modifications are introduced, however, both as regards the prep. of the reagents and general procedure. T. McLACHLAN.

**Use of potentiometric methods in steelworks' laboratories. II. Rapid determination of vanadium.** G. THANHEISER and P. DICKENS (Arch. Eisenhüttenw., 1931—2, 5, 105—110).—The steel is dissolved in 30 c.c. of 15%  $\text{H}_2\text{SO}_4$  and 50 c.c. of  $\text{H}_3\text{PO}_4$  (d 1.3) and the hot solution oxidised with  $\text{KMnO}_4$ , excess of which and any  $\text{CrO}_3$  formed are reduced with  $\text{FeSO}_4$ . After dilution with 200 c.c. of 10%  $\text{H}_2\text{SO}_4$ , the cold solution is again oxidised with an excess of  $\text{KMnO}_4$ , 25 c.c. of 1.25%  $\text{H}_2\text{C}_2\text{O}_4$  solution are added to reduce the  $\text{KMnO}_4$ , and, when the potential is const., the  $\text{V}_2\text{O}_5$  is titrated potentiometrically with  $\text{FeSO}_4$ . Good results are obtained by the use of a comparison solution and galvanometer as indicator. A. R. POWELL.

**Effect of small quantities of impurities in metals.** W. FRAENKEL (Z. Metallk., 1931, 23, 221—224).—The effect of small quantities of impurities on the m.p., physical and mechanical properties, recrystallisation, corrosion, deoxidation, and gas content of metals is discussed with especial reference to Cu. The recrystallisation temp. of Cu is increased by 100° by 0.1% Si and by 130° by 0.1% Mg. In Cu deoxidised with P max. electrical conductivity is obtained when the metal contains 0.01% P, when sp. gr. is also a max. The tensile strength and ductility, however, rise still further with increase in P up to a total of 0.1%. A. R. POWELL.

**Problem of grain segregation [in alloys].** E. SCHEUER (Z. Metallk., 1931, 23, 237—241).—Formulæ

are derived for calculating the composition of the mixed crystals which separate from a molten alloy in relation to the proportion of the alloy which has crystallised, assuming that no diffusion occurs during solidification. From these formulæ the composition of the  $\alpha$ -solid solution and the two stages of solidification can be calc. when the system contains a miscibility gap. The smallest possible average concentration of the  $\alpha$ -solid solution is much below the equilibrium concentration of the system, and only in the case of eutectic alloys or alloys which solidify at const. temps. are saturated mixed crystals formed without diffusion. In the  $\alpha$ -range in the Al-Cu and Cu-Sn systems the alloys solidify almost without diffusion; in the Al-Zn system they solidify with appreciable, and in the Cu-Zn system with almost complete, diffusion. A. R. POWELL.

**Effect of temperature on corrosion of zinc.** G. L. COX (Ind. Eng. Chem., 1931, 23, 902—904).—Samples were rotated in aerated, distilled  $\text{H}_2\text{O}$  at 20—100° for 15 days, the corrosion products being examined and removed, and loss in weight of the Zn determined. At 20—50° the corrosion products were gelatinous and adherent, forming a protective film; at 55—75° they were granular and non-adherent, at 95—100° dense and adherent. A graph, correlating temp. and corrosion rate, expressed as penetration in cm./year, showed slight increase in corrosion up to 50°, rapid increase with rising temp. to a max. at 65°, and then rapid decrease. The results confirm the predominating influence of the corrosion products on the corrosion, as compared with the temp. coeff. of the sp. reaction rate, the rate of transfer of  $\text{O}_2$  through the liquid, or the  $\text{O}_2$  solubility. W. J. WRIGHT.

**Distillation phenomena in brazing with zinc-copper alloys.** W. CLAUS (Z. Metallk., 1931, 23, 243—244).—From Merz and Brennecke's study of the mechanism of soldering (B., 1930, 1073) it would appear that a high brazing temp. facilitates diffusion and therefore gives a sounder joint. For practical brazing purposes, however, a high temp. is unsuitable, as not only does it result in the formation of undesirable intermediate layers which may have a low tensile or impact strength, but there is danger of serious loss of Zn by distillation. An example is quoted in which droplets of Zn containing 93% Zn and 1.6% Cu were collected from an Fe pipe which had been brazed with a 53 : 47 brass. A. R. POWELL.

**Miscibility gap in the lead-copper and lead-copper-tin systems.** S. BRIESEMEISTER (Z. Metallk., 1931, 23, 225—230).—Above 1000° Cu and Pb are miscible in all proportions; the boundary between one and two liquid phases starts from the monotectic line at 954° at 60%, extends in a smooth parabolic curve to the end of this line at 7.5% Cu, and reaches its max. at 998° with 35% Cu. Addition of Sn broadens the miscibility gap, min. solubility of Pb being reached when Cu and Sn are in the proportion of  $\text{Cu}_3\text{Sn}$ . The crit. point of the ternary system occurs at 1130—1140° with the alloy containing 35% Cu, 50% Pb, and 15% Sn. The composition of the common Pb bronzes is well outside the miscibility gap. A. R. POWELL.

**Effect of cold-stretching [of copper and aluminium] on the plasticity at high temperatures.** E. SCHMID and G. WASSERMANN (*Z. Metallk.*, 1931, 23, 242—243).—At temps. above 200° hard-drawn Cu wire is much more plastic than wire that has been annealed at 600°, the elongation at 350° of hard wire being 4 times that of soft wire in 15 min. Al wires behave similarly. This behaviour is attributed to the greater mobility of the atoms in the distorted lattice at high temps. at which there is an increased tendency for them to rearrange themselves into new undistorted crystals.

A. R. POWELL.

**Constitution of the aluminium-rich aluminium-iron-silicon alloys.** V. FUSS (*Z. Metallk.*, 1931, 23, 231—236).—A micrographical study of numerous alloys in the Al corner of the ternary diagram has shown that FeSi does not exist in this region, which contains only the following solid phases:  $\text{Al}_3\text{Fe}$ , Al, Si, and the ternary compound  $\text{Al}_6\text{Fe}_2\text{Si}_3$  which decomposes on melting into  $\text{FeAl}_3$  and Si and forms no solid solution with Al. The ternary compound forms eutectics with Si and with Al-Si solid solution and a ternary eutectic (12.5% Si, 0.5% Fe, 87% Al, m.p. 510°) with both. Two other binary eutectics also occur, one rich in Si and the other between  $\text{FeAl}_3$  and Si-saturated Al.

A. R. POWELL.

**Working up of colloidal mercury emulsions.** J. POSPIŠIL (*Chem.-Ztg.*, 1931, 55, 645—646).—Available methods for working up the Hg emulsions resulting from several technical operations have been examined and the yields obtained are generally low. A yield of almost 100% may be obtained by extracting the emulsion twice with light petroleum, drying the residue, and distilling under reflux for some hours, when the vapour condenses to large drops of Hg and org. matter is carbonised.

E. S. HEDGES.

**Electrodeposition of chromium and influence of the cathode metal.** J. W. CUTHBERTSON (*Trans. Amer. Electrochem. Soc.*, 1931, 59, 249—272).—The range of permissible  $\text{SO}_4^{--}$  concentration in Cr-plating baths operated at about 40° contracts with increasing  $\text{CrO}_3$  concentration (especially if Fe is present), so that the  $\text{CrO}_3$  should not exceed 3.25*M*. With higher  $\text{CrO}_3$  concentrations the current efficiency falls but the range of permissible c.d. is not much reduced except at higher temps. With lower  $\text{CrO}_3$  concentrations, raising the temp. increases the current efficiency and widens the range of c.d. for bright plating, but too high a temp. increases the equilibrium concentration of  $\text{Cr}^{+++}$ . The use of Fe anodes is condemned because Fe in the bath contracts the range of permissible  $\text{SO}_4^{--}$  concentration, increases the optimum ratio of  $\text{CrO}_3$  to  $\text{SO}_4^{--}$  concentrations and the resistance of the solution, and causes accumulation of  $\text{Cr}^{+++}$  owing to the low O overvoltage of Fe. Pb anodes and tanks lined with antimonial Pb keep down the concentration of  $\text{Cr}^{+++}$ . For plating on Fe, steel, or Ni the ratio of  $\text{CrO}_3$  molarity to  $\text{SO}_4$  normality should be as high as 60. Owing to the low H overvoltage of these metals, the high initial c.d. necessary to start deposition liberates much  $\text{H}_2$  and causes internal strains, and on lowering the c.d. the structure of the deposit tends to change. Hence for

deposition on Fe or steel an undercoat of a metal of moderately high H overvoltage, *e.g.*, Cu, increases the throwing power of the Cr-plating bath and gives Cr deposits more resistant to corrosion. In these respects Ni is not good as an undercoat, but it is frequently used because it leads to harder and brighter Cr deposits. The deposition of Ni undercoats on Zn and Al is described. Improved agitation and the use of specially-shaped anodes are suggested as means of increasing the throwing power of Cr-plating baths. H. J. T. ELLINGHAM.

**Thick chromium-plate.** R. J. PIERSON (*Chem. Met. Eng.*, 1931, 38, 445—448).—For thick deposits Cr is plated directly on to the foundation metal. The hardness, porosity, and internal stress of the plate are influenced by the c.d., temp., concentration of  $\text{CrO}_3$ ,  $\text{SO}_4^{--}$ , and  $\text{Cr}^{+++}$  ions, and by added ingredients, *e.g.*, Fe; these variables are interrelated. Shaped anodes are used to ensure uniform current distribution, as the rapid increase of current efficiency with c.d. tends to non-uniform deposition. There should be thermostatic control of the plating bath and continuous filtration of the plating liquid and maintenance of the concentration of  $\text{CrO}_3$  and  $\text{SO}_4$  ions. Pb-lined tanks and Pb anodes should be used. The article to be plated should be washed in grease solvent, *e.g.*, fish oil, and then cleaned in an electric cleaner. No fume preventive, *e.g.*, a layer of kerosene on the plating bath, should be used. A 5000-amp. motor will plate 10 sq. ft. at the rate of 0.002 in./hr. The economics of Cr-plating are given.

D. K. MOORE.

**Tarnishing of chromium-plated brass.** W. M. PHILLIPS (*Trans. Amer. Electrochem. Soc.*, 1931, 59, 397—398).—Tarnishing of Cr-plated brass or Cu articles is attributed to absence or insufficient thickness of the Ni undercoat. Outdoor exposure tests show the importance of an adequate Ni-coating as a preliminary to Cr-plating on steel.

H. J. T. ELLINGHAM.

**Control of acidity in low- $p_{\text{H}}$  nickel-plating baths.** L. C. PAN (*Trans. Amer. Electrochem. Soc.*, 1931, 59, 295—302).—For a low- $p_{\text{H}}$  bath with given concentrations of  $\text{NiSO}_4$ ,  $\text{NiCl}_2$ , and  $\text{H}_3\text{BO}_3$ , control of the total acidity, determined by titration with 0.1*N* aq.  $\text{NH}_3$ , is sufficient to keep plating conditions const. A chart is provided to facilitate calculation of the amount of acid or alkali to be added to a bath in order to correct any deviation from the optimum acidity for a given bath composition.

H. J. T. ELLINGHAM.

**Further developments in low- $p_{\text{H}}$  nickel deposition.** W. M. PHILLIPS (*Trans. Amer. Electrochem. Soc.*, 1931, 59, 393—396; cf. B., 1931, 161).—Steel plated successively with Cu, Ni, and Cr was much less liable to atm. corrosion if the Ni had been deposited from low- instead of high- $p_{\text{H}}$  baths. The hardness of Ni deposits is scarcely affected by the  $p_{\text{H}}$ , but with given  $p_{\text{H}}$  and c.d. it decreases with rise in temp. At a given temp. low- $p_{\text{H}}$  baths can be operated at a higher c.d. (20 amp./sq. ft. at 38°) without causing peeling of the deposit. In order to avoid roughness of heavy deposits from such baths, the work must be free from adhering particles of impurities, the anodes should be enclosed in muslin bags, and the solution filtered comparatively

often. The higher the  $\text{NiSO}_4$  concentration, the wider is the permissible range of operating conditions for solutions of given  $p_{\text{H}}$ .

H. J. T. ELLINGHAM.

**Preparation of pure electrolytic nickel. III. Chemical and physical properties.** C. G. FINK and F. A. ROHRMAN (Trans. Amer. Electrochem. Soc., 1931, 59, 399—408).—Electrolytic Ni prepared by the method previously described (A., 1930, 1382) is shown spectrographically to be free from all probable impurities (including Co and Fe) except for a trace of Cu (too small to be determined), but it contained traces of Na and B derived from the Na borate buffer used in the bath. These latter impurities can be avoided by using only  $\text{NH}_4$  salts as buffers. The pure Ni gave a temp. coeff. of electrical resistivity of 0.00640 and had m.p.  $1458^\circ$ .

H. J. T. ELLINGHAM.

**Electrodeposition of silver from sulphate, nitrate, fluoborate, and fluoride solutions.** E. B. SANIGAR (Trans. Amer. Electrochem. Soc., 1931, 59, 359—379).—Relatively compact deposits of Ag were obtained on a Ag surface, but, owing to simple immersion deposition, not on baser metals, from solutions of  $\text{Ag}_2\text{SO}_4 + \text{H}_3\text{BO}_3$  or  $\text{Fe}_2(\text{SO}_4)_3$ ;  $\text{AgNO}_3 + \text{H}_3\text{BO}_3$ ;  $\text{AgBF}_4$ ; or  $\text{AgF}$ ; but all the deposits except two from  $\text{AgBF}_4$  solution were more or less coarsely cryst. and tended to form trees. Also corrosion of the anode was generally poor or irregular. Even the best deposits from the  $\text{AgBF}_4$  bath did not compare favourably with those from the usual argenticyanide solution for plating purposes.

H. J. T. ELLINGHAM.

**Silver-plating solutions.** B. EGEBERG and N. PROMISEL (Trans. Amer. Electrochem. Soc., 1931, 59, 339—357).—The effects of temp., c.d., and the concentrations of Ag, cyanide, carbonate, and  $\text{CS}_2$  on the resistivity, polarisation, throwing power, and appearance of the plate in the deposition of Ag from Na and K argenticyanide solutions are shown by a series of curves. Many difficulties with new solutions are removed by an initial addition of carbonate, which decreases the resistivity and increases the throwing power. The formation of black slime on the anodes is avoided if the anodic polarisation is kept down by increasing the cyanide or decreasing the carbonate content. Addition of  $\text{CS}_2$  decreases cathodic polarisation, but lowers the throwing power. The max. c.d. permissible without "burning" the deposit is raised by increasing the temp., the agitation, and the  $\text{CS}_2$  content, and by using KCN instead of NaCN. Baths prepared from KCN have a higher conductivity, tend to give deposits of better appearance, and permit a higher cathodic polarisation before burning occurs. A slightly higher cyanide content is needed to maintain a low anodic polarisation with KCN solutions, but this polarisation increases only slightly with increase of carbonate concentration. The throwing power of these plating solutions appears to be increased by decrease of temp. or Ag content, or by increase of c.d. or concentration of cyanide or carbonate.

H. J. T. ELLINGHAM.

**Change in reflecting power caused by tarnishing electrodeposited silver-cadmium alloys.** L. E. STOUT and W. G. THUMMEL (Trans. Amer. Electrochem. Soc., 1931, 59, 303—314).—Ag-Cd has been electrodeposited under various conditions from unagitated

baths prepared by mixing a bright Cd-plating solution (B., 1929, 560) with varying proportions of an argenticyanide solution. Insol. anodes were used. The ratio Ag:Cd in the deposit is approx. proportional to the corresponding ratio in the solution, but decreases with increase of c.d. in a bath of given composition. Alloys of moderate Ag content had a reflecting power approaching that of Cd (very much lower than that of Ag), but were much harder and more resistant to abrasion than was electrodeposited Cd and suffered little loss of reflecting power on immersion in a Na polysulphide solution under conditions which reduced the reflecting power of Ag to about 11% of its original value. An alloy containing about 24% Ag seems to be particularly suited for use as a reflecting surface in these respects.

H. J. T. ELLINGHAM.

**Electrodeposition of tungsten from aqueous solutions.** C. G. FINK and F. L. JONES (Trans. Amer. Electrochem. Soc., 1931, 59, 273—293).—Thin, Ag-white deposits of W can be obtained on brass by electrolysis of alkaline tungstate solutions above  $60^\circ$ . Baths prepared by dissolving  $\text{WO}_3$  in NaOH solutions require an addition agent such as dextrose, apparently for regulating  $[\text{H}^+]$ , but this is unnecessary if the  $\text{WO}_3$  is dissolved in  $\text{Na}_2\text{CO}_3$  solution saturated at  $100^\circ$ . C.ds. up to 400 amp./sq. ft. give good deposits, but the best current efficiency is attained by operation at 75—100 amp./sq. ft. The effects of  $\text{WO}_3$  concentration and temp. on the quality of the deposit were examined. In all cases the current efficiency is very low. Pt anodes are employed since W anodes dissolve at high current efficiency and the alkalinity of the bath falls. Using Ni anodes deposits of Ni-W alloys insol. in HCl or NaOH solutions were obtained; Fe-W alloys were also prepared. The W deposits exhibit considerable resistance to corrosion if free from pits and flaws. Previous work on the electrodeposition of W is reviewed and the theory of the present method discussed.

H. J. T. ELLINGHAM.

**Definition and determination of "free cyanide" in electroplating solutions.** W. BLUM (Trans. Amer. Electrochem. Soc., 1931, 60, 21—26).—In view of the uncertainty as to the nature of the compounds existing in complex metallic cyanide solutions it is proposed to define the term "free cyanide" arbitrarily but unambiguously as the excess of alkali cyanide above the min. required to give a clear solution. The problem of the analytical determination of this quantity is discussed.

H. J. T. ELLINGHAM.

See also A., Sept., 1005, Systems Al-Ag and Cr-C. 1016, Transformation of white into grey tin. 1018, Electrolytic separation of Nb and Ta. 1025, Determining O in Al. 1026, Determining mixed Cr and Ni in solution.

#### PATENTS.

**Reduction of ores with gases.** F. M. WIBERG (B.P. 352,580, 23.4.30. Swed., 6.7.29).—The hot reducing gas is passed through a series of vertical chambers containing the ore in such a way that it passes downwards through the first chamber and upwards through the second, and so on. When the reducing power of the gas is considerably lowered part is withdrawn from the system and passed to a regenerator (e.g., a glowing column of coke), whilst

the remainder effects a preliminary reduction of the ore in the succeeding chambers, and finally by admission of air is burned in the last chambers to preheat the ore therein. As reduction is completed, the chambers are cut out of the circuit, emptied, refilled, and passed to the end of the series. A. R. POWELL.

**Reducing the iron in iron oxides to its metallic form.** W. W. PERCY (U.S.P. 1,792,507, 17.2.31. Appl., 10.6.26).—Preheated Fe ore is fed continuously into a vertical shaft up which is passed a current of hot water-gas or producer gas. The reduced Fe sponge is withdrawn from the bottom of the shaft, and the gases from the top (comprising equal vols. of CO and CO<sub>2</sub>) are passed downwards through a glowing mass of incandescent C from a hydrocarbon fuel contained in a bottom-blown gas regenerator, the hot gases from which are returned to the reducing chamber. A. R. POWELL.

**Manufacture of open-hearth steel.** A. L. FEILD, Assr. to REPUBLIC STEEL CORP. (U.S.P. 1,794,068, 24.2.31. Appl., 29.7.26).—Steel is refined in the open-hearth furnace by maintaining on the top of the liquid metal a slag containing a const. % of FeO, determined according to the equation  $\frac{dx}{dt} = 0.0000757kw \times (1.08 - 1/x)$ , where  $\frac{dx}{dt}$  is the desired rate of C elimination,  $k$  is a const.,  $w$  the % of FeO in the slag, and  $x$  the % C in the metal after time  $t$ . A. R. POWELL.

**Heat-treating furnace [for sheet steel].** R. S. COCHRAN, Assr. to SURFACE COMBUSTION Co., INC. (U.S.P. 1,792,074, 10.2.31. Appl., 3.10.28).—Steel sheets are conveyed through an elongated furnace on a hearth composed of longitudinal bars which alternately advance in a raised position and recede while lowered; the bars are deep in proportion to their width and are formed with transverse apertures through which gases pass from burners alternately on either side. To prevent the long edges of the sheets from bending, the sheets are encouraged to become corrugated transversely by forming the bars with a wavy upper surface. B. M. VENABLES.

**Heat-treatment of iron and steel sheets.** H. M. COX and C. M. SMITH, Assrs. to REPUBLIC STEEL CORP. (U.S.P. 1,792,573, 17.2.31. Appl., 31.1.29).—Mild steel sheets (0.07–0.20% C) are heated at 720° for 3 min., then at 985° for 3 min., cooled rapidly to 595°, maintained at this temp. for 1 min., reheated at 750° for 4 min., and allowed to cool slowly in the furnace to 590° and then normally in the air. Sheets thus treated have the good drawing and stamping properties of normalised sheets combined with the softness of box-annealed sheets, and can be stamped or deep-drawn without wrinkling or cracking. A. R. POWELL.

**Production of pure iron.** W. G. CLARK (U.S.P. 1,792,967, 17.2.31. Appl., 22.4.29).—Air is blown through molten Fe in a Bessemer converter until 75% of the Fe is converted into oxide; the air is then replaced by He, A, or N<sub>2</sub>, and this in turn is replaced by H<sub>2</sub>, which rapidly reduces the oxide to pure Fe containing < 0.002% C. A. R. POWELL.

**Casting. [Manufacture of cast iron.]** E. R. WILLIAMS, Assr. to VULCAN MOLD & IRON Co. (U.S.P. 1,793,268, 17.2.31. Appl., 22.6.29).—Cast Fe having

a pearlitic structure is produced by melting in a cupola a charge with a low content of C and Si with sufficient Na<sub>2</sub>CO<sub>3</sub> to reduce the S content below 0.05%, casting the charge into cold moulds, and allowing the castings to cool at a normal rate. A. R. POWELL.

**[Iron] ingot mould.** E. R. WILLIAMS, Assr. to VULCAN MOLD & IRON Co. (U.S.P. 1,795,065, 3.3.31. Appl., 22.6.29).—The mould is made of cupola-cast Fe containing < 0.05% S, which is claimed to have a high resistance to cracking through sudden expansion. A. R. POWELL.

**Case-hardening [of iron].** J. J. EGAN, Assr. to ELECTRO METALLURG. Co. (U.S.P. 1,793,309, 17.2.31. Appl., 26.2.30).—Ferrous metals are case-hardened by heating them at 460–580° in a 4 : 6 mixture of NH<sub>3</sub> and NO. A. R. POWELL.

**Manufacture of shaped articles from metal [iron] powders.** J. Y. JOHNSON. From I. G. FARBERININD. A.-G. (B.P. 354,562, 21.8.30).—Shaped metal articles produced from powdered Fe (carbonyl method) are sintered in such a position that the direction in which max. shrinkage occurs is about vertical, so that the sintered article is free from cracks. A. R. POWELL.

**Apparatus for treating flexible metal bodies, cleaning iron wires, bands, etc.** F. M. DORSEY, Assr. to MADSENELL CORP. (U.S.P. 1,793,914, 24.2.31. Appl., 2.9.27).—The wire is anodically treated by passing it up and down through a bath of conc. H<sub>2</sub>SO<sub>4</sub> over pulleys arranged alternately at the top and bottom so that the wire in its passages through the bath passes continuously between a series of cathodes. Means are provided for turning the wire to and fro about its longitudinal axis. A. R. POWELL.

**[Inhibitor for] pickling metals [iron].** V. VOORHEES, Assr. to STANDARD OIL Co. (U.S.P. 1,793,146, 17.2.31. Appl., 18.1.28).—The pickling bath is made up wholly or in part from acid sludge derived from the treatment of cracked petroleum oil with H<sub>2</sub>SO<sub>4</sub>, the org. compounds contained therein acting as inhibitors in the pickling of ferrous metals. A. R. POWELL.

**Inhibitor for [steel-]pickling baths.** E. C. WRIGHT, Assr. to NAT. TUBE Co. (U.S.P. 1,792,958, 17.2.31. Appl., 19.6.29).—The inhibitor is prepared by allowing H<sub>2</sub>SO<sub>4</sub> to react for 24 hr. with half its vol. of coal tar (boiling range 270–350°). A. R. POWELL.

**Manufacture of rustless iron alloys.** F. M. BECKET and J. H. CRITCHETT, Assrs. to ELECTRO METALLURG. Co. (U.S.P. 1,793,153, 17.2.31. Appl., 20.7.27).—Fe-Cr alloys containing > 2% C are blown in a side-blown converter with gases containing 50–100% O<sub>2</sub>, whereby a temp. above 1600° is obtained at which all the impurities are oxidised and the C is reduced below 0.2% without appreciable oxidation of the Cr. A. R. POWELL.

**Steel capable of resisting rust and corrosion.** R. KESSELRING and J. NOWAK (B.P. 354,177, 31.3.30. Ger., 30.3.29).—The steel is free from Si and contains 0.1–1% Cr, 0.25–0.5% Cu, 0.04–0.25% Ni, and 0.01–0.05% W, and is made by adding a ledeburitic cast-Fe alloy, containing Cr, Cu, Ni, and W, but free from graphite, to steel produced in the usual manner. A. R. POWELL.

**Welding electrode.** J. G. RUTTER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,794,983, 3.3.31. Appl., 29.6.28).—For welding ferrous metals and alloys a welding rod comprising 65–45 (58)% Fe and 35–55 (42)% Ni and having its surface Cu-plated is claimed. Owing to the low coeff. of expansion of this alloy the weld is free from internal stress and has no tendency to warp.

A. R. POWELL.

**Welding of steel parts.** L. C. DELACHAUX (B.P. 352,714, 30.6.30. Fr., 27.7.29).—For welding air-quenching steel having a Brinell hardness above 220 the welding rod contains up to 5% Ni, 2% Cr, 1% Mo, and 1.5% W, together with extra C up to 0.7% and, if desired, deoxidising metals, e.g., up to 1% Al, Ti, or B. The composition is so regulated that the weld has the same Brinell hardness and mechanical properties as the metal to be welded.

A. R. POWELL.

**Metal cement [for welding cutting-tool edges to shafts].** W. R. CHAPIN (U.S.P. 1,793,156–7, 17.2.31. Appl., 4.1.30).—A welding powder for joining WC and high-speed steel tips to steel shafts comprises (A) 65% of Si–Mn, 25% of fine steel filings, and 10% of  $\text{Na}_2\text{B}_4\text{O}_7$ , or (B) 40% of Si–Mn, 25% of powdered Cu, 25% of steel filings, and 10% of  $\text{Na}_2\text{B}_4\text{O}_7$ .

A. R. POWELL.

**Uniting iron with other alloys or elements [to form alloys without fusion].** W. H. SMITH (U.S.P. 1,793,757, 24.2.31. Appl., 5.2.27).—A mixture of sponge Fe and the powder of the element to be alloyed with it is compressed into the desired form, which is then heated until the mass sinters and becomes plastic; it is then worked into the final shape by the application of pressure.

A. R. POWELL.

**Magnetic material. [Nickel-cobalt-iron alloy.]** G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC., (U.S.P. 1,792,483, 17.2.31. Appl., 31.5.29).—Alloys containing 10–80% Ni, 5–80% Co, 9–50% Fe, and 1–10% Cr, together with small amounts of one or more of the elements, Mo, W, Mn, V, Ti, Ta, Zr, Cu, and Si, and heat-treated to have an initial permeability above 200, are claimed.

A. R. POWELL.

**Concentration of cassiterite ores and the like [by flotation].** E. J. LAWRENCE and J. A. DANIEL (B.P. 354,395, 24.7.30).—The cassiterite particles are superficially metallised by nascent H generated from Zn dust coated with Sn or from a powdered Sn–Zn alloy containing 1% Sn, and the pulp is then subjected to a flotation process.

A. R. POWELL.

**Selective agent for concentrating ores.** R. LUCKENBACH (U.S.P. 1,792,544, 17.2.31. Appl., 28.11.28).—The agent claimed is a mixture of 3 pts. of candle tar, 1 pt. of petroleum, and 1 pt. of  $\text{Na}_2\text{SiO}_3$ . This is fed on to an inclined moving belt down which the ore pulp is allowed to run; the sulphides, metals, and oxidised metallic minerals adhere to the oiled belt and are scraped off when the gangue and water have been removed.

A. R. POWELL.

**Aluminium-copper light-metal alloys.** O. KAMPS (B.P. 354,202, 2.5.30).—The alloy comprises Al with 2–5% Cu, 0.2–1% Ag, 0.02–0.2% Ti, 0.2–0.8% Ni, and 0.1% Pd or V. Prior to casting, the molten alloy is treated with an Al cartridge containing  $\text{SrCl}_2$ .

A. R. POWELL.

**Production of oxidic coatings on articles made from aluminium and its alloys.** R. E. MÜLLER (B.P. 352,656, 26.5.30. Ger., 24.5.29).—In the production of protective oxide coatings on Al articles by electrolysis, minute particles of  $\text{Fe}_2\text{O}_3$ , CuO,  $\text{SiO}_2$ , or  $\text{MnO}_2$  are incorporated into the oxide film. This may be effected by first coating the metal with a thin film of Fe, Cu, or Mn, and then subjecting it to anodic oxidation. [Stat. ref.]

A. R. POWELL.

**Fabrication of [nickel or cobalt] metal-coated articles.** F. V. KNAUSS, Assr. to MADSENELL CORP. (U.S.P. 1,793,936, 24.2.31. Appl., 31.1.25).—Ferrous metal articles are given a soft malleable coating of Ni in a sulphate bath containing chloride and boric acid, and the plated articles are subjected to cold-work, then passivated by anodic oxidation in a bath containing > 85%  $\text{H}_2\text{SO}_4$ , and finally again plated with Ni from a bath which yields hard wear-resisting deposits.

A. R. POWELL.

**Compound-coated metallic bodies and application of such coatings.** ELECTRICAL RES. PRODUCTS, INC., Assees. of E. G. STURDEVANT (B.P. 354,185, 29.4.30. U.S., 8.6.29).—A warm, plastic mixture containing reclaimed rubber 25%, smoked sheet rubber 20%, mineral rubber 5%, ZnO 5%, S 2%, pine tar 2%, whiting 38.5%, and oleic acid 2.5% is extruded on an electrical conductor and vulcanised.

J. S. G. THOMAS.

**Metal [aluminium] alloys.** C. VANGREVENYNGE, and SOC. LES FILS DE V. BIDAULT ET CIE. (B.P. 352,759, 30.7.30. Fr., 7.1.30).—Alloys of Al with 1.5–5 (3.4)% Cu, 0.25–2.5 (0.5)% Ni, 0.25–1.5 (0.5)% Cr, and 0.25–2 (0.75)% Mg are claimed. After quenching from 500° and ageing at the ordinary temp. for 8–10 days, the alloys have a tensile strength of 45 kg. per sq. mm. and an elongation of 20%.

A. R. POWELL.

**Production of light-metal pistons running in cylinders of harder material.** F. FEHRENBACH (U.S.P. 1,792,580, 17.2.31. Appl., 12.8.27).—The pistons are made by casting an alloy of Al with > 18 (e.g., 20–25)% Si to which has been added 5% of finely-divided hard metal which is almost or entirely insol. in solid Al. The castings are immediately chilled, whereby the hard metal becomes distributed over the surface, thus increasing its wearing properties.

A. R. POWELL.

**Manufacture of tungsten carbide alloys.** J. T. TERRY (U.S.P. 1,792,943, 17.2.31. Appl., 22.12.28).—W powder mixed with CaO is fused in a graphite crucible in a resistance furnace, whereby a layer of WC containing no free graphite is formed below the  $\text{CaC}_2$  slag.

A. R. POWELL.

**Manufacture of hard metal compositions [cobalt-tungsten carbide].** (A) F. C. KELLEY, and (B) S. L. HOYT, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,794,300 and 1,794,229, 24.2.31. Appl., [A] 7.1.28, and [B] 5.12.29).—(A) A mixture of W with 3–10% C and < 25% Co is ground intimately, moistened with  $\text{COMe}_2$ , and rubbed into a stiff paste with 2% of a glycerolphthalic anhydride resin. The paste is shaped, dried, and sintered under pressure at 1375°. (B) A mixture of 97–80% WC and 3–20% Co in powder form is pressed

into bars which are sintered at 1375° and worked into the desired shape by pressure at this temp.

A. R. POWELL.

**Lead-purifying process.** J. B. SCHUETTENHELM, ASSR. to BUNKER HILL & SULLIVAN MINING & CONCENTRATING Co. (U.S.P. 1,794,647, 3.3.31. Appl., 31.3.30).—Cu and As are removed from Pb by treating the molten metal with  $\text{NH}_4\text{Cl}$  in a closed Fe pan containing a revolving paddle inside a centrally disposed cylindrical chamber through which the Pb and  $\text{NH}_4\text{Cl}$  are caused to circulate by the rotation of the paddle.

A. R. POWELL.

**Alloys of palladium, silver, and copper.** INTERNATIONAL NICKEL Co., INC., ASSEES. of E. M. WISE (B.P. 354,216 and 354,239, 28.1.30. U.S., 28.1.29).—(A) The alloy contains 25–98% Pd, 50–1% Cu, and >1% Ag, preferably 40–50% Pd, 14–30% Cu, and the remainder Ag. The alloys can be hardened by quenching from above 750° and tempering at 350–550°, preferably 450°. (B) An alloy of Ag with < 25% Pd and 1–50% Cu deoxidised with Zn, Mn, B, Si, Mg, or Ca is claimed.

A. R. POWELL.

**Manufacture of chromium or its alloys.** GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 354,387, 10.5.30).—Cr or its alloys are melted in  $\text{H}_2$ , and this atm. is replaced by one of  $\text{N}_2$  about 15–60 sec. before casting.

A. R. POWELL.

**Forming floating metal powders.** J. R. CAIN, ASSR. to RICHARDSON Co. (U.S.P. 1,794,585, 3.3.31. Appl., 3.5.26).—Metal powder is moistened with  $\text{C}_6\text{H}_6$ , which is evaporated by gentle heating; the powder will then float on the surface of aq. solutions. Fe powder so treated will float on  $\text{CuSO}_4$  solution and the Fe will be replaced by Cu, which will also float; if this floating Cu film is made the cathode in the  $\text{CuSO}_4$  solution a thin Cu foil is readily obtained.

A. R. POWELL.

**Continuous chromium-plating of metallic wires or strips.** B. V. MCBRIDE, ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING Co. (U.S.P. 1,794,973, 3.3.31. Appl., 27.3.28).—The wire or strip is drawn continuously through a Cr-plating bath downwards between insol. anodes, horizontally along the bottom underneath an insulating diaphragm, and upwards out of the bath between a second series of insol. anodes, so that at all times when the metal is being plated it is equidistant at all parts from the anodes, but is protected from the action of the current during its passage across the bottom of the bath.

A. R. POWELL.

**Fractionally [chromium-]plating objects.** V. L. SÖDERBERG (U.S.P. 1,794,929, 3.3.31. Appl., 7.5.27).—The parts of the article which are not to be plated are covered with a film of nitrocellulose lacquer.

A. R. POWELL.

**Plating of metallic surfaces [with chromium].** E. O. DUNKLEY, ASSR. to STANDARD OIL Co. of CALIFORNIA (U.S.P. 1,793,069, 17.2.31. Appl., 23.1.28).—The interior of cylindrical oil-refining vessels is plated with Cr by means of a continuous, ring-like, electrolyte chamber, the outer wall of which is formed by the inner wall of the vessel to be plated. The chamber contains a cylindrical anode along its inner wall, and is adapted to be moved up and down the walls of the vessel; means

are provided also for keeping the electrolyte at a const. level in the chamber.

A. R. POWELL.

**Electrodeposited [nickel] diaphragm.** A. HEWITT, ASSR. to VICTOR TALKING MACHINE Co. (U.S.P. 1,793,483, 23.2.31. Appl., 11.5.26).—An acoustic diaphragm of thin Ni sheet is produced by the electrodeposition of Ni on a rotating cathode mould having a corrugated surface, a uniform thin layer being first formed and the deposition subsequently continued while part of the cathode is shielded.

A. R. POWELL.

**Electroplating zinc on (A—C) iron or steel, (C) ferrous articles.** (A, B) S. H. DAVIS, C. O. ANDERSON, W. N. SMITH, H. R. HANLEY, with, in (C), R. J. STENGL, ASSRS. to CENTURY ZINC Co. (U.S.P. 1,795,079—1,795,081, 3.3.31. Appl., [A] 12.9.28, [B] 2.11.28, [C] 13.3.29. Renewed [B] 21.8.30).—(A) The process claimed utilises as electrolyte the purified leach liquor obtained by leaching roasted Zn ores with dil.  $\text{H}_2\text{SO}_4$ . The articles are cleaned in the usual way, given a flash coating of Zn in a feebly acid  $\text{ZnSO}_4$  solution at 120 amp./sq. ft., and finally plated to the desired thickness at 60 amp./sq. ft. in a bath containing 10%  $\text{H}_2\text{SO}_4$ . In both plating operations insol. anodes are used and the electrolyte from the second operation is progressively returned to the leaching tank as its acidity increases and is replaced by electrolyte from the first plating bath, which is made up directly from the purified leach liquor. (B) The articles are first plated with a thin film of Pb which is brushed or burnished into the surface before the Zn coating is applied. (C) Three Zn coatings are given, the first at 100 amp./sq. ft. from a bath containing 2% of  $\text{H}_2\text{SO}_4$ , the second at 150 amp./sq. ft. from a solution containing 5%  $\text{H}_2\text{SO}_4$ , and the third at 100 amp./sq. ft. from the same solution.

A. R. POWELL.

**Mixing mill.**—See I. **Moulding sand.** **Furnaces for enamel ware.**—See VIII. **Electric furnace.** **Anodes for electroplating.**—See XI.

## XL—ELECTROTECHNICS.

**Söderberg electrode, oblong electrodes, and other new developments.** M. SEM (Trans. Amer. Electrochem. Soc., 1931, 60, 199–210).—The difficulties in operating Söderberg electrodes of oblong section have been largely overcome, and they have been used in ferro-alloy and other furnaces. The economic advantage of these electrodes over the ordinary cylindrical type is still doubtful. Söderberg electrodes can be fed into the furnace by raising the holder by means of a winch, or by arranging for automatic slipping of the electrode through the holder, taking advantage of the shrinkage on baking. The use of a dust shield around the upper part of the electrode also permits better temp. control in this region. At a suitable temp. the binder is melted, but baking above the holder is prevented, and the electrode material can be introduced in cold lumps and no ramming is needed. By using flexible cables encased in flexible metal hose, through which  $\text{H}_2\text{O}$  is passed to cool the cable itself and the electrode holder, a 7-fold increase in c.d. over that permissible with ordinary Cu flexible cable can be attained.

H. J. T. ELLINGHAM.



**Electrolytic phenomena in oxide-coated filaments.** J. A. BECKER (Trans. Amer. Electrochem. Soc., 1931, 59, 381—391).—From a crit. examination of published work it is concluded that the current through BaO coatings on metallic filaments is carried partly by electrons and partly by Ba and O ions, the proportion depending on the previous history of the coating. Preliminary results of a study of polarisation effects and the temp. variation of conductivity support this conclusion.

H. J. T. ELLINGHAM.

**Electrical strength of porcelain.** J. O. KRAEHNBUHL and C. W. PARMELEE (Trans. Amer. Electrochem. Soc., 1931, 59, 409—425).—The puncture track in a specimen of porcelain is largely a matter of chance and depends on flaws in the material. Hence a large number of samples must be tested in order to obtain a proper estimate of quality. A statistical study has been made of the results obtained with a number of samples prepared under controlled conditions.

II. J. T. ELLINGHAM.

**Electro-ultrafiltration.**—See I. **Hydrocarbons from water-gas.**—See II. **Determining V in steel.** **Plating of Cr, Ni, Ag, Ag—Cd, W.** **Determining "free cyanide" in plating solutions.**—See X.

See also A., Sept., 1005, **Magnetic properties of Co—Cr mixed crystals.** 1013, **Electrolysis of NaCl and KCl solutions.** 1014, **The Leclanché cell.** 1018, **Separation of Nb and Ta.** 1038, **Oxidation of sugars.**

#### PATENTS.

**[Electric] induction heating.** E. F. NORTHROP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,791,934, 10.2.31. Appl., 21.12.25).—A horizontal drum having a magnetic shell containing material to be treated and wound with an inductor surrounding substantially its whole length, connexions for sending current through different parts of the inductor either in series or in parallel, and power factor correction for the current is claimed.

J. S. G. THOMAS.

**Electric furnaces [for heat-treatment of steel, ceramic material, etc. above 1100°].** CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 354,172, 3.3.30).—An outer insulating casing surrounds a non-metallic refractory lining, *e.g.*, of SiC, Al<sub>2</sub>O<sub>3</sub>, or fireclay, forming a heating chamber and having a thermal conductivity of > 0.006 c.g.s. unit and a heat capacity comparable with that of the charge. Heating resistors having positive and/or negative temp. coeffs. of resistance, *e.g.*, of SiC and/or C, are arranged in the heating chamber.

J. S. G. THOMAS.

**Mounting means for the heating elements of electric resistance furnaces.** HEVI DUTY ELECTRIC Co., Assees. of E. L. SMALLEY (B.P. 351,928, 21.6.30. U.S., 22.6.29).—A preformed heating element of the return-bend type is self-interlocked in the apertures of the supporting pedestal.

J. S. G. THOMAS.

**Arc-extinguishing fluids.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. D. HILLIARD (B.P. 355,145, 26.11.30. U.S., 26.11.29).—A mixture of a dry mineral oil of high dielectric strength and a detonation suppressor, *e.g.*, NH<sub>2</sub>Ph, C<sub>6</sub>H<sub>6</sub>, PhMe, is claimed.

J. S. G. THOMAS.

**Compositions having arc-resistant properties.** BAKELITE CORP., Assees. of F. GROFF (B.P. 354,166, 29.1.30. U.S., 31.1.29).—The condensation product obtained by heating a phenol and a fatty oil, *e.g.*, tung oil, with a promoter, *e.g.*, H<sub>3</sub>PO<sub>4</sub>, is heated, with or without addition of a methylene-containing agent, until free phenol is eliminated, and is then mixed with a filler, *e.g.*, gelatinisable cotton flock.

J. S. G. THOMAS.

**Production of [electrical] insulating materials.** ELECTRICAL RES. PRODUCTS, INC., Assees. of A. R. KEMP (B.P. 354,380, 9.5.30. U.S., 3.2.30. Addn. to B.P. 307,966; B., 1929, 401).—Latex or crude rubber is treated at 145—150° with caustic alkali solution of concentration up to 2% under pressure (0.5—5 kg./sq. cm.). Latex, during treatment, is diluted with 0.5—3 times its vol. of H<sub>2</sub>O and finally coagulated by addition of dil. AcOH.

J. S. G. THOMAS.

**Electrolytic apparatus [for production of gases].** A. E. KNOWLES (B.P. 354,232, 5.5.30).—The asbestos sleeves, envelopes, or bags enclosing electrodes which are adjacent to the wall of the cell container are made thicker on the side adjacent to the wall than on that facing the opposite electrode.

J. S. G. THOMAS.

**[Soluble] anodes for electroplating.** UDYLYTE PROCESS Co., Assees. of G. DUBERNELL (B.P. 354,939, 30.6.30. U.S., 13.11.29).—Relatively large balls of the metal to be deposited are supplied to a vertical steel helical cage in which the distance between adjacent turns of the helix decreases in a downward direction, and the bottom end of which is tapered and closed.

J. S. G. THOMAS.

**Positive plates for electrical storage batteries.** S. J. WILLIAMS (B.P. 354,318, 7.5.30).

**[Miniature] dry-cell electric battery.** BURGESS BATTERY Co., Assees. of W. B. SCHULTE and J. S. ZOOK (B.P. 354,415, 15.5.30. U.S., 16.5.29).

**Manufacture of sealed evacuated envelopes containing a liquid. [Mercury switches.]** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. R. WALKER (B.P. 354,346, 29.4.30. U.S., 29.4.29).

**Determining viscosity.**—See I. **Treatment of petroleum.**—See II. **Regeneration of sulphides.** **Treatment of leucite etc.** **Ionised O. S from minerals and gases.**—See VII. **Glass yarn.**—See VIII. **Cleaning Fe wires etc.** **Welding electrode.** **Magnetic alloy.** **Oxidic coatings on Al.** **Ni- or Co-plated articles.** **Coated bodies [conductors].** **Floating metal powders.** **Cr-plating.** **Ni diaphragm.** **Zn on Fe or steel.**—See X. **Medicinal products from yeast.**—See XVIII.

#### XII.—FATS; OILS; WAXES.

**Cacao butter.** VII. **Attempts to distinguish between expressed and extracted cacao butters.** H. P. KAUFMANN (Chem. Umschau, 1931, 38, 241—243; cf. B., 1931, 123).—The ultra-violet absorption spectra afford no distinction between the expressed and extracted fats: both (in pentane solution) show a more or less well-marked inflexion in the absorption curve at λ 2650—2750 Å.

E. LEWKOWITSCH.

**Reichert, Polenske, and Kirschner values of rancid butters and margarines.** G. D. ELSDON, R. J. TAYLOR, and P. SMITH (Analyst, 1931, 56, 515—518).—Mixtures of palm-kernel oil and oleomargarine, of coconut oil, palm-kernel oil, and oleomargarine, and of palm-kernel oil, butter fat, and oleomargarine give an increased Reichert val. on keeping, which corresponds with the amount of butter fat present. The Polenske val. increases slightly and, although the Kirschner val. increases, there is no obvious relationship to the constituents. Butter fat, however, when stored alone or as butter, shows these changes only to a very slight extent. The changes may be biological and caused by the presence of different organisms in various types of products. T. McLACHLAN.

**Physico-chemical methods for determining the purity of fatty acids.** E. L. LEDERER (Chem. Umschau, 1931, 38, 243—246).—The limits are discussed within which impurities are revealed by the usual chemical and physical characteristics; more accurate results are anticipated from determinations of m.p. by dilatometric methods, of the protective action on Au sols, and temp.-viscosity measurements. E. LEWKOWITSCH.

**Analysis of sulphonated oils. I. [Report of a Committee of Leather Trades Chemists.]** D. BURTON and G. F. ROBERTSHAW (J. Soc. Leather Trades' Chem., 1931, 15, 308—328).—Methods are given for sampling and analysing sulphonated oils including determinations of  $H_2O$ , unsaponifiable matter, acidity, ash, total alkali, organically combined  $SO_3$ , neutral combined  $SO_4$ , combined and free fatty acids, free fatty acids, and  $NH_3$ . D. WOODROFFE.

**Erratum.**—On page 551, col. 1, line 22 from bottom, for "Calcutta" read "La Plata."

**Extraction apparatus for fats.**—See I. Volatile products in cottonseed meats. Oil in canned sardines.—See XIX. *Monarda punctata*.—See XX.

See also A., Sept., 1033, Soya-bean lecithins. 1034, Separation of glycerides. 1035, Determination of OH in castor oil. 1067, Esters of capsanthin. 1101, Nutmeg oil. Pæony seed oil. Brazil-nut oil.

#### PATENTS.

**Degreasing apparatus.** M. DE W. HIRST, ASST. to L. A. GOODWIN (U.S.P. 1,795,170, 3.3.31. Appl., 17.9.27).—Plant for recovery of used solvent from dry-cleaning of textiles, wool, etc., including removal of the liquid from the fabrics, is described.

E. LEWKOWITSCH.

**Preservation of soaps, fats, and fatty oils.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 355,055, 10.9.30. U.S., 10.9.29).—Small amounts of a hydroxylated diaryl compound, in which the aryl residues are directly linked, e.g., dihydroxydiphenyl, hydroxylated phenyl-naphthyl, are added. E. LEWKOWITSCH.

**Manufacture of derivatives of higher fatty acids containing nitrogen.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 355,139. 21.11.30. Addn. to B.P. 339,675; B., 1931, 355).—Halogenated derivatives of amido-compounds of higher saturated or unsaturated fatty acids, or hydroxy-fatty acids (e.g., oleic acid),

are treated with solutions of  $NH_3$  in org. solvents, as in the prior patent. E. LEWKOWITSCH.

**Manufacture of modified [mineral oil-soluble] castor oil.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,783, 9.5.30. Cf. B.P. 317,391 and 321,724; B., 1930, 826, 652).—Castor oil (alone or admixed with mineral oil) is heated, preferably in vac. or in a current of inert gas, in the presence of  $H_3PO_4$  and/or its esters (e.g., of BuOH, cresol) at 200—260° until it becomes sol. in mineral oil. Further removal of  $H_2O$  (up to 3 mol. proportions) by prolonged heating furnishes a drying oil. E. LEWKOWITSCH.

**Production of drying oil [from pine oil].** I. W. HUMPHREY, ASST. to HERCULES POWDER CO. (U.S.P. 1,793,220, 17.2.31. Appl., 15.3.27).—A polymerised terpene product (from alcohols or hydrocarbons) is distilled so as to remove the polymerides boiling below 335° [ $(C_{10}H_{16})_2$ ]. The residue (which may be further fractionated or blown with air) dries to a film insol. in gasoline. E. LEWKOWITSCH.

**Treatment of fish-liver oils and application of the products thereof.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. A. SEXTON, and A. STEWART (B.P. 354,417, 17.5.30).—Squalene, or an oil rich in it, is treated with conc.  $H_2SO_4$  or oleum at temps. below 60° (preferably < 30°) in the presence or absence of, e.g.,  $Ac_2O$ , salted out, and washed; the product is used as an emulsifying agent. E. LEWKOWITSCH.

**Deresinification of waxes containing alcohols of high mol. wt. or of alcohols obtained therefrom.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,334, 3.3.30).—The waxes etc. are treated at room temp. with a mixture of two org. solvents, one of which can homogeneously dissolve waxes, e.g.,  $C_6H_6$ , hydrocarbons (in amount insufficient to dissolve the wax completely in the cold), whilst the other can dissolve waxes dispersely, e.g., alcohols, esters. E. LEWKOWITSCH.

**Evaporator [for glycerin].**—See I. Gelatinised oils. Lubricants.—See II. Treatment of textiles.—See VI.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Philippine rosin from *Pinus insularis* (Endlicher).** I. DE SANTOS, A. P. WEST, and J. FONTANOZA (Philippine J. Sci., 1931, 45, 383—390).—*P. insularis*, Endl. (Benguet pine), grown in the Philippines, yields slightly more resin than do Florida pines. An average sample of the resin contains turpentine 16.34%, rosin 83.66%, and typical samples of the latter have characteristics within the following limits:  $d_{1.033}$ —1.072, softening point 55—105°, m.p. 62—119°, acid val. (aq. alkali) 148.9—165.3, sap. val. 153.0—189.9, ester val. 1.0—25.4, unsaponifiable matter 4.35—6.82%,  $[\alpha]_D^{20}$ —26.95° to —64.57°. The Na salt of the rosin acids yields about 74% of abietic acid, m.p. 152—153.8°, which has  $[\alpha]_D^{20}$  varying from —3° to —82.54° according to the solvent used. E. H. SHARPLES.

See also A., Sept., 1021, Mo-blue. 1081, Blue pigment, *Haiiotis*-indigo. 1099, Pigment of the elderberry. Anthocyanin pigments. 1101, Black pigments of *Sarothamnus scoparius*.

## PATENTS.

**Manufacture of paints and the like.** I. G. FARBENIND. A.-G. (B.P. 353,319, 23.9.30. Ger., 23.9.29).—A monoacylalkylenediamine, an acylpolyalkylenepolyamine (e.g., mono-oleyltriethylenetetramine), or an alkylation product thereof including the quaternary  $\text{NH}_4$  bases, is incorporated with pigment and/or binding agent in the prep. of ground pigment pastes.

S. S. WOOLF.

**Manufacture of zinc white.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 354,794, 14.5.30).—NaOH solution is added to  $\text{ZnCl}_2$  lye until the liquor has  $p_{\text{H}}$  12—12.8, and the ppt. is washed and dried below redness. Alternatively, the  $\text{Zn(OH)}_2$  is precipitated with milk of lime and, after washing, is suspended in NaOH solution of such concentration that the liquor has  $p_{\text{H}}$  12—12.8.

L. A. COLES.

**Reducing the oil absorption of titanium dioxide pigment.** SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 354,799, 17.2.30. U.S., 16.2.29).—The pigment, after calcination and wet grinding, is dried and ground.

L. A. COLES.

**Coating of surfaces.** BRIT. THOMSON-HOUSTON CO., LTD., ASSES. of R. H. KIENLE and L. V. ADAMS (B.P. 354,544, 1.8.30. U.S., 10.8.29).—A resin that can be rendered infusible by heat-treatment is powdered and sprayed through a flame, where it is partially and superficially melted, on to the article, which is then baked to produce an even, adherent film. E. LEWKOWITSCH.

**Coating materials.** M. DESENISS and A. NIELSEN (B.P. 353,311, 15.9.30).—Mixtures of chlorinated rubber and tar, pitch, asphalt, or other bituminous substances, dissolved in a suitable solvent, e.g., solvent benzene, and incorporated, if desired, with fillers, e.g., finely-ground asbestos, are claimed.

S. S. WOOLF.

**Production of water-resistant coatings.** L. KIRSCHBRAUN, ASSR. to FLINTKOTE Co. (U.S.P. 1,794,522, 3.3.31. Appl., 23.9.27).—The surface is coated with an aq. dispersion of bitumen etc. (suspended with clay etc.), and, after drying, a coating of an aq. slurry of hydraulic (Portland) cement, containing a small amount of bitumen, with or without, e.g.,  $\text{CaCl}_2$  to accelerate hardening, is applied.

E. LEWKOWITSCH.

**Non-inflammable nitrocellulose lacquer.** M. THAU (U.S.P. 1,793,726, 24.2.31. Appl., 21.4.25).—Sufficient  $\text{CCl}_4$  is added to a solution of nitrocellulose in  $\text{BuOAc}$  and/or  $\text{Et}$  lactate (i.e., in a liquid of high flash point and solvent power, the vapour of which has a sp. gr. not much above that of  $\text{CCl}_4$ , whilst the vapour tension of the mixed solvent(s) and  $\text{CCl}_4$  is scarcely greater than that of  $\text{CCl}_4$  alone). E. LEWKOWITSCH.

**Liquid polish.** A. S. FINTER (B.P. 354,487, 23.6.30).—A coloured mixture of oil varnish and petroleum spirit or other light hydrocarbon is claimed.

H. ROYAL-DAWSON.

**Treatment of rosin.** F. W. KRESSMAN (B.P. 354,332, 10.2.30. U.S., 11.2.29).—Rosin is batch-distilled at low temps. and pressure (at not above  $260^\circ/10$  mm.) and fractionally condensed; resenes pass into the first fractions (approx. 12%), whilst the still residues are enriched in abietic acid. Volatile oils,  $\text{H}_2\text{O}$ , etc. are removed by preheating at lower temps.

E. LEWKOWITSCH.

**Manufacture of synthetic resin.** E. E. NOVOTNY, ASSR. to J. S. STOKES (U.S.P. 1,793,715, 24.2.31. Appl., 31.7.26).—Xylenols are heated with furfuraldehyde at ordinary pressure at  $182$ — $193.5^\circ$  and the  $\text{H}_2\text{O}$  is continuously removed from the condensed vapours by a gravity separator, the reactive ingredients being returned to the digester. The reactive product is preferably combined with a hardening agent, e.g.,  $\text{CH}_2\text{O}$ .

E. LEWKOWITSCH.

**Synthetic resins from chlorinated toluol and phenol.** C. ELLIS, ASSR. to ELLIS-FOSTER Co. (U.S.P. 1,793,310—2, 17.2.31. Appl., [A] 23.3.23, [B] 12.5.24, [C] 23.10.24).—The dark red, syrupy, resinous material obtained by treating phenol (3 mols.) with crude benzotrichloride (1 mol.) in alcoholic solution in the presence of  $\text{AlCl}_3$  or  $\text{FeCl}_3$ , or the resin (X) obtained by combining it with  $\text{CH}_2\text{O}$  (10%) may be cold-moulded (with fillers) and hardened by baking. (b) Hexamethylenetetramine is incorporated with the product X. (c) The material is acidified with  $\text{HCl}$  (up to 1%) before combination with  $\text{CH}_2\text{O}$ .

E. LEWKOWITSCH.

**Manufacture of (A) plastic materials, (B) materials having a basis of cellulose derivatives.** H. DREYFUS (B.P. 354,860—1, 20.5.30).—(A) The finely-divided cellulose ester(s) containing little or no solvent is impregnated with a plasticiser (e.g., toluenesulphonamide, alkyl phthalates) in a volatile liquid vehicle (e.g.,  $\text{Et}_2\text{O}$ ) which is not a solvent for or sol. in the cellulose derivative, but is evaporated before the material is worked. (b) The plasticiser is applied uniformly by spraying on to the finely-divided cellulose ester (preferably itself as a spray) substantially in the absence of solvents or diluents; solid plasticisers may be used if dissolved in a highly volatile solvent which evaporates before contact with the ester.

E. LEWKOWITSCH.

**Manufacture of plastic substances.** J. C. PATRICK (B.P. 354,310 and 354,394, 2.5.30. U.S., [A] 3.6.29, [B] 10.6.29. Cf. B.P. 302,270; B., 1929, 826).—Hard thermoplastic materials are prepared by treating olefine (dihalides with aq. solutions of (Na) polysulphides (A) containing an at. proportion of S between 1.5 and 3.75, (B) in the presence of 1—5% of finely-divided oxides, hydroxides, or carbonates of Ca etc.  $\text{EtOH}$  may be added to increase the solubility of the halide. [(A) Stat. ref.]

E. LEWKOWITSCH.

**[Plastic] fibrous compositions.** H. WADE. From BAKELITE CORP. (B.P. 353,070, 17.4.30).—Mouldable compositions comprising a loose fibrous material containing fibres long enough to overlap and stiff enough to avoid knotting and tangling (either naturally stiff, e.g., sisal, or a soft fibre spun or twisted into threads, cords, etc.) together with a resinoid bonding agent are claimed. The latter may be applied as a varnish with subsequent removal of solvent, or part, e.g., 10%, may be applied in this manner and the remainder incorporated as dry powder.

S. S. WOOLF.

**Treatment [increasing the elasticity] of objects from plastic masses.** C. F. BOEHRINGER & SOEHNE, G.M.B.H. (B.P. 354,957, 11.7.30. Ger., 18.7.29).—Objects, e.g., billiard balls, prepared from masses containing cellulose derivatives and a plasticiser, e.g., acetethylanilide, are treated with a solvent for the plasti-

ciser, *e.g.*,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , to remove it from the surface layer. L. A. COLES.

**Manufacture of sealing wax in a more or less finely-distributed form.** W. HOCHGESAND (B.P. 354,576, 29.8.30).—The wax drips from a sieve (or other suitable devices) into a cooling liquid (*e.g.*,  $\text{H}_2\text{O}$ ), kept at a const. temp. and in which the wax is insol.

H. ROYAL-DAWSON.

**Manufacture of ornamental sheets, veneers, and the like from thermoplastic materials.** CELLULOID CORP. (B.P. 354,834, 16.5.30. U.S., 16.5.29).

**Mixing mill. Revivifying spent adsorbent. Materials for bearings.**—See I. **Resins from indene and phenols.**—See III. **Solution of cellulose derivatives.**—See V. **Arc-resisting composition.**—See XI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Microturbidimeter for determination of rubber content of latex.** S. D. GELMAN and J. S. WARD (Ind. Eng. Chem. [Anal.], 1931, 3, 300—304).—A modification of the microturbidimeter of Conklin (B., 1925, 571), based on the obscuring of an incandescent filament, has been applied to the rapid determination of the % rubber in *Hevea* latex. The time required is less than 5 min. and the accuracy is approx. 1% in 35% latex. The turbidity of  $\text{H}_2\text{O}$ -diluted latex obeys the turbidity-dilution law up to a proportion of 15% of rubber.

D. F. TWISS.

**Cause of variability in the plasticity of plantation rubber after storage.** G. MARTIN and L. E. ELLIOTT (Trop. Agric., 1931, 76, 342—344).—Storage of crêpe and sheet rubber in an atm. of  $\text{N}_2$  causes hardening which increases with rising temp. In an atm. of  $\text{O}_2$  rubber is always softer than in  $\text{N}_2$  at the same temp., the difference increasing with temp. Storage changes in the plasticity of rubber result from (1) spontaneous hardening such as occurs in an inert atm., and (2) softening induced by  $\text{O}_2$ . At low temps. (1) predominates, whereas (2) is greater at higher temps.; there is a crit. temp. at approx. 30°. Rubber extracted with aq.  $\text{NH}_3$  becomes very soft in  $\text{O}_2$  at 45° although hardened at 15°, but unextracted samples tend to harden even at 45°. Extraction with  $\text{H}_2\text{O}$  causes softening in  $\text{O}_2$  at 45°, but to a smaller extent than that caused by  $\text{NH}_3$  treatment.

A. G. POLLARD.

**Scorching and other plasticity changes in rubber compounds on heating.** E. O. DIETERICH and J. M. DAVIES (Ind. Eng. Chem. [Anal.], 1931, 3, 297—300).—The plastometer described recently (B., 1931, 351) is applied to testing samples of rubber mixtures which have been heated for various periods. The rate of development of the initial stages of vulcanisation thus revealed is a measure of the tendency to scorching and is used for estimating safe operating temps. and periods.

D. F. TWISS.

**Autographic machine for testing tensile properties of rubber.** G. J. ALBERTONI (Ind. Eng. Chem. [Anal.], 1931, 3, 236—239).—A specially designed machine of the pendulum type records the stress-strain curve of deformable material such as rubber; no calculations are required and the records can be used

directly, *e.g.*, for measurement of the area under the curve. Mechanical means are also provided to correct for variation in the cross-section of the test-piece and to produce equal chart displacements for equal angular deviations of the pendulum.

D. F. TWISS.

**Extraction apparatus for rubber.**—See I.

See also A., Sept., 1967, **Isoprene and caoutchouc.**

#### PATENTS.

**Manufacture of rubber-like masses.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,976, 5.3.30).—Butadienes, with or without cyclic diolefines or olefines having a benzene ring in the mol., are polymerised in the presence of an unsaturated ketone,  $\text{R}\cdot\text{CO}\cdot\text{CR}'\cdot\text{CR}''\cdot\text{R}'''$  ( $\text{R}$  = aryl or alkyl, and  $\text{R}'$ ,  $\text{R}''$  and  $\text{R}'''$  = H or hydrocarbon radicals). Mixtures of butadiene with  $\alpha$ -isopropenyl Me ketone and oleic *p*- $\beta$ -diethylaminoethoxyanilide hydrochloride or Na and Mg oleates, or with styryl Me ketone and oleic *p*- $\beta$ -diethylaminoethoxyanilide hydrochloride with or without  $\alpha$ -isopropenyl Me ketone, give rubber-like products when heated for several days at 60°.

C. HOLLINS.

**Manufacture of india-rubber-like products.** G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 353,194, 19.6.30).—Polymerised vinyl derivatives are incorporated with unvulcanised indiarubber and the mixture is vulcanised.

S. S. WOLF.

**Vulcanisation of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 350,161, 29.4.30).—2:4-Dinitro-1-naphthyl 2-benzthiazolyl sulphide, m.p. 144°, is a vulcanisation accelerator showing delayed action, especially in conjunction with diarylguanidines.

C. HOLLINS.

**[Preservation of] oxidisable organic compounds [e.g., rubber or transformer oils].** GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 347,916, 23.5.30. U.S., 27.7.29).—See U.S.P. 1,779,390; B., 1931, 579.

**Mixing of rubber.**—See I. **[Rubber-]coated bodies.**—See X. **Insulating materials.**—See XI. **Coating materials.**—See XIII.

#### XV.—LEATHER; GLUE.

**Microscopical study of the effect of follicular mange on skins, hides, and leather.** F. O'FLAHERTY and W. RODDY (J. Amer. Leather Chem. Assoc., 1931, 26, 394—403).—Photomicrographs of follicular mange in raw skins and leather and of damages caused by them are given. No evidence is adduced to indicate that the mange can spread to other skins in storage of raw skins.

D. WOODROFFE.

**Use of reflected light in microscopical studies of leather.** A. KÜNTZEL (Collegium, 1931, 381—391).—Several applications of the use of such light in the study of leather are described and illustrated.

D. WOODROFFE.

**Dung bates.** H. GÖLLER (Collegium, 1931, 334—339).—Very poor bating effects were produced on skin by fresh dog puer alone; good bating was obtained

with fresh puer and a little deliming agent. The skin had a smoother feel and was rendered more flaccid by the use of more deliming agent, but the fibres were not properly separated. The skin was rendered very flaccid and the fibres were well separated by the use of an excess of fresh puer alone, but the bating was incomplete. Even with sufficient deliming agent the bating was insufficient after the use of old fermented puer. An excess of old puer was required to effect complete bating even in presence of the necessary amount of deliming agent, which confirms general practice.

D. WOODROFFE.

**Causes of and remedies for faults arising during the curing [of raw hides].** W. SCHINDLER (Collegium, 1931, 358—365).—Recent publications on this subject are summarised.

D. WOODROFFE.

**Liming and depilation of animal skin.** E. R. THEIS (J. Amer. Leather Chem. Assoc., 1931, 26, 352—388).—The time of unhairing was greatly diminished by additions of NaCN or Na<sub>2</sub>SO<sub>3</sub> and increased by cane sugar or aq. NH<sub>3</sub>, but not much affected by additions of NaOH or KOH to a lime liquor. The degree of unhairing increased as the  $p_H$  of the lime liquor was increased. Two stages of unhairing were observed in most lime liquors. The first step was expedited by additions of Sn and Fe salts to the Ca(OH)<sub>2</sub>, and the hair removal was expedited by additions of NaNO<sub>3</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>SO<sub>3</sub>, or NaCN. Min. time for unhairing was obtained by using *M*-NaCN and *M*-Ca(OH)<sub>2</sub>. Each liquor had an optimum  $p_H$  for unhairing. Greater swelling and hydration were shown by the epidermal layer than by the corium and much greater hydration by old hair. Skin became more hydrated during the liming process. The hydration was increased by (a) changing the lime liquor each day, (b) stirring the liquors, (c) additions of Na<sub>2</sub>S, NH<sub>2</sub>Me, or NH<sub>3</sub>, and retarded by Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, or Ba(OH)<sub>2</sub>. Greater hydration and greater swelling were obtained by liming skin with Ca(OH)<sub>2</sub>-Na<sub>2</sub>S or Ca(OH)<sub>2</sub>-Na<sub>2</sub>S-NaCl than by Ca(OH)<sub>2</sub> alone. Greater hydration, but less swelling, was obtained in a mellow lime liquor than in a sharp one. 70% of the N in a lime liquor was derived from the epidermis. The N derived from the true skin decreased as the Na<sub>2</sub>S concentration increased. The N distribution was changed as the rate of unhairing was altered. Less N was dissolved in Ca(OH)<sub>2</sub>-Na<sub>2</sub>SO<sub>3</sub> liquors than in Ca(OH)<sub>2</sub> alone. Photomicrographs of sections of pelt limed in different liquors are appended.

D. WOODROFFE.

**Determination of amines in tannery lime liquors.** E. K. MOORE, J. H. HIGHBERGER, R. KOPPENHOEFFER, and F. O'FLAHERTY (J. Amer. Leather Chem. Assoc., 1931, 26, 341—351).—A modification of Weber and Wilson's method (B., 1918, 638 A) is described. The "total volatile alkali," *A*, is determined by distilling a measured vol. of the settled lime liquor into 0.2*N*-H<sub>2</sub>SO<sub>4</sub>. The titrated distillate is diluted, rendered alkaline, well shaken with yellow HgO in a brown glass bottle, filtered out of contact with air through cotton, and the filtrate distilled into standard H<sub>2</sub>SO<sub>4</sub>; the "total volatile amines," *B*, is calc. therefrom. Then NH<sub>3</sub> = *A* - *B*. A second portion of the titrated

distillate is acidified with conc. H<sub>2</sub>SO<sub>4</sub>, evaporated down, cooled, mixed with saturated NaNO<sub>3</sub> and glacial AcOH, left for 45 min., well shaken, the NO expelled with a current of air, and the mixture rendered alkaline is distilled into 0.1*N*-H<sub>2</sub>SO<sub>4</sub> and (*C*) the NMe<sub>3</sub> calc. therefrom. Zn and conc. HCl are added to this titrated distillate, which is then heated for 10 min., the solution decanted into a Kjeldahl flask, the Zn washed thrice with H<sub>2</sub>O, NaOH is added, and the whole distilled into 0.1*N*-H<sub>2</sub>SO<sub>4</sub> and (*D*) the NHMe<sub>2</sub> and NMe<sub>3</sub> are calc. therefrom. Then NHMe<sub>2</sub> = *D* - *C*; and NH<sub>2</sub>Me = *B* - *D*.

D. WOODROFFE.

**Moisture in vegetable-tanned leather.** R. F. INNES and J. G. M. COSTE (J. Soc. Leather Trades' Chem., 1931, 15, 302—303).—A reply to Chambard's criticism (B., 1931, 771) that moisture in leather can be determined by heating it to const. wt.

D. WOODROFFE.

**Determination of strong acid in vegetable tanned leather.** R. F. INNES (J. Soc. Leather Trades' Chem., 1931, 15, 303—308).—Leathers can be divided into four groups according to their "difference figure" (B., 1929, 729) (D.F.) and the  $p_H$  of their aq. extract: (*A*) D.F. 0.7 or over,  $p_H$  < 3.0; (*B*) 0.7 or over, > 3.0; (*C*) < 0.7, > 3.0; (*D*) < 0.7, < 3.0. Large amounts of weak acids are present in leathers in group *D*. About 0.35% of free H<sub>2</sub>SO<sub>4</sub> is present in leathers of group *B*. Strong acids are present to a harmful extent in leathers of group *A* if the  $p_H$  of the aq. extract is 2.9 or less; if 2.9—3.0 the leather is open to suspicion. Not more than 0.45% of free H<sub>2</sub>SO<sub>4</sub> can be present in a leather the aq. extract from which has  $p_H$  2.9.

D. WOODROFFE.

**Tannin analysis. Report of a Committee [of the French section of the Society of Leather Trades' Chemists].** P. VIGNON (J. Soc. Leather Trades' Chem., 1931, 15, 360—367).—Good results were obtained by the Riess method, but no advantage was shown over the official method. The Schmidt method required too long. The sedimentation method was too indefinite to draw any conclusions. Poor concordance was obtained by all filter-paper methods, but good concordance by the filter candle. Difficulty was experienced with dry American chestnut extract and pine-bark extract owing to their content of larger particles of insol. matter. The Darmstadt apparatus for detannisation proved inconvenient and the Freiberg-Keigueloukis apparatus was impracticable. The Jamet apparatus was more rapid for one or two analyses. It is proposed that the filtration by the Berkefeld filter candle should be an official method, and that no modifications which do not improve the concordance obtained should be made in any official method.

D. WOODROFFE.

**Extraction of raw tanning materials for analysis. Report of Committee—1931 [of the American Leather Chemists' Association].** W. K. ALSOP (J. Amer. Leather Chem. Assoc., 1931, 26, 403—431).—Preliminary comparative tests on the official method of the A.L.C.A. and the provisional International method of extracting tanning materials for analysis indicate that different methods will be required for different tanning materials.

D. WOODROFFE.

**Presence of traces of heavy metals in tannin extracts.** P. VIGNON (J. Soc. Leather Trades' Chem., 1931, 15, 385—387).—Tannates of heavy metals are peptised by the tannin in a tannin solution and remain in colloidal solution. Alteration in colour of the solution can be detected by the tintometer and confirmed by a tanning test. A greyish tone is imparted to leather by minute quantities of Fe which are not detected by the tintometer. Neither the colour of the extract nor of the leather tanned with it were affected by  $< 0.06\%$  Cu in a chestnut extract. D. WOODROFFE.

**Tanning with tara (*Caesalpinia tinctoria*) and its production in Italy and Italian colonies.** F. VIGNOLO-LUTATI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 297—303).—Complaints by Indian tanners that putrid fermentations occur during tanning with *Caesalpinia* pods probably result from the use of *C. digyna* (tari) or Indian divi-divi and not of *C. tinctoria* (tara). Small-scale experiments showed that extract of tara pods (freed from seed) from Cagliari exhibits mould growth when kept for some time, but that it is not inferior to other similar extracts in this respect; no putrid fermentation was observed. The possibility of cultivating this material in Erythraea is discussed. T. H. POPE.

**Formation of gases in tan liquors.** II. MACHON (Collegium, 1931, 392—396).—Most of the gases formed in vegetable tan liquors during tannage consist of  $H_2S$  arising from the use of sharpened CaO liquors on the pelts. The amount increases as the quantity of  $Na_2S$  used increases, and diminishes as the amount of deliming is increased.  $CO_2$  is present in old fermenting tan liquors and much less  $H_2S$  is present in them than in fresh liquors. Gases are formed also by the decomp. of tannins with micro-organisms with the accompanying formation of insol. matter, e.g., untreated quebracho extract. D. WOODROFFE.

**Gas-meter leathers.**—See II. Egg yolk.—See XIX.

See also A., Sept., 1094, Tannin-decomposing micro-organisms.

#### PATENTS.

**Manufacture of leather for coating rollers of a spinning machine.** C. NITTA (B.P. 353,098, 29.4.30).—Tanned skins are drummed with rice bran and water for 1 hr., washed, dried, the grain surface is buffed and calendered, and the skin shaved to level substance. D. WOODROFFE.

**Wetting etc. agents.**—See III. Waterproofing of textiles.—See VI. Gelatin-composition container.—See XXIII.

#### XVI.—AGRICULTURE.

**Soils of the Nile and Gash.** II. P. VAGELER and F. ALTEN (Z. Pflanz. Düng., 1931, 21A, 323—346; cf. B., 1931, 772).—A discussion of earlier results from the view point of soil- $H_2O$  relationships. A. G. POLLARD.

**Biodynamics of alkali soils.** M. I. PRIKHODKO and M. I. BELIKOVA (Pochvovedenie, 1929, 24, No. 3—4, 145—166).—On soils in the open field NaCl hindered the activity of *Azotobacter*, whilst  $Na_2SO_4$  stimulated

their multiplication, and the amount of N fixed was as great as in the controls. *Clostridium pasteurianum* developed better on the NaCl soil. Anaërobic and aërobic decomp. of cellulose were stimulated by treating the soil with NaCl. Nitrification was stimulated in the soils treated with  $Na_2SO_4$ , but not in those treated with NaCl. The alkalinity of the soils was increased by treatment with NaCl and subsequent leaching by rain water. CHEMICAL ABSTRACTS.

**Correlation between plant communities and the reaction and microflora of the soil in S. Central Texas.** M. B. MORROW (Ecology, 1931, 12, 497—507).—The relation between plant cover and soil reaction is definite and significant to soil depths of 12 in. The relative numbers of the various groups of soil micro-organisms do not vary definitely with soil reaction, but are more closely related to moisture content and aeration. A. G. POLLARD.

**Coagulation of aqueous suspensions of soil with barium sulphide and calcium sulphide.** B. G. ZAPROMETOV (Pochvovedenie, 1929, 24, No. 3—4, 37—42).— $BaS$ ,  $CaS$ ,  $BaCl_2$ , and  $CaCl_2$  are effective as coagulants for mastic; the first pair only are absorbed by the mastic. CHEMICAL ABSTRACTS.

**Influence of calcium sulphide on percolation of water through soil.** A. N. ROZANOV (Pochvovedenie, 1929, 24, No. 3—4, 31—35).— $CaS$  is more effective as a coagulating agent than  $CaSO_4 \cdot 2H_2O$ ; it decreases the alkalinity of the soil. CHEMICAL ABSTRACTS.

**Can a water extract serve as an index to characterise the degree of salt treatment of soils?** D. I. TARASOV (Pochvovedenie, 1929, 24, No. 3—4, 61—99).— $H_2O$  extracts all the sol. salts; hence the chlorides and sulphates are not in an adsorbed state. CHEMICAL ABSTRACTS.

**Rôle of peat in increasing absorption capacity and buffer properties of soils.** D. V. DRUZHININ (Udobr. Urozhai, 1930, 2, 645—650).—The effect is especially important in connexion with physiologically acid fertilisers. CHEMICAL ABSTRACTS.

**Optical method for investigation of humus.** G. I. POKROVSKI (Pochvovedenie, 1929, 24, No. 1—2, 124—130).—A photometric method for the determination of humus is described. CHEMICAL ABSTRACTS.

**Neutral salt decomposition in humus soils.** W. U. BEHRENS (Z. Pflanz. Düng., 1931, 21A, 363—368).—Treatment of washed peat with neutral salt solutions results in a true salt decomp., direct analytical vals. being confirmed by titration of the free acid produced. A. G. POLLARD.

**Lime studies.** The " $p_H$  span" as a function of the buffer power of soils, and a new manner of buffering. S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1931, 21A, 346—363; cf. B., 1931, 897).—The difference between the  $p_H$  of soil in aq. suspension and in KCl suspension (" $p_H$  span") is related to the buffer power of the soil. The buffer power of a soil in neutral salt suspension exceeds that in aq. suspension. The  $p_H$  span is a function of the increased area of the base-fixing zone of the soil-titration curve produced by

the action of neutral salts. This neutral salt action involves a reduction of the base area of the titration curve and of the  $p_H$ , without actual loss of base. The quantity of base necessary to satisfy the  $p_H$  span may be determined electrometrically and is greater in soils containing relatively high proportions of mineral colloids. The org. colloids are less affected by neutral salts. Manuring of soils with neutral salts reduces the  $p_H$  span by lowering the  $p_H$  in  $H_2O$ . A. G. POLLARD.

**Determination of lime requirement by hydrolytic acidity of soil.** S. S. YARUSOV (Udobr. Urozhai, 1930, 2, 638—642).—From the equation  $[H^+] = K[AcOH]/[NaOAc]$  a series of equations is deduced from the reactions which take place when a soil is treated with NaOAc for the determination of hydrolytic acidity in terms of  $Ca^{++}$ . CHEMICAL ABSTRACTS.

**[Determination of] lime requirement of soil.** V. M. FILIPENYA (Udobr. Urozhai, 1930, 2, 642—645).—The  $CaO$  requirement is calc. from the hydrolytic acidity (NaOAc method); the coeff. 2.0 is used. CHEMICAL ABSTRACTS.

**[Determination of] replaceable sodium in soils.** K. K. GEDROIZ (Pochvovedenie, 1929, 24, No. 1—2, 1—14).—The soil (10 g. if  $< 0.1\%$ ; 100 g. if  $> 0.1\%$ ) is mixed with  $CaCO_3$  (10%) and  $H_2O$  (1 litre), and  $CO_2$  is passed for 2—3 hr. The Na is determined in the filtrate. If sol. Na salts are present they are washed out before the soil is treated with the  $CaCO_3$  and  $CO_2$ . CHEMICAL ABSTRACTS.

$p_H$  determinations [of soils] in water and in potassium chloride solution and the effect of the alkaline earths, especially magnesium. II. ELLEDER (Z. Pflanz. Düng., 1931, 21A, 368—378).—Differences in the  $p_H$  of soils in  $H_2O$  and in KCl suspension vary with the total ( $CaCO_3 + MgCO_3$ ) content and probably with the ratio  $CaCO_3 : MgCO_3$  which exists therein. A. G. POLLARD.

**Fineness of grinding of phosphates for podsolised soils.** N. D. SMIROV (Udobr. Urozhai, 1930, 2, 765—769).—Certain raw phosphates of fineness up to 0.01 mm. gave effects in pot experiments of 75—100% compared with acid phosphate. CHEMICAL ABSTRACTS.

**Influence of various nitrogenous fertilisers on the reaction of soils and on [crop] yields.** K. NEHRING (Z. Pflanz. Düng., 1931, 10B, 361—370).—Changes in soil reaction following applications of N-fertilisers varied with soil type. On a relatively well-buffered soil exhibiting exchange acidity the reaction was not affected until the second application, and then altered only very slightly. With a light sandy soil a definite change in reaction was apparent in the first season. The utilisation of the various fertilisers by crops varied both with soil reaction and with the nature of the crop. On a sandy loam N was most easily assimilated from  $(NH_4)_2SO_4$  by potatoes and less easily by barley and roots. A. G. POLLARD.

**Causes of reduced yields of tubers and of starch in potatoes produced by various potash manures.** I. Technique of fertiliser trials and conclusions from these. TOMZIG and H. PERNICE (Z. Pflanz. Düng., 1931, 10B, 370—395).—The use of kainite and

40% potash salts increased the yield of potato tubers, but the % starch and total starch yield declined. K Mg sulphate ("Kalimagnesia") and  $K_2SO_4$  increased the yield of tubers and the total starch yield, with a slight reduction in % starch content. A. G. POLLARD.

**Effect of organic substances and especially of green manures on the nitrogen and other nutrient contents of soils.** E. MUSSLA (Landw. Versuchs-Stat., 1931, 112, 115—159).—The incorporation of straw, paper, or sawdust with soil resulted in a decreased nitrate content over a period of many months. Addition of peat to soil reduced its nitrate content for about 1 week, but subsequently increased it. In all cases the  $NH_3$  and total N content of the treated soils remained unaltered, the addition of org. matter merely causing a fixation of easily-sol. N. The crop yields on treated soils were directly proportional to the nitrate contents. Green manuring with legumes increased the  $NH_3$  content of the soil for a brief period, and the nitrate content throughout the cropping season. The added N was very largely withdrawn by the succeeding crop. Green manuring increased the root-sol. K content of the soils, but did not affect the proportion of citric-sol. P. The C of the green manure was reduced to approx. 50% within 2 months of ploughing-in, but subsequently remained practically unaltered. The conductivity of green-manured soils was closely related to their nitrate contents. A. G. POLLARD.

**Intensive system of grassland management.** Digestibility and feeding value of (IV) artificially dried grass; (V) grass silage made in a tower, and the digestibility and comparative yield of artificially dried grass obtained from the same source; (VI) grass silage made in a stack; (VII) grass silage made in a pit. S. J. WATSON (J. Agric. Sci., 1931, 21, 414—424, 425—441, 452—457, 469—475; cf. B., 1931, 82).—IV. Artificially dried grass, fed whole or ground to sheep, had a high digestibility. The material has a narrow nutritive ratio and high digestible protein content and is superior to good hay.

V. Highly nutritive silage may be made from grass in a tower silo. Losses by drainage and fermentation are small. Silage may replace the whole of the hay ration for cows without affecting the milk yield. In comparison with artificially dried grass, silage has a slightly lower starch equivalent and digestible crude protein content and a considerably lower proportion of digestible true protein.

VI. Analyses and digestibility values for grass-stack silage are recorded. The process is suited to the conservation of surplus grass, particularly the aftermath.

VII. The digestibilities of the constituents of pit silage made from grass cut at the flowering stage were similar to those of meadow hay, but the digestibility of the protein was lower. On a dry-matter basis the silage had a greater starch equiv. than had hay. A. G. POLLARD.

**Insect injury of blue grass in relation to environment.** L. F. GRABER, C. L. FLUKE, and S. T. DEXTER (Ecology, 1931, 12, 547—566).—Injury by white grubs is associated with deficient soil fertility and shortage



of org. food reserves in the plants. Liberal use of fertilisers reduced the injury in nearly all cases.

A. G. POLLARD.

**Effect of sulphur on citrus trees.** E. A. MCGREGOR (California State Dept. Agric., Mo. Bull., 1931, 20, 167—170).—Dusting of citrus trees with S caused some burning of the fruit but practically no injury to the foliage, only the old leaves being affected.

A. G. POLLARD.

See also A., Sept., 1099, **Influence of KI, KIO<sub>3</sub>, and KIO<sub>4</sub> on germination etc. of cereals.** 1101, **Utilisation of fixed N by root nodules of legumes.** 1102, **K deficiency of fruit trees.**

#### PATENTS.

**Manufacture of fertilisers.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 353,763, 22.9.30. Nor., 28.9.29.).—In the process already described (B., 1931, 201), the  $\text{Ca}(\text{NO}_3)_2$  may be converted into  $\text{KNO}_3$  by means of K salts, part of this  $\text{KNO}_3$  being added to the  $\text{Ca}(\text{NO}_3)_2$  mother-liquor. The mixture is then neutralised with  $\text{NH}_3$  and evaporated to dryness or neutralised during evaporation. Prior to addition of  $\text{KNO}_3$ , the mother-liquor may be freed from Ca by precipitation with a suitable reagent, *e.g.*, HF,  $\text{H}_2\text{SO}_4$ , or fluorides or alkali sulphates.

W. J. WRIGHT.

**Production of [granulated] fertilisers.** W. K. HALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 354,307, 31.3.30.).—A mixture (preferably < 1.5 : 1) of  $\text{H}_2\text{O}$ -insol. ballast material [*e.g.*,  $\text{CaCO}_3$ ,  $\text{Ca}_3(\text{PO}_4)_2$ ,  $\text{CaSO}_4$ ] with a hot, conc. urea solution containing 2–5%  $\text{H}_2\text{O}$  (calc. on the total wt. of dry solids) is broken up at 95–115° into droplets, 0.5–3 mm. in diam., which are solidified by contact with cold gas, *e.g.*, as described in B.P. 309,299 (B., 1929, 448).

L. A. COLES.

**Rendering insoluble phosphates available [as fertilisers].** H. LEHRECKE (U.S.P. 1,795,173, 3.3.31. Appl., 28.1.29. Ger., 26.1.28.).—Mineral phosphates are heated at 1000–1400° with powdered C,  $\text{SiO}_2$ , and, if necessary, CaO, so that for every 1 pt. of  $\text{P}_2\text{O}_5$  in the charge there are present 0.18–0.24 pt. of C, 0.18–0.24 pt. of  $\text{SiO}_2$ , and 0.85–1.13 pt. of CaO. In this operation 25–55% of the  $\text{P}_2\text{O}_5$  is reduced to P and volatilised, and a sintered residue of  $5\text{CaO} \cdot \text{P}_2\text{O}_5 \cdot \text{SiO}_2$  is obtained, which, after fine grinding, forms a valuable fertiliser having practically all its  $\text{P}_2\text{O}_5$  content sol. in citric acid.

A. R. POWELL.

**Powdered deodoriser for the acceleration of ripening of organic fertilisers.** K. YAMAJI (U.S.P. 1,791,918, 10.2.31. Appl., 1.8.29.).—A mixture of bran with powdered cereals containing a large quantity of enzyme (*e.g.*, wheat, barley, bean) is moistened and allowed to ferment, and the product is dried, powdered, and mixed with  $\text{Ca}_3(\text{PO}_4)_2$  and with germinated cereal seeds which have been dried and powdered.

L. A. COLES.

### XVII.—SUGARS; STARCHES; GUMS.

**Effect of scum particles in boiling up and in [sugar] thin-juice evaporation.** J. HAMOUS (Z. Zuckerind. Czechoslov., 1931, 55, 681–682).—When

the thin-juice was filtered only twice, *i.e.*, after the second carbonatation and after boiling up, the heaters were less incrustated than when it was thrice filtered, *i.e.*, after the 1st and 2nd carbonatations and after boiling up. Further, when the usual filtration after the 2nd carbonatation was omitted, so that unfiltered juice went into the boiling-up apparatus, the bodies of the evaporator were distinctly less incrustated. (Cf. B., 1928, 765.)

J. P. OGILVIE.

**Porosity of carbonatation scums.** J. DĚDEK and E. DOSTÁL (Z. Zuckerind. Czechoslov., 1931, 55, 671–681).—Very varying results were obtained when determinations were made of the porosity (*c.e.* of air liberated from 100 g.) of carbonatation scums from Kelly, Sweetland, and Kroog filter-presses. Even the same cake was very unhomogeneous. An increase in the CaO addition in carbonatation considerably diminished the porosity value, *viz.*, from 62.4 with 1.2% of CaO to 43 with 4.0%.

J. P. OGILVIE.

**Wood-sugar problem. II. Saccharification of cellulose by means of sulphuric acid with recovery of the acid.** L. LEONE (Annali Chim. Appl., 1931, 21, 343–352; B., 1928, 619).—Higher yields with greater acid and fuel economy are obtained by the Ost and Wilkening method (A., 1910, i, 364) when the concentration of the acid during cold saccharification is 72% for dry cellulose or 75% for cellulose of 6%  $\text{H}_2\text{O}$  content. The hot digestion is carried out at 102–103° for 30 min. and the acid concentration is 15%. The yield of dextrose is increased by increasing the ratio acid : cellulose, but beyond a certain concentration small increases in yield require great expense of acid. The recovery of the acid by the  $\text{Ca}(\text{OAc})_2$  method followed by esterification of AcOH with EtOH by the process of the Holzverkohlungs-Industrie A.-G. (G.P. 459,604) and the British Celanese, Ltd. (B.P. 284,582; B., 1928, 223) has not given satisfactory results because it is probable that the alcoholic groups of the sugar prevent esterification. The Ca citrate process is also of limited application because secondary products of saccharification prevent the crystallisation of citric acid. A modification of G.P. 305,180 (B., 1920, 203 A), *i.e.*, precipitation as  $(\text{NH}_4)_2\text{SO}_4$  in alcoholic solution, showed that recovery of the acid cannot be effected economically in this way.

O. F. LUBATTI.

See also A., Sept., 1038, **Oxidation of sugars electrolytically.** 1039, **Determination of levulose in mixed carbohydrates.** 1100, **Glucosides.** Plant gums. 1102, **Determination of mixed reducing sugars.**

### XVIII.—FERMENTATION INDUSTRIES.

**Research methods in brewing technology.** F. KUTTER (Woch. Brau., 1931, 48, 321–323).—A discussion of the conditions requisite for analytical methods.

F. E. DAY.

**Analysis of hops.** L. HEINTZ (Woch. Brau., 1931, 48, 315–320, 325–330, 335–338, 369–373).—Hops were extracted successively with light petroleum,  $\text{Et}_2\text{O}$ , EtOH,  $\text{H}_2\text{O}$ , and 5% NaOH, and the composition of the extracts was investigated. Except that light petroleum dissolves no hard resin and but little chlorophyll,

the materials extracted by the first two solvents are similar. In addition to resins and colouring matter, both extract waxes, essential oils, fats, and fatty acids. To obtain complete extraction the samples must be ground. The fats and waxes are separated by their insolubility in EtOH, and the fatty acids by esterification with hot abs. EtOH and extraction by light petroleum from the diluted liquid after addition of sufficient alkali to neutralise the resins. These acids consist chiefly of oleic acid. It is claimed that the EtOH solution of the soft resin gives no ppt. with  $\text{Pb}(\text{OAc})_2$  after removal of the fatty acids. The EtOH and to a less degree the  $\text{H}_2\text{O}$  extracts contain substances giving some of the reactions of tannin, but no true tannin is present. The EtOH extract contains a substance which with time loses its solubility in EtOH and  $\text{H}_2\text{O}$ , and may be identical with the "amorphous matter" described by Brown (B., 1913, 442), and the  $\text{H}_2\text{O}$  extract a substance which gives rise to beer turbidity when added to wort before fermentation. Based on these experiments a titrimetric method for hop analysis is described, in which the sample is extracted successively with light petroleum and  $\text{Et}_2\text{O}$ , the evaporated extracts are heated with abs. EtOH, and an aliquot is titrated with 0.1N-alkali in a comparator to  $p_H$  9.18. A further portion is extracted with  $\text{Et}_2\text{O}$ , the residue dissolved in 94% EtOH, and similarly titrated. In the first two titration portions the oleic acid is esterified and from the difference between their sum and the result of the third titration its amount is calc. (1 c.c. of 0.1N-alkali = 36 mg. of soft, or 27 mg. of hard, resin.)

F. E. DAY.

**Extract differences at mash-tun taps.** G. JAKOB (Woch. Brau., 1931, 48, 330—333, 338—343, 352—355).—The conditions affecting the flow of sparge-liquor through the grains are discussed, and the importance of uniformity of sparging is emphasised. F. E. DAY.

**Purification of alcohol by activated charcoal.** FRITZWEILER and K. R. DIETRICH (Z. Spiritusind., 1931, 54, 209—210).—When 100 g. of 94.4 wt.-% raw spirit containing MeCHO 0.114 g., esters 0.1 g., fusel oil 0.01 g., and furfuraldehyde 0.1 g., is treated with 20 g. of activated charcoal for 6 hr., 0.022 g. of MeCHO, 0.021 g. of esters, and undetermined portions of the fusel oil and furfuraldehyde are absorbed. Similar results are obtained with 45 wt.-% raw spirit. Since these impurities can be removed more completely by rectification, the treatment with charcoal should follow rectification instead of preceding it. Less frequent regeneration of the charcoal would be required, and small amounts of MeCHO produced by oxidation of EtOH on the surface of the charcoal could be eliminated by a second rectification. C. RANKEN.

**Use of barium oxide for alcohol dehydration.** B. L. MEREDITH and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1931, 20, 653—655).—91.90% EtOH (wt./wt.) refluxed with 250 g. of ground BaO (16-mesh) for 2 hr. with mechanical agitation gave 97.5% EtOH, a yield of 98% of the total alcohol originally present. Using more BaO or distilling under diminished pressure reduced the yield (cf. B., 1929, 449).

E. H. SHARPLES.

**Calculation of alcohol from the sp. gr.** J. F. LIVERSEEGE (Analyst, 1931, 56, 529—530).—For calculating the relationship of the different ways of stating alcoholic concentration, it may be assumed that 1 g. of abs. EtOH per 100 c.c. = 0.7940 c.c. of abs. EtOH per 100 c.c. = 0.4533 proof spirit. Any of these figures divided by the sp. gr. at 60°/60° F. will give the wt. of EtOH per 100 g.

T. McLACHLAN.

**Determination of lactic acid in wine.** F. WIRTILE (Chem.-Ztg., 1931, 55, 646—647).—Modifications of the usual procedure are described. E. S. HEDGES.

**Barley. Determining gluten. Feeding value of dreg meal.**—See XIX.

See also A., Sept., 1089, Cryst. amylase. 1090, Cryst. pepsin. 1092, Production of mannitol from dextrose.

## PATENTS.

**Mashing of cereal products.** F. B. DEHN. From CANADIAN RES. CORP., LTD. (B.P. 354,258, 2.5.30).—Malt or a mixture of malt with unmalted cereals is mashed at 37.5—56° with 2.5 times its wt. of  $\text{H}_2\text{O}$ . A portion of the liquid is withdrawn after  $\frac{1}{2}$ —1½ hr. and mixed with the boiled and cooled remainder, and the mixture is kept at 75—80° until the conversion of starch is complete.

C. RANKEN.

**Hydrolysed protein products and method of using the same.** C. N. FREY, ASSR. to STANDARD BRANDS, INC. (U.S.P. 1,794,370, 3.3.31. Appl., 26.9.28).—A protein-containing substance, such as compressed yeast, is hydrolysed by an acid, such as  $\text{H}_2\text{SO}_4$ , and after a portion of the free acid is subsequently neutralised by  $\text{Ca}(\text{OH})_2$  and the ppt. separated the further portion is neutralised by aq.  $\text{NH}_3$  and evaporated to dryness. Its use in making leavened bread is described.

C. RANKEN.

**Production of yeast without alcohol.** E. JELLINEK (B.P. 354,118, 20.10.30. Austr., 22.10.29).—Yeast which is propagated by the discontinuous process in an aerated solution containing sugars and nutrients is separated from the fermenting liquid, and after dilution with  $\text{H}_2\text{O}$  is strongly aerated and the separated alcoholic wort slowly added to it.

C. RANKEN.

**Treatment of yeast and manufacture of medicinal products therefrom.** P. HALL (B.P. 354,371, 8.5.30).—Dried, powdered brewers' yeast, either alone or mixed with dried milk powder, is irradiated with ultra-violet rays and mixed with substances containing Ca and P in assimilable form.

C. RANKEN.

**Manufacture of iodised yeast.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,611, 6.10.30).—Yeast is grown in a carbohydrate mash to which is gradually added 5—100 g. of I per cu. m. of the mash.

C. RANKEN.

**Dehydration of [ethyl] alcohol.** KODAK, LTD., ASSECS. of D. F. OTIMER (B.P. 354,553, 8.8.30. U.S., 29.8.29).—An azeotropic mixture of EtOH,  $\text{H}_2\text{O}$ , and  $\text{C}_2\text{H}_4\text{Cl}_2$  is distilled and the  $\text{C}_2\text{H}_4\text{Cl}_2$  layer of the distillate is returned to the still, whilst the aq. layer is freed from traces of  $\text{C}_2\text{H}_4\text{Cl}_2$  by redistillation and finally rectified.

C. RANKEN.

Retting of fibres.—See V. Ripening of fertilisers.—See XVI.

### XIX.—FOODS.

**Condition of barley after removal of husk by sulphuric acid.** J. GRÜSS (Woch. Brau., 1931, 48, 343—345).—A sample of barley, de-husked by treatment with 45%  $\text{H}_2\text{SO}_4$  for 15 min. at 50°, gave about 98% germination. The nature of the slight damage is discussed.  
F. E. DAY.

**Occurrence and properties of wheat with slimy gluten.** W. KRANZ (Mühlenlab., 1931, 17—19).—"Leimkleber" wheat of poor baking qualities, from which the gluten cannot be obtained by washing the flour in the usual way, has been observed chiefly in samples from Eastern Europe and Jugoslavia. As the 1930 harvest produced much more of this wheat than that of 1929, it appears that meteorological conditions are partly responsible, although the normal bushel-wt. of much of this wheat does not support this. The poor quality is not due to admixture of germinated grain. No abnormalities except lack of elasticity of the gluten are evident. Some samples give an abnormally high yield of moist gluten when the flour is washed, the  $\text{H}_2\text{O}$  content of the gluten being high, and when the latter is kept under  $\text{H}_2\text{O}$  for some hours it breaks up. The ratio of total protein to  $\text{H}_2\text{O}$ -sol. protein is low (5.0—5.5), but normal wheat may show similar values.  
W. J. BOYD.

**Fermentation method for rapid determination of gluten quality of wheat and wheaten flour.** P. PELSSENKE (Mühlenlab., 1931, 1—3).—Wheat is tested in the form of grits, flour in admixture with its own wt. of grits of 4% ash content. A 5-g. sample is made into dough with 0.25 g. of yeast and formed into a ball with smooth surface. The ball is placed in a glass vessel 6 cm. in diam. and 7 cm. high, containing 75 c.c. of  $\text{H}_2\text{O}$ , and the vessel is placed in an incubator with glass doors and kept at 31—33°. The time in min. elapsing between the moment of placing the dough in the vessel and that when the first large piece of the gas-disrupted ball touches the bottom of the vessel is taken as the test val. of the sample. Dough from bad wheat is disrupted quickly, that from good wheat slowly, the limiting test val. between good and bad being 30. Values for samples of various origins are given.  
W. J. BOYD.

**Determination of gluten quality and flour quality.** T. RUEMELE (Mühlenlab., 1931, 3—5).—Of the moist gluten from 20 g. of flour, 2.5 g. are dispersed in 75 c.c. and 2.5 g. in 150 c.c. of 0.02N-lactic acid at 27°, and the viscosities at 20° are measured hourly for 5 hr. in a special viscosimeter. The smaller the difference between the viscosities of the two solutions, the better is the quality of the gluten. The quality val.  $X$  is found from the equation  $X = \eta$  of the 75-c.c. solution/ $\eta$  of the 150-c.c. solution. For a good gluten  $X$  is 1.140—1.300 after 5 hr. The method can be applied to flour directly, using 8—10 g., but the result is comparable only with other results obtained with flour, and not with those obtained when gluten is used.  
W. J. BOYD.

**Application of viscosity measurements to evaluation of gluten quality.** T. RUEMELE (Mühlenlab., 1931, 19—21).—The relation of quality and viscosity of gluten is discussed with special reference to the author's quality val. (cf. preceding abstract).  
W. J. BOYD.

**Water-insoluble and water-soluble fractions of bread.** H. AY and P. RZYMKOWSKI (Pharm. Zentr., 1931, 72, 465—467).—Rye bread, dried at 105° and finely ground, was extracted with 26 times its wt. of  $\text{H}_2\text{O}$ . The insol. residue dried at 75—80° in vac. amounted to 83.6% of the dry bread and contained 6.34% of nitrogenous matter, 0.12% of ash, 1.10% of fat, 1.42% of roughage, 84.92% of starch (including 4.23% of pentose), and 6.10% of residue difficult to hydrolyse. After prolonged inversion with 25%  $\text{HCl}$ , this residue, consisting of 21.49% of nitrogenous matter, 7.29% of roughage, and 0.85% of ash, yielded 35.22% of reducing substance (calc. as dextrose), and 19.89% remaining undissolved. The aq. extract of the bread gave on evaporation to dryness a residue containing 10.72% of ash. It had an odour of caramel, brown colour, sweet taste, and was slightly hygroscopic. After removal of heat-coagulable protein, inversion for 3 hr. with 1%  $\text{H}_2\text{SO}_4$ , and removal of the latter by means of baryta, the liquid was incubated with yeast till no more  $\text{CO}_2$  was evolved. The yeast was filtered off and the liquid evaporated to dryness in vac. The dark brown, hygroscopic residue of pleasing odour like that of bread, and sharp taste (neither acid nor saline), amounted to 46% of the  $\text{H}_2\text{O}$ -sol. extract, or 7.52% of the dry bread. It consisted of 17.99% of ash, 7.20% of nitrogenous materials, 3.95% of pentosans, and 2.95% of other reducing substances (calc. as dextrose), leaving 67.89% of material (5.11% on the dry bread), corresponding to Deininger's "other nitrogen-free extractives" (Diss., Würzburg, 1920), of which he found 5.5% in war bread. It gave the reactions of vegetable gum except that it yielded little pentose on boiling with  $\text{H}_2\text{SO}_4$ .  
W. J. BOYD.

**Value of tapioca flour and sago-pith meal in the nutrition of swine.** H. E. WOODMAN, A. W. M. KITCHIN, and R. E. EVANS (J. Agric. Sci., 1931, 21, 526—546).—Tapioca flour forms a highly digestible foodstuff for pigs and has a favourable effect on the colour and texture of the carcass fat and the quality of the bacon. It may replace barley or maize meal in pig rations. Sago-pith meal may be substituted for barley meal up to 20% of the ration without affecting live-wt. increases. Inferior results obtained with higher proportions of the meal are probably due to its depressing influence on the digestibility of other food constituents, notably of protein. Sago-pith meal is more suitable for pigs of > 100 lb. live-wt.  
A. G. POLLARD.

**Digestibility and feeding value of dreg meal.** S. J. WARSON (J. Agric. Sci., 1931, 21, 410—413).—Feeding trials with sheep show that dried distillers' dregs have a food value similar to that of linseed cake. The material has high protein and oil contents and low proportions of fibre and ash. The ash is almost devoid of Ca.  
A. G. POLLARD.

**Volatile products and water-soluble proteins in cotton-seed meats as related to variations in cooking conditions.** M. K. THORNTON, JUN., and C. R. BAILEY (Ind. Eng. Chem., 1931, 23, 833—834).—The influence of time, temp., and rate of heating have been studied in order to determine the optimum treatment prior to expression of the oil. The volatile products of the cook were condensed, scrubbed, titrated, and analysed (Kjeldahl). The sol. N in the cake was determined in an aq. extract (at 40° for 30 min.). The decomp. of pectins, as measured by the amount of volatile acids, increases with temp., with rate of rise of temp., and with duration of heating. Heating at a high temp., or prolonged treatment at a moderate temp., causes caramelisation. Loss of N, as measured by titratable bases, is increased by prolonged heating and high temp., a corresponding decrease in the H<sub>2</sub>O-sol. N in the cake occurring simultaneously. The ideal conditions of finishing the cooking are 107·2° for not longer than 20 min. H. J. DOWDEN.

**Determination of added water in milk.** A. SCHNECK and A. RUSTOM (Milch. Zentr., 1931, 60, 129—134, 189—196).—The content of added H<sub>2</sub>O in milk, found from the formula (1)  $X = 100(S - S_1)/(S_1 - 1)$ , or (2)  $X = 100(R - R_1)/R_1$ , or (3)  $X = 100(F - F_1)/F_1$  (where  $X$  is the H<sub>2</sub>O added to 100 pts. of milk,  $S$  the sp. gr. of the original milk,  $S_1$  that of the watered milk,  $R$  the % fat-free solid content of the original milk,  $R_1$  that of the watered milk,  $F$  the fat content of the original milk, and  $F_1$  that of the watered milk), shows variations from the true content due to the limitations of the methods in use for determining these values. If the sp. gr. be determined by pycnometer the value of  $X$  obtained from formula (1) is closer to the true value, but still low, in proportion to the fat present. In the case of skim milk the correct value is obtained. The discrepancy is due to sudden crystallisation of the fat and alteration in its sp. gr. on addition of H<sub>2</sub>O. Crystallisation of the fat also occurs slowly in normal milk kept below 13° for some hrs. On warming such normal or watered milk to 37° the effect is reversed. The phenomenon is not affected by the degree of dispersion of the fat, nor is it dependent on alteration of the casein or on the presence of the sludge, which can be removed by the centrifuge. By applying a correction to  $S_1$  appropriate to the fat content, or by ensuring that the fats in the watered and in the unwatered samples are both liquid before determining the sp. gr., the added H<sub>2</sub>O can be accurately determined from formula (1). Formula (2) does not give good results owing to the difficulty of determining  $R$  and  $R_1$  accurately. The reliability of Fleischmann's formula for determination of the solids is affected by the crystallisation of the fat. Formula (3) gives good results for full milk if  $F$  and  $F_1$  are determined accurately by the Gottlieb-Röse method. It is not applicable to skim milk. W. J. BOYD.

**Dependence of the lime content of the ash from cheese on the acidity of the milk.** A. SCHNECK and A. ELGER (Milch. Zentr., 1931, 60, 205—209).—Three samples of whey from which cream had been removed by different methods and the acidities of which were,

respectively, 5·45, 5·7, and 5·8, were made into cheese under identical conditions; the CaO contents of the ash from the cheeses were 32·8, 30·1, and 29·05%. Cheese from fresh centrifuged whey (fat 0·6%) when ashed contained 32·6% CaO. The CaO does not vary inversely as the measured acidity, but is closely related to the bacterial state produced by the time and temp. conditions prior to skimming. Milk of acidity 5·35, when de-creamed by different methods, gave wheys of acidity 5·4 and 5·5, and when made into cheese the ashes contained 24·3 and 21·89% CaO, respectively. The whey from pasteurised milk when mixed with 5% of soured milk, as in the manufacture of Edam cheese, had an acidity of 10·2, and the ash of the cheese contained 23·88% CaO. Whey from the same milk, but unpasteurised and without the addition of soured milk, gave a cheese the ash of which contained 27·33% CaO. H. J. DOWDEN.

**Analysis of egg yolk.** M. AUERBACH (Collegium, 1931, 396—400).—H<sub>2</sub>O is determined by the xylene distillation method. CHCl<sub>3</sub> should be used to extract the fat, and a correction of 0·5% deducted for H<sub>3</sub>BO<sub>3</sub> in products containing it. NaCl is titrated in the aq. extract of the ash. Proteins are determined by Kjeldahl and using the factor 6·25. Sap. val., unsaponifiable matter, I val., P and N contents are used to judge its purity. H<sub>3</sub>BO<sub>3</sub> is tested for. The practical value is judged from the appearance of a few g. when shaken with 200 c.c. of H<sub>2</sub>O at 30°. D. WOODROFFE.

**Approximate determination of egg content in macaroni paste.** A. SOLDI and S. TESTORI (Annali Chim. Appl., 1931, 21, 338—343).—The test is based on Lifschütz' reaction for cholesterol (A., 1913, ii, 886). 10 g. of powdered material are refluxed with 50 c.c. of xylene for 3 hr. on a water-bath. The residue is collected and washed with 20 c.c. of cold xylene. The filtrate, reduced to 2—3 c.c. by distillation, is evaporated slowly until a brown oily residue is obtained, which after cooling is dissolved in 20 c.c. of CHCl<sub>3</sub>. 5 c.c. of this solution are treated in a stoppered glass cylinder with 2 c.c. of Ac<sub>2</sub>O and 10 drops of H<sub>2</sub>SO<sub>4</sub> ( $d$  1·84). After shaking and keeping for 5 min., the greenish-blue tinge is compared with the colour obtained from a paste of known egg content prepared in the laboratory. O. F. LUBATTI.

**Analysis of oil in canned sardines.** L. ZANCHI and L. CINI (Annali Chim. Appl., 1931, 21, 353—354).—The fat naturally contained in the fish diffuses into the olive oil used for filling the cans, causing an alteration of the physical and chemical consts. of the oil. The usual analytical tests are capable of giving useful information only in cases of gross adulteration. O. F. LUBATTI.

**Vitamins in canned foods. X. Vitamin content of some common vegetables.** E. F. KOHMAN, W. H. EDDY, and C. Z. GURIN (Ind. Eng. Chem., 1931, 23, 808—811; cf. B., 1930, 1089).—Carrots, celery, and lettuce are all relatively low in vitamin-C, there being little difference between the inner and outer leaves of lettuce and between green and bleached celery. The vitamin content of carrots is reduced by shredding and

still further so by storage of the shredded material. Canned turnip greens are rich in vitamin-C and resemble spinach in this respect. The doses required to promote equal growth are 2—4 g. of greens, 10 g. of celery, 15 g. of carrots, and 25 g. of lettuce. Canned turnip greens are very rich in vitamin-A, 25 mg. being equiv. to 5 g. of bleached or 500 mg. of unbleached celery. Canned greens, lettuce, and celery are not rich in vitamin-B.

H. J. DOWDEN.

**Cacao butter. Rancid butters and margarines.**—See XII. **Intensive grassland management.**—See XVI. **Treatment of milk waste.**—See XXIII.

See also A., Sept., 1933, **Soya-bean lecithins.** 1081, **Camel flesh.** 1098, **Vitamins in sour-milk preparations.**

#### PATENTS.

**Treatment of baking flour.** METALLGES. A.-G. (B.P. 354,354, 3.5.30. Ger., 18.5.29).—Perphosphates of the alkali metals or Ca are added to flour as combined bleaching and raising agents. Up to 2½% is used and bleaching is effected by heating to 65—70°, the excess of perphosphate decomposing during baking, to assist the raising process.

E. B. HUGHES.

**Cream process and article. Production of butter.** H. D. WENDT, ASSR. to SHARPLES SEPARATOR CO. (U.S.P. 1,791,068—9, 3.2.31. Appl., 4.2.30).—(A) Normal cream is centrifuged by means of a high-speed centrifuge to produce a plastic cream with more than 65% of butter fat. This cream is miscible with skim milk or whole milk. (B) Cream containing more than 65% of butter fat, prepared as in (A), is squeezed or kneaded to form butter.

E. B. HUGHES.

**Meat cure and its manufacture.** E. H. WRIGHT (U.S.P. 1,792,561, 17.2.31. Appl., 9.12.27).—A product for imparting a smoked flavour to meat is prepared by destructively distilling a hard wood such as hickory. The retort is maintained at first between 38° and 71° and the gases produced are allowed to escape. The temp. is slowly raised to 482° and the resulting gases are condensed. The condensate is dehydrated in a vac. still and the product may be mixed with pickling salt, introduced into brine, or diluted and brushed over meat.

E. B. HUGHES.

**Treatment of tea and similar leaf [with humidified air]. Controlled withering of tea leaf and similar material.** J. A. MAIN (B.P. 354,791—2, 14.5.30).

**Canned goods.**—See I. **Mashing of cereal products.** **Hydrolysed protein products.**—See XVIII. **Edestin-calcium from whey.** **Vitamins.**—See XX.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Determination of phenolphthalein in pharmaceutical mineral oil emulsions.** J. A. SERRALLACH and R. J. OWEN (J. Amer. Pharm. Assoc., 1931, 20, 648—650).—2 c.c. of a 10% aq. mixture of the emulsion are diluted to 25 c.c. with EtOH. 0.5 c.c. of this solution is mixed with 20 c.c. of 0.01N-NaOH in a 25-c.c. Nessler tube and the colour is compared with that

produced by a similar amount of a solution of 10 mg. of phenolphthalein in 100 c.c. of 96% EtOH. The colours are matched by suitable addition of 0.01N-NaOH.

E. H. SHARPLES.

**Practical stability limits of some common [ophthalmic] solutions.** E. BERG (Pharm. Ztg., 1931, 76, 959—960).—The sensibility to light and the behaviour on keeping of various solutions used in eye treatment are described.

E. H. SHARPLES.

**Monarda punctata, L.** A. A. HARWOOD (J. Amer. Pharm. Assoc., 1931, 20, 631—637; cf. B., 1931, 273).—The following have been isolated from the EtOH extract of *M. punctata* leaves: hydrothymoquinone and a glucoside thereof, a volatile oil (89% of phenols), and a fatty oil (5.3% of drug) containing thymol, phytosterol, and glycerides of linolenic, oleic, and, possibly, palmitic acids.

E. H. SHARPLES.

**Identification and determination of dicodide, eucodal, and dilauidide.** J. KING (Analyst, 1931, 56, 498—503).—The behaviour of these three drugs towards various reagents is described and certain const. of the alkaloids and their salts are given.  $\text{HIO}_3$  in the presence of  $\text{H}_2\text{SO}_4$  gives a yellowish-brown colour, changed to mahogany by aq.  $\text{NH}_3$ . Wagner's reagent gives a cryst. ppt. with eucodal only. When the alkaloids occur as bases they may be titrated directly, or they may be liberated from their salts and titrated. Methods are given for their separation from morphine and its esters and from cocaine.

T. McLACHLAN.

**Guarana paste.** G. BERTRAND and P. DE BERREDO DE CARNEIRO (Bull. Soc. chim., 1931, [iv], 49, 1093—1096). The dry paste as prepared by natives contains about 4.8% of caffeine; the industrial paste, containing a little starch, contains about 4.2% of caffeine. No alkaloid analogous to morphine is present, the phenolic colour reaction observed by Schär and Thoms being due to tannins present.

R. BRIGHTMAN.

**Characters of essential oils of Sicilian flora.** L. LUISI (Boll. Uff. Staz. Sperim. Ess. Calabria, 1931, 6, 110—119).—*Mentha pulegium* gave 1.5% of oil,  $d^{20}_4$  0.9406,  $\alpha_D +9^\circ$ ,  $n^{20}_D$  1.4870, acid val. 4, sap. val. 24.14, esters (as menthyl acetate) 8.48%, ketones (Burgess method) 76.59%, solubility in 70% EtOH 1:2. Lavender gave 0.24% of oil,  $d^{15}_4$  0.8919,  $\alpha_D -4.58^\circ$ ,  $n^{18}_D$  1.4705, acid val. 0.44, sap. val. 26.35, esters (as linalyl acetate) 9.5%, free alcohols (as linalool) 60.02% solubility in 70% EtOH 1:2. *Thymus capitatus* gave 0.72% of oil  $d$  0.9460,  $\alpha_D$  0,  $n^{19}_D$  1.5011, phenols 67.65%, solubility in 90% EtOH 1:0.8. *Myrtus communis* gave 0.156% of oil,  $d^{21}_4$  0.8794,  $\alpha_D +17.60^\circ$ ,  $n^{18}_D$  1.4680, acid val. 1.02, esters (as myrtenyl acetate) 17.2%, free alcohols (as myrtenol) 20.16%, cineole 22% (by vol.), solubility in 90% EtOH 1:0.50. *Salvia* gave 0.22% of oil,  $d^{16.5}_4$  0.9123,  $\alpha_D -12^\circ$ ,  $n^{18}_D$  1.4735, acid val. 1.50, ester val. 8.70, esters (as bornyl acetate) 3.55%, total alcohols (as borneol) 15.65%, free alcohols (ditto) 12.87%, ester val. after reduction and acetylation 98.00, ketones (as  $\text{C}_{10}\text{H}_{16}\text{O}$ ) 9.95%, cineole 25.00% (by vol.), solubility in 80% EtOH 1:10. *Ruta* gave 0.22% of oil,  $d^{26}_4$  0.8389,  $\alpha^{26}_D +3.20^\circ$ ,  $n^{26}_D$  1.4302, ester

(C<sub>10</sub>H<sub>17</sub>·OAc) 29.64%, solubility in 70% EtOH 1:3, congealing point -15°; Me *n*-heptyl ketone was isolated.

T. H. POPE.

**Italian lemon and orange oils and U.S.P. standards.** E. BERTÉ (Annali Chim. Appl., 1931, 21, 303—307).—The reports (1926—1929) of the Laboratorio Chimico del Consiglio Prov. dell' Economia, Messina, and analyses completed during the year 1930, show the following variations. Oil of lemon:  $\alpha_D^{25} +56^\circ$  to  $+62^\circ$ ,  $d_4^{25}$  0.856—0.859, aldehydes (as citral) 4.5—6.0 wt.-%, residue on evaporation on water-bath 2.5—3.5 wt.-%,  $n_D^{20}$  1.474—1.4759; the differences between the original *n* and those of the first 5 c.c. of distillate vary between 0.0015 and 0.0025. Oil of orange:  $\alpha_D^{25} +98^\circ$  to  $+100^\circ$ ,  $d_4^{25}$  0.8485—0.8500, residue on evaporation 1.5—2.5 wt.-%,  $n_D^{20}$  1.473—1.475, solubility in 90% alcohol 1:8. These data taken from a very large number of genuine samples do not agree with the standards set by the U.S.P.

O. F. LUBATTI.

**Total alcohols [of geranium oil].** R. M. REED (Perf. Ess. Oil Rec., 1931, 22, 275).—The total alcohol content of oils containing both free alcohols and esters may be correctly calc. from the formula  $(1-0.00075a) \times by/(561-0.42b)$ , where *a*, *y*, and *b* are the same as in Cocking's formula for the determination of free alcohols (B., 1918, 166 A). Gildemeister and Hoffmann's formula is not satisfactory for such oils. E. H. SHARPLES.

**Vitamins in vegetables.**—See XIX.

See also A., Sept., 1023, Determination of H<sub>2</sub>O by distillation. 1075, Local anæsthetics. 1077, Synthesis of aporphine alkaloids. 1078, Derivatives of *p*-arsanilic acid. 1079, Colour reaction of ephedrine. Determination of citrates, salicylates, and benzoates. 1096, Extraction of insulin from blood. 1098, Isolation of the antineuritic vitamin. Vitamins in sour-milk preparations. 1100, Harmine from *Peganum harmala*.

#### PATENTS.

**Conservation of pyrethrum extract.** J. GUILLISSEN, and UNION CHIM. BELGE, SOC. ANON. (B.P. 354,680, 26.1.31).—The extract is enclosed in a glass phial or other receptacle capable of being hermetically sealed in a flame and not attackable by the product.

H. ROYAL-DAWSON.

**Producing a colloidal solution of the edestin-calcium in a whey free from milk-albumin and milk-ferment.** J. SIREK (B.P. 352,608, 5.5.30).—Part of the casein is removed from milk by MgSO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub>, the remainder by neutralising the acid with M<sub>2</sub>CO<sub>3</sub> and boiling. Lactalbumin and lactoglobulin are removed by boiling the filtrate with NaOH followed by addition of H<sub>3</sub>PO<sub>4</sub>, excess acid then being neutralised with MgCO<sub>3</sub> and filtered. The final whey is boiled with Ca(OH)<sub>2</sub>, ground hempseed added (at above 57°), the solution neutralised with H<sub>3</sub>PO<sub>4</sub>, brought to the boil, and filtered above 57°, sugar being added to prepare a syrup.

E. B. HUGHES.

**Manufacture of aminoalkoxy-derivatives of arylquinolines.** SOC. CHEM. IND. IN BASLE (B.P. 349,761, 16.5.30. Switz., 16.5.29).—2-, 3-, and 4-Phenylquinolines

containing aminoalkoxyl groups are prepared by general methods. Examples are: 7-β-diethylaminoethoxy-2-phenylquinoline (+2HCl, m.p. 230—231°), from 7-hydroxy- or 7-β-bromoethoxy- (m.p. 112—113°) 2-phenylquinoline; 2-β-diethylaminoethoxy-3-phenylquinoline (+HCl, m.p. 150—151°), from the 2-chloro-compound, m.p. 54—55°; 2-*p*-β-diethylaminoethoxyphenylquinoline (+HCl, m.p. 209—210°), from 2-*p*-β-hydroxyphenylquinoline; 6-β-*N*-piperidylethoxy-2-phenylquinoline-4-carboxylic acid, m.p. 220—221°, from *p*-aminophenyl β-*N*-piperidylethyl ether, m.p. 66—67°, benzaldehyde, and pyruvic acid; 6-β-diethylaminoethoxy-2-phenylquinoline (+2HCl, m.p. 211—212°); 7-β-diethylaminoethoxy-4-phenylquinoline (+2HCl, m.p. 250—251°); 7-β-dicyclohexylaminoethoxy-4-phenylquinoline, m.p. 104—105° (+2HCl, decomp. 110°); 2:4-bis-β-diethylaminoethoxy-3-phenylquinoline, b.p. 208—210°/4 mm. The prep. of β-dicyclohexylaminoethyl chloride, m.p. 186°, and alcohol, b.p. 135°/2 mm., and 2:4-dihydroxy-3-phenylquinoline, m.p. 94°, is mentioned.

C. HOLLINS.

**[Production of] barbituric acid derivatives.** COMP. DE BÉTHUNE (B.P. 349,455, 21.2.30. Fr., 16.1.30. Addn. to B.P. 343,140; B., 1931, 824).—The use of autoclaves is avoided by distilling off EtOH progressively and finally heating at 115°. New 5-Δ<sup>2</sup>-cyclopentenyl-barbituric acids described are the 5-*n*-propyl (m.p. 147—148°), 5-*isopropyl* (m.p. 171—172°), and 5-*n*-butyl (m.p. 145—146°) derivatives.

C. HOLLINS.

**Manufacture of therapeutic agents.** W. W. GROVES, FROM I. G. FARBENIND. A.-G. (B.P. 349,640, 3.3.30).—Basic anæsthetics etc. of the type Ar·CO<sub>2</sub>Y or Ar·CHYZ, in which Y and/or Z represent(s) an aminoalkyl group and Z may be hydrogen, are solubilised by treatment with CO<sub>2</sub>; thus β-diethylaminoethyl *p*-aminobenzoate, precipitated from aq. solution of its hydrochloride by NaOH, is redissolved on adding CO<sub>2</sub>. Other examples are: δ-dimethylamino-*sec*-isoamyl *p*-aminobenzoate, β-*N*-piperidylethyl *p*-β-methoxyethylaminobenzoate, etc.

C. HOLLINS.

**Manufacture of organo-mercury compounds and preparations comprising them.** JOHNSON & JOHNSON (GT. BRITAIN), LTD. FROM JOHNSON & JOHNSON (B.P. 349,685, 18.3.30).—*m*-Nitro-*p*-cresol is treated with Hg(OAc)<sub>2</sub> etc., in presence of alkali, if desired, to give, e.g., nitroacetoxymercuri-*p*-cresol, which is then converted by hot alkali into the sol. alkali salt of 3-nitrohydroxymercuri-*p*-cresol. Addition of Na<sub>3</sub>PO<sub>4</sub> or other buffer salt to the solution of the product prevents absorption of CO<sub>2</sub>.

C. HOLLINS.

**Manufacture of acylaminobenzenestibinic acids.** I. G. FARBENIND. A.-G. (B.P. 349,796, 13.6.30. Ger., 15.6.29).—An *o*-substituted arylamine carrying an acylamino-group in position 3, 4, or 5 is diazotised and treated with an alkali antimonite; or the corresponding arylaminestibinic acid is acylated. Examples include the prep. of 5-acetamidotoluene-*o*-stibinic acid, 2:5-dichloro-4-acetamidobenzene-1-stibinic acid, 4-acetamidoanisole-*o*-stibinic acid, 3-glycollamido-6-hydroxybenzene-1-stibinic acid, and 3-acetamido-4-methoxytoluene-*o*-stibinic acid.

C. HOLLINS.

**Preparing a remedy for cancerous tumour and product obtained thereby.** T. ISHIIHARA, and NIPPON SHINYAKU KABUSHIKI-KAISHA (B.P. 352,620, 9.5.30).—Human umbilical cord or the eggs of birds, fishes, or reptiles are boiled with  $H_2O$  and the aq. extract is purified by treatment with org. solvents.

E. H. SHARPLES.

**Extraction of certain vitamins from materials containing the same, and increasing the potency of the extracted vitamins.** B. SURE (B.P. 354,421, 20.5.30).—After removal of the org. solvent, preferably in presence of a small amount of acid, from an extract of material containing  $H_2O$ -sol. vitamins, the aq. residue is treated with glacial  $AcOH$  and  $COMe_2$  to ppt. inert material.  $AcOH$  and  $COMe_2$  are removed from the residue, which is then treated with activated C at suitable  $pH$  and the vitamin is removed from the adsorbent by washing with  $HCl$  and extracting with  $EtOH$ .

E. H. SHARPLES.

**Manufacture of substances having the character of sexual hormones.** SCHERING-KAHLBAUM A.-G. (B.P. 355,057, 11.9.30. Ger., 28.9.29).—Solutions of hormones in aq. org. liquids are extracted with non-miscible solvents in which the hormone is sol. (cf. B., 1931, 515).

E. H. SHARPLES.

**Production of hormone preparations.** "PHARMAGANS" PHARMACEUTISCHES INST., L. W. GANS A.-G. (B.P. 354,184, 29.4.30. Ger., 30.4.29).—Hormones, e.g., insulin, adrenaline, etc., salt-like compounds thereof, or substances containing hormones are treated with deoxycholic acid or other like acids or their derivatives in presence of solvents to give sol. addition compounds which are capable of oral administration.

E. H. SHARPLES.

**Wetting etc. agents.**—See III. Colloidal Pb phosphate.—See VII. Medicinal products from yeast.—See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., Sept., 1018, "Speed" of photographic emulsions. 1019, Actinometer for sunlight intensity.

### PATENTS.

**Photomechanical process of printing.** E. B. ELDRIDGE, Assr. to J. A. HAESELER (U.S.P. 1,793,070, 17.2.31. Appl., 3.9.26. U.K., 15.9.25).—A substantially grainless printing surface for collotype is produced by very weak development, giving a feeble image with the tone values in correct proportion, followed by hardening with a special  $CrO_3$  hardener.

J. LEWKOWITSCH.

**Manufacture of photographic pictures, especially of cinematographic film pictures.** F. BÜCHENBACHER (B.P. 354,731, 12.5.30).

## XXII.—EXPLOSIVES; MATCHES.

**Ignition of firedamp by coal-mining explosives.** I. Gallery experiments. II. C. GRIMSHAW and W. PAYMAN (Safety in Mines Res. Brd., 1931, Paper No. 69, 1931, 45 pp.).—In tests on Heathfield natural

gas in galleries of 5 and 6 ft. diam. with Samsonite No. 3 and Celmonite the range of ignition, and hence the charge limit, was determined under varying conditions. When fired by direct initiation in a gun of 46-in. bore, without stemming,  $\frac{7}{8}$ -, 1 $\frac{1}{2}$ -, and 2-oz. cartridges of Samsonite No. 3 gave 20 (full gun), 14, and 10 oz., and of Celmonite 14 (full gun), 16, and 14 oz., respectively. The corresponding values with inverse initiation were 16, 12, 10, and 14 (full gun), 10, 10 oz. Reduction of the bore length to 21 $\frac{1}{2}$  in. decreased the charge limit of 1 $\frac{1}{2}$ -oz. cartridges, with direct initiation, to 6 oz. for Samsonite No. 3 and 4 oz. for Celmonite. When the explosive was placed at the muzzle or suspended in the gallery a charge limit of only 2 oz. was obtained. A 2-oz. shot fired by direct initiation from the back of a 46-in. bore into a gallery of only 1 ft. diam. caused an ignition; when a mortar of  $\frac{1}{2}$  in. diam. and 6 in. long was used,  $\frac{1}{2}$  oz. of explosive ignited the gas. It is possible to raise the charge limit by placing a plate in front of the gun; this is probably due to the fact that the detonation products are caused to expand and not, as claimed by Audibert, that hot particles are arrested. In tests with stemmed shots, the difference between the results with direct and indirect initiation was accentuated, comparative figures of 28 and 16 oz. being obtained. Flame photographs of two explosives fired under various conditions definitely rule out the possibility of using such photographs to distinguish between explosives or methods of firing them. They show only luminous matter and, even so, not at a particular instant, but during the whole of its existence. Direct-flame photography is being supplemented by Schlieren photography. French and Belgian theories and Audibert's and Lemaire's views on the ignition of firedamp are discussed. The chief factors with unstemmed shots are the flame of the explosive itself, the flames sent out by the explosive (including hot luminous particles), the gaseous detonation products, and the pressure effect in the surrounding atm. The chief safeguards against ignitions are the use of the smallest possible charge and the least possible number of cartridges, the employment of direct initiation, and sufficient stemming. A preliminary test for firedamp is imperative.

W. J. WRIGHT.

### PATENTS.

**Blasting cartridges and charges.** A. C. SCOTT and D. HODGE (B.P. 354,305—3 and 354,312, [A, B] 4.3.30, [C] 8.4.30).—In blasting cartridges containing compressed  $CO_2$ , the heating element consists of: (A)  $KClO_4$  or other cryst. O-carrying substance coated with a phenol- $CH_2O$  condensation product, the  $KClO_4$  having a fineness of 200-mesh and the coated product of 30—120-mesh; (B) a mixture of  $KClO_4$ , finely-divided asbestos, one or more liquid nitro-compounds, a hydrocarbon oil, and castor oil; (C) a mixture of  $KClO_4$ , a liquid nitro-compound, a hydrocarbon oil, and castor oil.

W. J. WRIGHT.

**Production of smokeless powder.** WOLFF & Co. KOMM. GES. AUF AKT., R. WEINGAND, and H. I. SCHULZ (B.P. 353,634, 13.6.30. Ger., 22.6.29).—Nitrocellulose containing not above 12.8% N is gelatinised below 0° with org. gelatinising agents that are normally solvents



for nitrocellulose of lower N content, *e.g.*, MeOH. Further treatment of the product is also effected below 0°.

W. J. WRIGHT.

Gas-cartridges.—See XXIII.

### XXIII.—SANITATION; WATER PURIFICATION.

**Removal of "biochemical oxygen demand" by the trickling filters of Decatur, Ill.** W. D. HATFIELD (Sewage Works J., 1931, 3, 175—186).—Comparison of data obtained from trickling filters treating sewage containing widely varying proportions of a starch waste indicate that the percentage reduction of biochemical O demand is more closely correlated with temp. than with any other known factor. The filter loadings varied from 2000 to 10,000 lb. per acre per day of 5-day biochemical O demand. With conc. sewage the max. load of the filters was 5000 lb. per acre, but pre-aeration, by removing the more easily oxidisable matter, enabled loads of 7500 lb. per acre to be dealt with. More recent reductions in the proportion of starch waste due to recovery at the factory and trade depression are enabling the filters to operate at a load of 2000 lb. per acre and produce excellent effluents.

C. JEPSON.

**Effect of the dilution water on the biochemical oxygen-demand determination.** H. HEUKLEKIAN and N. S. CHAMBERLIN (Sewage Works J., 1931, 3, 187—198).—The influence of the dilution  $H_2O$  on the biochemical O demand (B.O.D.) of tank and filter effluents was determined by a series of comparative tests using 3 stream waters and 5 artificial dilution waters. The stream waters showed much greater variation than the artificial ones; distilled  $H_2O$  was an exception, the lower results obtained in this case and in one of the stream waters being due to deficiency of salts. Generally, the B.O.D. of tank effluent with artificial diluents was similar to the stream water most deficient in salts, whilst that of the filter effluent agreed with the other two streams. The salts present in sewage tend to overshadow the effect of the salts in the various artificial dilution waters, especially if the sewage is weak and the dilution low. In determining the B.O.D. load of a stream it is advisable to use a stored sample from an unpolluted part of the same stream as a diluent, whilst for theoretical and plant studies an artificial diluent with a greater variety of ions than was present in those used in these experiments is required.

C. JEPSON.

**Treatment of combined sanitary sewage and milk waste.** E. F. ELDRIDGE and W. E. ZIMMER (Sewage Works J., 1931, 3, 199—204).—Such a sewage is amenable to treatment if the primary sedimentation is complete within 3 hr. from the introduction of the waste into the sewer and the sludge produced is digested in a separate compartment. Stable effluents can be obtained by operating 6—7-ft. filters at a rate of 1.5 million gals. per acre per day, or 7—8-ft. filters at a 2.0 million-gal. rate.

C. JEPSON.

**Comparison of two methods for determination of hydrogen sulphide in sewage.** H. E. JOHNSON (Sewage Works J., 1931, 3, 205—209).—The  $H_2S$  was

determined by difference in non-aerated and fully aerated samples by the I titration and the tartar emetic coloration methods. Both methods, though accurate when applied to sulphides in distilled  $H_2O$ , were inaccurate with sewage containing over 3 p.p.m., owing to the colloidal and suspended matter. The addition of gelatin to the tartrate solution prevents flocculation of the ppt., and gives a clear amber solution which can readily be compared with the  $K_2Cr_2O_7$  standards. The end-point in the I titration is indistinct with sewage, and it is better to destroy the excess I with  $Na_2S_2O_3$  and titrate back with I. When determining total sulphides by this method, a blank determination in an acidified and aerated sample should be made to correct for the presence of I-absorbing substances other than  $H_2S$ . Details of a recommended procedure for carrying out both methods are given.

C. JEPSON.

**Rapid-stage sludge digestion.** A. M. BUSWELL and E. L. PEARSON (Sewage Works J., 1931, 3, 210—213).—A digestion tank of 2300 gal. capacity during 10 months' operation has shown that 1.25 lb. of gas are produced by the digestion of 1 lb. of org. matter. The rate of gas evolution increased suddenly after about 220 days' operation and was accompanied by an increase in alkalinity and ammoniacal N. The  $p_H$  rose steadily from 7.1 to 7.8.

C. JEPSON.

**Bacteriology and chemistry of water purification in the Breslau waterworks.** W. WAGENKNECHT (Gas- u. Wasserfach, 1931, 74, 673—678).—Details of the systems of water purification used and of the methods of overcoming difficulties which have arisen from time to time are discussed.

A. B. MANNING.

**Determination of  $SO_4$  in boiler water.**—See I.

See also A., Sept., 1933, Carcinogenic potency of mineral oils.

### PATENTS.

**Regenerating the breathing air in closed rooms.** H. STELZNER (B.P. 354,601, 24.9.30).—The reconditioned air is cooled and dried by passage through a multi-surface condenser.

C. JEPSON.

**Gelatin-composition container.** [Gas cartridges containing asphyxiating chemicals.] B. C. GOSS, Assr. to LAKE ERIE CHEM. CO. (U.S.P. 1,792,010, 10.2.31. Appl., 24.4.29).—The cartridges are made of or lined with a composition prepared by melting at about 77° gelatin, which has been soaked in cold  $H_2O$ , and stirring in 9—35% of glycerin, calc. on the wt. of dry gelatin; after they have been filled, the cartridges are sealed with a similar composition.

L. A. COLES.

**Apparatus for distilling water.** APPAREILS ET EVAPORATEURS KESTNER (B.P. 353,346, 1.11.30. Ger., 2.8.30. Addn. to B.P. 208,503; B., 1924, 572).—Inside the separator is a vessel acting as a precipitating and decanting chamber through which the raw water and precipitants flow.

D. K. MOORE.

**Gas masks.** DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 354,612, 8.10.30. Ger., 28.10.29).

**Dryer [for sewage etc.]. Absorbent for gas filters.**—See I. O for ventilation.—See VII.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

OCT 23 and 30, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Relations between elasticity and plasticity, toughness and brittleness. Practical means of characterising them.** P. REGNAULD (New Internat. Assoc. Test. Mat., Sept., 1931, 4 pp.).—Elasticity and plasticity correspond to properties of crystal arrangements, and are measured by tension diagrams, the tension being applied to ordinary test bars. Toughness and brittleness correspond to properties for which all the crystals are in the same state. The localisation of stresses and the rate of loading being given, differentiation is made between the properties of various substances by carrying out tests on notched bars.

C. W. GIBBY.

**Elasticity, plasticity, toughness, brittleness, and hardness.** M. ROŠ and A. EICHINGER (New Internat. Assoc. Test. Mat., Sept., 1931, 15 pp.).—Terms are defined.

C. W. GIBBY.

**Fundamental and practical (test) connexion between elasticity and plasticity, tenacity and brittleness.** A. SCHOB (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—Definitions are discussed.

C. W. GIBBY.

**Elasticity and plasticity, tenacity and fragility.** G. SACHS (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—Terms are defined.

C. W. GIBBY.

**Unification of the term viscosity.** C. H. WEISS and P. WOOG (New Internat. Assoc. Test. Mat., Sept., 1931, 23 pp.).—Since the determinations of viscosity by various instruments are not comparable and the term is only of relative accuracy, it is suggested that the coeff. of absolute viscosity should be measured in "poises," and of kinematic viscosity in "stokes." Various viscosimeters are described. A table showing the relationship between kinematic viscosity and the viscosity as determined by various instruments is given.

T. A. SMITH.

**Accuracy of measurement and variability of test results.** F. C. LEA (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—Modern testing machines in works laboratories can be depended on to a very considerable degree of accuracy and in many cases the reliability is greater than that of the homogeneity of most metals. Tensile test machines, extensometers, and torsion meters are available with an error of <1%, and for precise work this figure may be reduced by using refined devices.

C. A. KING.

**Heat transfer and pressure drop in empty, baffled, and packed tubes.** I. Heat transfer in packed tubes. II. Pressure drop in packed tubes. III. Relationship between heat transfer

and pressure drop. A. P. COLBURN (Ind. Eng. Chem., 1931, 23, 910—913, 913—919, 919—923).—I. Coeffs. of heat transfer to air flowing through a tube filled with granular material are found to be about 8 times those for an empty tube. An empirical equation from which the heat-transfer coeffs. can be calc. for turbulent flow in packed tubes is given.

II. [With T. H. CHILTON.] Tubes packed with dry or wet and hollow packing have been studied. For solid packing the actual velocity through the packing is dependent on the effective free cross-sectional area, and is about 10 times the value based on gross area.

III. [With W. J. KING.] The heat-transfer coeffs. for almost any type of baffle or packing can be estimated if the pressure drop be determined. Equations are given.

F. J. WILKINS.

**Heat transfer in stream-line flow.** T. B. DREW, J. J. HOGAN, and W. H. McADAMS (Ind. Eng. Chem., 1931, 23, 936—945).—Theoretical equations for the transfer of sensible heat by conduction in fluids flowing in stream-line motion inside heated or cooled pipes are discussed and applied to new experimental data.

F. J. WILKINS.

**Mechanism of heat transmission. I. Distribution of heat flow about the circumference of a pipe in a stream of fluid.** T. B. DREW and W. P. RYAN (Ind. Eng. Chem., 1931, 23, 945—953).—The peripheral variation of the rate of heat flow from the surface of a single, vertical, round pipe placed transversely in a stream of fluid is shown to be of the type predicted by Lohrisch, in which there are maxima at the front and back of the pipe and minima at the sides.

F. J. WILKINS.

**[Behaviour of] materials at high temperatures.** R. G. BATSON and H. J. TAPSELL (New Internat. Assoc. Test. Mat., Sept., 1931, 8 pp.).—A satisfactory method of testing the behaviour of materials at high temp. comprises the determination of the stresses corresponding to a rate of creep of about  $10^{-5}$  in. per in. per day at the end of a 40 days' test. Above 300° the fatigue limits under  $10^7$  alternations are entirely dependent on the rate of alternations applied. Safe ranges of stress in which the superior limit of stress is greater than the ultimate stress in tension can be applied at 2400 cycles per min. for  $10^7$  cycles, but are likely to cause trouble arising from the yielding of the material. Up to 200—250° the practical fatigue limits under mean tensile stresses are dependent on the yield point at the temp. considered; at about 200° the practical fatigue limits depend on a suitable proof stress which takes the place of the yield point.

A. R. POWELL.

\* The remainder of this set of Abstracts will appear in next week's issue.

**Instruments for measuring cooling power: the coolometer.** W. S. WEEKS (*J. Ind. Hyg.*, 1931, 13, 261—265).—A resistance thermometer circuit is adapted for measuring the cooling power of the environment of a solid body. C. W. GIBBY.

**Grading aggregates. I. Mathematical relations for beds of broken solids of maximum density.** C. C. FURNAS (*Ind. Eng. Chem.*, 1931, 23, 1052—1058; cf. *B.*, 1931, 181).—A mathematical development of the laws of packing of broken solids to give beds of max. density, applicable to concrete, paint, putty, rubber, coal storage, fuel beds, catalytic masses, etc. E. S. HEDGES.

**Grain size and fineness.** A. H. M. ANDREASEN (*New Internat. Assoc. Test. Mat. Sept.*, 1931, 6 pp.).—The term "sorting value" is suggested for the grain size at which a sieve separates. Its ratio to the size of mesh for ordinary substances is 0.8—0.9. A single number is insufficient to characterise the fineness of materials consisting of particles of different sizes, and a curve such as the distribution curve must be used. C. W. GIBBY.

**Size determination of free grains.** H. W. GONELL (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 11 pp.).—A bolting apparatus suitable for grading particles of sizes from  $60\ \mu$  to  $2\text{--}10\ \mu$  according to sp. gr. is described. C. W. GIBBY.

**Particle size of pulverulent materials.** L. R. FERET (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 9 pp.).—The "mean width" of a particle is defined as the mean, for a sufficient number of particles, of the distances between two tangents on opposite sides of the apparent outline of the particle, parallel to an arbitrary fixed direction. The whole range of granular and pulverulent materials is classified by limiting values of the mean widths of the various classes. C. W. GIBBY.

**Ageing of organic materials such as rubber, oils, resins, fibres, etc.** F. FRANK (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 9 pp.).—The ageing of such materials is mainly due to an oxidation process, and the final products of decomp. in all cases are  $\text{HCO}_2\text{H}$ ,  $\text{CH}_2\text{O}$ ,  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{H}_2\text{O}$ . Phenomena of fatigue resembling those in metals also occur in rubber, fibrous materials, and paints made from fatty oils and resins; the ageing and fatigue are succeeded by mechanical disintegration and mol. decomp. The literature is reviewed. D. F. TWISS.

**Ageing of organic materials.** G. BARR (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 10 pp.).—Although oxidation is the most general chemical process operative in the ageing of org. materials, other chemical changes are also involved. Acceleration of ageing tests by raising the temp. is unsatisfactory in that it may affect unequally the competing chemical reactions, and also the physical processes on which the properties of the final product depend. The effects of an increase in  $\text{O}_2$  concentration with or without rise in temp. are discussed and the associated difficulties are indicated. The difficulties of accelerated ageing tests are most pronounced when the natural ageing is due, wholly or partly, to the action of light; the shortcomings of

various methods of artificial illumination are analysed. "Weathering cabinets," which expose materials to a cycle of artificial weather changes, have an advantage in avoiding extremes of temp., but the difficulty of proportioning the cycle so as to represent "average weather" is evident. Accelerated ageing tests are probably of most value in assisting development of improvements in manufacture, and only rarely should be included in specifications. D. F. TWISS.

**Ageing of organic materials.** H. STAEGER (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 18 pp.).—It is important to distinguish between ageing changes which occur in service and those which occur during manufacture of a material. The cresol- $\text{CH}_2\text{O}$  resins, insulating woods, and "presspan" are discussed as illustrating the importance of ageing processes during manufacture. As providing examples of ageing in service, there are cited electrical insulating materials and steam-turbine oils; in the latter case the phenomena arising from the catalytic influence of Pb on the natural oxidation process and the gradual development of emulsifiability into  $\text{H}_2\text{O}$  are discussed. D. F. TWISS.

**Application of mineralogical and petrographical data to technical testing of non-metallic inorganic substances.** R. GRENGG (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 15 pp.).—A discussion. C. W. GIBBY.

**Ideal and practical (test) relation between elasticity, plasticity, tenacity, and brittleness.** F. B. SEELY (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 5 pp.).

**Present status of particle size measurement.** L. WORK (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 15 pp.).

**Application of mineralogy and petrography to the testing of non-metallic inorganic materials.** P. NIGGLI and F. DE QUERVAIN (*New Internat. Assoc. Test. Mat.*, Sept., 1931, 9 pp.).

**Grading aggregates. Testing resistance of constructional materials.**—See IX. **Retention of flue dust. Lubricated valves and corrosion.**—See X. **Microporous rubber.**—See XIV.

#### PATENTS.

**Furnace construction.** W. F. DETWILER, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,793,913, 24.2.31. Appl. 27.4.28).—A furnace for heat-treating metal sheets etc. is provided with a lining to the goods chamber composed of ferrous alloy containing 8% Cr. The flow of gases is from a combustion chamber over a fire-bridge, under a baffle, over another baffle, across the goods chamber, and back underneath the goods chamber. The baffles and false floor of the goods chamber are formed of the same Fe-Cr alloy.

B. M. VENABLES.

**Continuous-heating furnace.** F. J. WINDER, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,793,037, 17.2.31. Appl. 1.3.28).—A furnace for bars, slabs, or any material that can be assembled in rectangular bundles of which the length is greater than the width is formed as a vertical cylinder with removable lid and a hearth at the bottom supported on a hydraulic ram; some

distance above the lowest or normal position of the hearth are fixed ledges, the distance between which is less than the length of the bundles of goods, but greater than their width. A stack of bundles is maintained on the ledges, the bundles being alternately at right angles, and one bundle at a time is accepted on the hearth by raising it and rotating it through 90°, another bundle being placed on the top of the stack at the same time. After lowering the hearth and leaving one bundle in the lower high-temp. zone for a sufficient time, the material is slowly pushed sideways and one article at a time is caused to fall out downwards through a shoot.

B. M. VENABLES.

**Roasting oven.** W. L. PHILLIPS and J. R. NEAL, Assrs. to MAXWELL HOUSE PRODUCTS Co., INC. (U.S.P. 1,793,009, 17.2.31. Appl., 29.3.29).—A roaster for coffee etc. comprises a number of superposed, rotary, perforated cylinders through which the material is caused to travel by internal worms; the whole is enclosed in a casing which is ventilated at the top and provided with fuel burners opposite the lowest kiln, direct impingement of the flames on that kiln being prevented by screens. Thermostatic control is provided for the fuel and ventilation.

B. M. VENABLES.

**Sintering plant.** H. ERIKSSON (B.P. 353,833, 23.3.31. Swed., 27.3.30).—A rotating platform, preferably circular, supports the sintering pans, which may be transported to a dumping place by means of a crab. Material to be sintered is prepared and conveyed to the pans by mechanical means.

C. A. KING.

**Heating of substances. [Setting for a pan.]** S. E. MEYERS, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,792,918, 17.2.31. Appl., 21.11.25).—A setting for a boiling pan or similar vessel is described in which the control of the heating burner can be effected from the working platform of the pan; the gases take a generally U-shaped course downwards from the burner and upwards around the pan.

B. M. VENABLES.

**Apparatus for carrying out chemical reactions with the aid of electric [arc] discharges.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,735, 5.2.30).—The gases on their way to a zone where the reaction is effected by means of an arc are completely freed from turbulence by passing them through a narrow annular or conical passage between surfaces having an area at least 10 times the cross-section of the arc chamber. Examples are the production of  $C_2H_2$  from  $CH_4$  and of  $HNO_3$  from air.

B. M. VENABLES.

**[Charging of a] kiln or the like.** H. KRONSTAD, Assr. to BESSEMER CEMENT CORP. (U.S.P. 1,793,408, 17.2.31. Appl., 9.1.30).—A rotary inclined kiln is charged through apertures in the cylindrical wall any desired distance from the upper end. The apertures are preferably slanting, and on the lands between are situated external inclined blades which in conjunction with a surrounding stationary casing lift any material that falls out of the apertures when in a lower position. The material, which may be either slurry or paste, is delivered to the upper part of the casing.

B. M. VENABLES.

**Heat exchanger.** H. W. HOW, Assr. to STRUTHERS-WELLS Co. (U.S.P. 1,790,151, 27.1.31. Appl., 29.2.28).—

The apparatus comprises small bundles of tubes within surrounding conduits. The tubes are closely spaced, and the bundle is surrounded by a polygonal sheet so as to give a velocity to the outer fluid of the same order as that of the inner. The tube "plates" are very thick on account of the weakness due to close spacing and one end of each bundle is permitted to slide.

B. M. VENABLES.

**Heat exchangers.** A. E. WHITE. From O. E. FRANK HEATER & ENG. Co., INC. (B.P. 352,129, 23.4.30).—The exchanger comprises groups of U-tubes suspended in a tank which is provided with baffles under and over which the outer fluid flows. It is arranged so that units may be added or removed as desired.

B. M. VENABLES.

**Heat-exchange apparatus.** N. H. GAY (U.S.P. 1,789,739, 20.1.31. Appl., 5.11.29).—In a vessel, inlet and outlet headers, which are spaced apart vertically and at right angles, are connected by pipe coils grouped round an axial pipe through which the outer fluid passes to jets producing a swirl among the coils.

B. M. VENABLES.

**Heat-exchanger apparatus.** G. T. JACOBS, Assr. to ALCO PRODUCTS, INC. (U.S.P. 1,794,336, 24.2.31. Appl., 6.7.29).—The inner fluid passes through each half of a bundle of tubes in opposite directions, the outer fluid being given a combined longitudinal and transverse motion by intermediate tube plates having staggered apertures.

B. M. VENABLES.

**[Liquid] heat transferrers for high temperatures.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,886, 26.5. and 14.7.30).—In the Merrill or similar system of transferring easily regulable heat by means of a circulating liquid of high b.p. and non-coking properties, claim is made for the use of a mixture (b.p.  $<60^\circ$ ) of two or more polynuclear aromatic compounds one of which is diphenyl,  $CH_2Ph_2$ , or  $Ph_2O$ .

B. M. VENABLES.

**Air preheaters and like surface apparatus for exchange of heat between two fluids.** KINGS PATENT AGENCY, LTD. From AKTIEBOLAGET CALVERT & Co. (B.P. 351,984, 25.3.30).—An exchanger comprising parallel corrugated sheets with flow of both fluids transverse to the corrugations is constructed in the following proportions: the distance between the plates is  $<\frac{1}{20}$  the average speed of the fluids per sec., the height of the corrugations is about  $\frac{2}{3}$  the distance between plates, and the pitch of the corrugations is 4–7 times the height.

B. M. VENABLES.

**Apparatus for transmitting heat.** C. C. PLUMB (U.S.P. 1,790,555, 27.1.31. Appl., 10.8.28).—Non-coking oil is pumped in a closed circuit, at one point in which it is heated by a bath of fused salts and at another gives up heat usefully. The compressed air used for firing the salt bath may also be used to blow out the oil pipes prior to shutting down.

B. M. VENABLES.

**Manufacture of [large single] crystals.** P. W. BRIDGMAN (U.S.P. 1,793,672, 24.2.31. Appl., 16.2.26).—An apparatus for producing single crystals of a metal or other fusible crystallisable substance comprises a mould capable of being lowered at a slow controlled speed down

the axis of an electric furnace. The mould is composed of material that is entirely unaffected by the metal and it is conveniently pencil-shaped with the point downward; through the latter is inserted a copper rod or other means of cooling the point to form a seed crystal with definite orientation, the axis of which will be maintained throughout the solid produced provided the mould is lowered out of the hot zone at a rate not greater than the velocity of crystallisation.

B. M. VENABLES.

**[Solid] absorbent for refrigerating apparatus.** H. F. SMITH, Assr. to FRIGIDAIRE CORP. (U.S.P. 1,791,515, 10.2.31. Appl., 30.5.29).—As an absorbent for  $\text{NH}_3$  in refrigerators using  $\text{NH}_3$ , a mixture of  $\text{SrCl}_2$  and 4–10 (5)% of  $\text{LiNO}_3$  is claimed.

A. R. POWELL.

**Method of grinding.** E. KRAMER, Assr. to HARTSTOFF-METALL A.-G. (HAMETAG) (U.S.P. 1,793,098, 17.2.31. Appl., 22.6.29. Ger., 22.6.29).—A gas-borne grinding and classification system is described in which gases at different pressures or even of different composition are used at different stages, air-locks being provided at inlet, outlet, and transfer between stages. The final "sifting" is effected at low pressure and, if desired, in  $\text{H}_2$  or inert gas so that only very fine material is lifted and spontaneous oxidation prevented.

B. M. VENABLES.

**Pulveriser.** A. C. DANKS (U.S.P. 1,788,825, 13.1.31. Appl., 5.9.29).—A grinding mill of the slow-speed, ball-mill type is adapted to work with a small quantity of air to convey away the ground material by providing grooves in the rotating shell for the balls and an internal stationary drum having diaphragms which guide the air closely past the balls.

B. M. VENABLES.

**Grinding mills.** HARDINGE Co., INC., Assees. of H. HARDINGE (B.P. 353,261, 29.7.30. U.S., 30.8.29).—A mill of the cylindro-conical type is provided with inlet for air and outlet for air-borne dust through the same trunnion. The outlet is axial, and the inlet annular and provided with an internal baffle to guide the air along the wall of the mill.

B. M. VENABLES.

**Compartment[ed]-tube grinding mill.** J. V. DURNIN (U.S.P. 1,787,897, 6.1.31. Appl., 13.8.29).—A tube mill is divided into compartments by one or more diaphragms the openings through which are adjustable by sliding one plate on another.

B. M. VENABLES.

**Edge-runner grinding mills with revolving pans.** SMEDLEY BROS., LTD., and D. A. SMEDLEY (B.P. 353,611, 2.6.30).—A portable type of machine with framework of rolled steel is described.

B. M. VENABLES.

**Grinding mill.** R. S. IGLEHART, Assr. to BAUER BROS. Co. (U.S.P. 1,792,841, 17.2.31. Appl., 28.10.29).—An impact pulveriser and fan are constructed in such a way that they may be assembled for either direction of rotation.

B. M. VENABLES.

**Ball or tube mill.** E. G. STONE (U.S.P. 1,794,041, 24.2.31. Appl., 9.12.29. Austral., 21.1.29).—The mill comprises a number of cylinders grouped around the axis of rotation, and a common annular inlet header feeds the material to the tubes through automatically operated doors combined with scoops. At the outlet end a screen is provided for each tube and products of

two sizes are collected. The whole mill is subjected to reciprocation.

B. M. VENABLES.

**Separation or concentration of solid substances by flotation.** A. D. J. ELIÉ (B.P. 355,211, 13.2.30. Fr., 29.3.29).—The process comprises forming an emulsion of air or other gas in  $\text{H}_2\text{O}$  or other liquid to which has been added a foam-producing agent, but no oil, and allowing the  $\text{H}_2\text{O}$  and reagent to circulate in substantially closed circuit, driven by the lifting effect of the air- $\text{H}_2\text{O}$  emulsion; the separately oiled ore is added to the latter in a countercurrent direction. Mechanical stirring is excluded. Forms of apparatus described comprise a cylindro-conical vessel with cylindrical and conical guides, air injectors, and an air compressor.

B. M. VENABLES.

**[Pneumatic] separation of intermixed divided materials.** A. E. WHITE. From R. PEALE, W. S. DAVIES, and W. B. OAKES (B.P. 355,313, 23.4.30).—In a shaking table with upward air currents through a pervious deck, control of the supply of material and/or the air is effected mechanico-electrically by the depth of the bed of material.

B. M. VENABLES.

**Apparatus for recovery of solid materials by extraction.** LE R. C. TRESCOTT, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,794,874, 3.3.31. Appl., 16.2.29).—Pulverulent material is contained in a basket and extracted by the Soxhlet system. The solid material is kept in agitation by a mechanical stirrer, and a draw-off pipe is provided through which it is withdrawn by suction when the extraction is completed; a new charge may be inserted through a suitable aperture without stopping the action of the still.

B. M. VENABLES.

**Leaching.** J. M. COAHN (U.S.P. 1,793,465, 24.2.31. Appl., 9.6.27).—The apparatus comprises a tower in which countercurrent leaching of comminuted material is effected, the solids passing downwards and the liquor upwards. In the upper stage the material is compacted between two diaphragms, being delivered thereto from a lock device by a horizontal worm conveyor and abstracted by a vertical worm running at equivalent speed. In an intermediate zone the material settles through the liquor, but is hindered by conical shelves alternately rotating and fixed and of opposite inclination. In the lowest zone the remaining solids are allowed to fall unhindered and in a diffused condition through the incoming barren liquid; they are finally removed by another lock and worm conveyor.

B. M. VENABLES.

**Centrifugal dryer.** C. A. OLCOTT, Assr. to S. S. HEPWORTH Co. (U.S.P. 1,795,179, 3.3.31. Appl., 27.12.27).—A centrifuge of the basket type is provided with a swinging device automatically to divert the stream of outflowing liquor from a reservoir for mother-liquor to that for wash liquor when the latter is turned on.

B. M. VENABLES.

**Centrifugal separators.** H. W. FAWCETT (B.P. 355,366, 27.2.30).—Discharge nozzles of a centrifuge are provided with devices which tend to close them when discharge is taking place, and to open them wide when discharge ceases owing to obstruction by solid matter.

B. M. VENABLES.

**Centrifugal separators.** AKTIEB. SEPARATOR (B.P. 355,632, 8.1.31. Swed., 9.1.30).—A centrifuge for the separation of liquids (*e.g.*, transformer oils) the lighter constituent of which it is desirable to keep out of contact with air is provided with an inlet through the hollow driving shaft and an outlet in the opposite direction through a pipe with universal joints, both the bearings for the shaft being on one side of the bowl. The constituent unaffected by air is allowed to spray out of the bowl. B. M. VENABLES.

**Device for drawing off fluid from fine granular material in centrifugal machines.** L. ALTPETER (U.S.P. 1,793,204, 17.2.31. Appl., 15.8.28. Ger., 19.8.27).—A drainage device for a centrifugal machine with imperforate bowl comprises a thick-walled tube inserted radially through the wall of the bowl and having holes formed in the wall of the tube which are inclined towards the axis of the tube and away from the wall of the bowl. The inward inclination of the outlets substantially prevents exit of solids, but, as a further precaution, the tube may have an interior lining of gauze. B. M. VENABLES.

**Driving mechanism of centrifuges.** AKTIEB. SEPARATOR (B.P. 355,581, 27.10.30. Swed., 30.10.29).—A drive comprising motor, clutch, worm-wheel, and worm is described. The motor and male part of the clutch may be quickly removed. B. M. VENABLES.

**Separation of substances from liquids.** W. C. GRAHAM, H. S. RUMSEY, and A. U. WETHERBEE, ASSRS. to GILCHRIST & Co. (U.S.P. 1,794,374, 3.3.31. Appl., 3.8.26).—A multidecked thickener is described. All decks (but not the bottom of the tank) rotate and the rakes (except the lowest) are stationary; a common outlet for sludge is produced by downward tubular extensions of the decks, and peripheral spaces are left for upward flow of scum to a circumferential launder under which the feed conduit is situated. The clear liquid is withdrawn from underneath the decks as near to the axis as is convenient, so that the flow of clear liquid and sludge is roughly parallel and horizontal. B. M. VENABLES.

**Clarifying plant.** F. MIEDER (U.S.P. 1,792,919, 17.2.31. Appl., 29.3.28. Ger., 4.4.27).—A reciprocating rake for removal of settled sewage etc. is adapted to skim the surface of the liquid in the opposite direction, but not necessarily at every cycle. B. M. VENABLES.

**[Overload indicator for] sedimentation apparatus.** B. E. DUTTON, ASSR. to DORR CO. (U.S.P. 1,787,437, 6.1.31. Appl., 21.12.28).—In a traction thickener an overload will cause a slowing up of the raking arm so that a speed-indicating device may be used to operate an overload alarm. A device of this nature comprises an idle pulley running on the traction rail and formed with recesses arranged in a circle and communicating by narrow passages with each other and with a central recess; the latter contains a fixed but adjustable electric contact and the whole is provided with a fluid-tight cover and is then partly filled with Hg, which will be lifted away from the contact at normal speeds but descend and close an electric circuit should the speed drop unduly. B. M. VENABLES.

**Separation of two liquids of different sp. gr.** AKTIEB. SEPARATOR (B.P. 352,704, 23.6.30. Swed., 3.7.29).—The outlet passages of a centrifuge are so arranged that the lighter constituent has to pass through a liquid seal of the heavier. B. M. VENABLES.

**Receiving vessel and frame of centrifugal separators.** AKTIEB. SEPARATOR (B.P. 351,885, 20.11.30. Swed., 2.12.29. Addn. to B.P. 347,993; B., 1931, 659).—In a separator as described in the prior patent the receiving vessels have outlets extending completely round the machine. The dividing walls may be of cast or sheet metal. B. M. VENABLES.

**Liquid filter.** A. L. DORFNER (U.S.P. 1,794,281, 24.2.31. Appl., 12.6.29).—The filter comprises a number of annular filter bags clamped at the centre to wheel-like elements and separated by discs of mesh work to prevent bursting and permit exit of filtrate. The assemblage of wheels on the axis forms the conduit for prefilter. B. M. VENABLES.

**Filtration system.** H. D. ELFRETH, ASSR. to COCHRANE CORP. (U.S.P. 1,794,841, 3.3.31. Appl., 11.1.24. Renewed 27.3.29).—An enclosed sand filter is provided with an outlet for filtrate which is designed to prevent exit of sand and to eliminate spaces where liquid can stagnate. B. M. VENABLES.

**Filtration.** J. A. PICKARD (B.P. 354,739, 10.2.30).—Filter packs are assembled on tubes, a number of which are contained horizontally in a pressure-tight casing which is revolvable while in use and is easily opened. B. M. VENABLES.

**Filtering apparatus.** J. J. NAUGLE (U.S.P. 1,793,289, 17.2.31. Appl., 12.1.24).—A pressure filter for use with Suchar or other filter aid is described. A number of leaves are supported on a framework, preferably rotatable, within a pressure-tight casing, the filtrate from each leaf being conducted separately through a trunnion to a sight glass and then to a common outlet pipe which can be swung to deliver to a clear or cloudy filtrate receiving tank. Provision is made for reverse washing and for circulation of the prefilter. B. M. VENABLES.

**Filter unit.** J. J. ARMSTRONG (U.S.P. 1,792,797, 17.2.31. Appl., 3.12.28).—An edge filter is constructed of a metal strip wound in a spiral; radial drainage is provided by slots pierced on the centre line of the strip, and the spirals are inserted in frames which are assembled alternately with blank frames in a press. B. M. VENABLES.

**Filter.** E. M. BASSLER, ASSR. to D. J. MURRAY MANUFACTURING CO. (U.S.P. 1,793,449, 17.2.31. Appl., 1.11.27).—The filter screens consist of several plies of fabric (*e.g.*, wire) and may be applied to any type of filter. An apparatus described is suitable for clarification (*e.g.*, of white water from a paper-making machine), and comprises a number of pans attached to a conveyor, the ends of the pans being formed in such a way that they will overlap when on the straight upper run of the conveyor and their bottoms are formed of the multi-ply screens. B. M. VENABLES.

**Prefiltering means.** J. J. NAUGLE (U.S.P. 1,794,862, 3.3.31. Appl., 22.5.22).—An unactivated but highly

porous filtering medium is prepared from carbonised lignin residues from which the alkali has been entirely removed, *e.g.*, by dil. acid, but in which a substantial proportion of the mineral matter is allowed to remain.

B. M. VENABLES.

**Means for treating liquids.** J. J. NAUGLE (U.S.P. 1,794,916, 3.3.31. Appl., 3.5.26).—The liquid is forced backwards through a device resembling a centrifugal pump the rotor of which is formed with some of its surfaces of wire mesh and is charged with an adsorbent. Special application to the treatment of sugar melts, molasses, vegetable oils, etc. is indicated.

B. M. VENABLES.

**Device for producing dispersions.** G. A. BROWN, Assr. to BENNETT, INC. (U.S.P. 1,792,067, 10.2.31. Appl., 16.4.26).—An apparatus for carrying out the process described in U.S.P. 1,787,338 (B., 1931, 746) comprises helical blades rotating in an elongated, cylindrical, inclined vessel; the driving spiders of the rotor may divide the vessel into zones so that materials may be added at different stages of the mixing.

B. M. VENABLES.

**Processing apparatus.** I. E. COLVIN, G. N. MANISON, and H. G. WALTERS, Assrs. to PFAUDLER Co. (U.S.P. 1,794,670, 3.3.31. Appl., 17.3.26).—The liquid to be treated, *e.g.*, milk to be pasteurised, is drawn into and discharged from a series of tanks by means of vac. and pressure alternately applied, the valves being operated in correct succession by means of a cam shaft.

B. M. VENABLES.

**Evaporators.** W. W. TRIGGS, From STRUTHERS WELLS-TITUSVILLE CORP. (B.P. 353,248, 23.7.30).—An evaporator of the inclined bundle-of-tubes type is provided with return pipes for liquor both from the upper header of the tubes and from a separator, the pipes being bent at right angles and forming feet for the apparatus.

B. M. VENABLES.

**Desiccation of liquids.** F. F. PEASE, Assr. to F. F. PEASE, INC. (U.S.P. 1,794,978, 3.3.31. Appl., 9.6.25).—The liquid is centrifugally sprayed in a substantially transverse direction across a single, or two successive, axial annular current(s) of air or other gas, which alone should be sufficient to deflect and completely to dry the liquid; as an additional precaution, to prevent liquid reaching the walls, a tangential stream of air is also supplied.

B. M. VENABLES.

**Splitting up of a mixture of liquids into its components.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 355,294, 21.5.30. Holl., 3.6.29).—Two auxiliary liquids are chosen which are mutually immiscible or nearly so and have different solvent powers on the constituents to be separated; they should also be of different sp. gr., to avoid the use of a diaphragm. The auxiliary liquids are caused to flow in opposite directions in contact and the mixture to be separated is introduced at an intermediate point. For the separation of heavy hydrocarbon oils  $C_6H_6$  and liquid  $SO_2$ , and for middle hydrocarbons  $MeOH$  and  $CS_2$ , may be used.

B. M. VENABLES.

**Vapour-liquid column.** C. B. SCHNEIBLE, Assr. to C. B. SCHNEIBLE, K. F. SCHREIER, and B. B. SCHNEIDER (U.S.P. 1,794,986, 3.3.31. Appl., 5.4.26. Renewed

8.1.31).—The tower is provided with alternate annular shelves and central discs; under the latter and extending to the former are substantially vertical helical blades producing whirling of the vapour. The blades have their lower edges cut away so that they do not make contact with the inner edges of the shelves, thus enabling liquid to drip from the complete inner circumference.

B. M. VENABLES.

**Reaction towers for treatment of liquids with gases.** I. G. FARBERIND, A.-G. (B.P. 355,091, 7.10.30. Ger., 7.10.29).—The floor of a tower that is made of natural stone is divided into segments, each of which has a drain and a raised rim all round; the circumferential rims are embedded in the wall of the tower, and those along the radii and at the centre are covered by hollowed-out stones. The total capacity of the segmental dishes is sufficient to hold the drainage from the tower when the flow of gas is stopped.

B. M. VENABLES.

**Separation of gases.** R. L. HASCHE and W. H. DARGAN, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,794,377, 3.3.31. Appl., 25.8.27).—The mixture is compressed until the partial pressure of the desired constituent rises considerably above atm. (*e.g.*, to 2 atm.); the mixture is then passed over a suitable absorbent at room temp. until the desired constituent commences to escape. The adsorbed constituent is recovered by reduction of pressure to atm., only sufficient heat being added to maintain the adsorbent at room temp.

B. M. VENABLES.

**Separation of gaseous mixtures of low b.p. by partial liquefaction.** M. FRÄNKEL (B.P. 355,257, 16.4.30. Ger., 19.4.29).—The apparatus comprises 4 reversing heat accumulators, upper and lower rectifiers with condenser-evaporator in between, a supercooler with separate cooling coils for upper wash liquor composed of  $N_2$  and middle wash liquor rich in  $O_2$ , and an expansion engine providing misty vapour of  $N_2$  which is passed outside the supercooling coils. Choking of the accumulators is prevented by passing the air (or other mixed gas) in at increased pressure (about 4 atm.) and passing  $N_2$  and  $O_2$  out at a lower pressure (about 1.1 atm.), whereby the solid  $H_2O$  and  $CO_2$  will be sublimed at each cycle.

B. M. VENABLES.

**Filters for gases.** H. WITTEMEIER, Assee. of DEUTS. LUFTFILTER-BAUGES. M.B.H. (B.P. 355,431, 30.6.30. Ger., 28.6.29).—The filter is composed of a sheet of expanded metal rolled into the form of a hollow cylinder with multi-ply walls.

B. M. VENABLES.

**Apparatus for indicating and/or recording the change in volume of gases [during analysis].** J. W. & C. J. PHILLIPS, LTD., and B. JAMES (B.P. 354,852, 20.5.30).—A sample of definite vol. of a gas is aspirated by one side, say the bottom, of a piston on its out-stroke, the previous sample being delivered to waste by the top; on the in-stroke the sample is passed through a solvent, or other means of absorbing a constituent or otherwise changing the total vol. of the gas, and on the same stroke is received on the top of the piston, which is considerably smaller than the bottom by the specially chosen area of the piston rod. The change in vol. of the gas is measured by (a) holding both sides of the piston



at the same pressure (*e.g.*, atm.) and measuring the vol. not taken into the top of the cylinder by means of a floating bell, or (*b*) forcing the whole sample into the top of the cylinder and measuring the pressure.

B. M. VENABLES.

**Condensing apparatus for steam or other gaseous fluids.** G. R. SHEPHERD (B.P. 355,438, 3.7.30).—A condenser comprising a long bundle of tubes is divided into sections by intermediate tube-supporting plates and each section is provided with an air offtake; communication between sections is permitted through steam-inlet spaces both at the sides and top.

B. M. VENABLES.

**Conditioning of air for heating and/or ventilating purposes.** T. NEILSON and J. MARSHALL (B.P. 355,327, 21.5.30).—Steam is withdrawn from an intermediate stage of a turbine or engine and used in a conditioning apparatus. [Stat. ref.]

B. M. VENABLES.

**Viscosimeter.** H. W. KLINGER, Assr. to HERCULES POWDER CO. (U.S.P. 1,793,807, 24.2.31. Appl., 30.7.27).—The readings of a viscosimeter of the falling-ball type are observed by means of X-rays when operating with an opaque liquid.

B. M. VENABLES.

**Colorimeter [or colour comparator].** J. C. BAKER and C. F. WALLACE, Assrs. to WALLACE & TIERNAN PRODUCTS, INC. (U.S.P. 1,794,134, 24.2.31. Appl., 8.6.26).—The apparatus comprises a pair of tubes having opaque walls of which the surface is roughened or screw-threaded; one of the transparent bottoms is coloured to form a permanent standard, drain cocks are provided for both tubes, the liquid under test is conveniently adjusted to the standard by altering its depth, and a dip tube is provided for measuring that dimension. The apparatus is designed especially for use in making the  $\alpha$ -tolidine test for  $H_2O$ .

B. M. VENABLES.

**Grates for furnaces.** H. E. G. ROWLEY. From APPAREILLAGE INDUSTRIEL (B.P. 355,612, 29.11.30).

**Refrigerating methods and apparatus.** A. J. HAYWARD and A. T. A. D. MIDDLEMASS (B.P. 355,665, 20.5.30).

**[Adjustable stop for] Bourdon manometers.** Soc. ANON. L'ACCESSOIRE DE PRÉCISION (B.P. 354,968, 16.7.30. Ger., 13.8.29).

**Dissolving xanthates. Condenser for S.**—See VII. **Ore-roasting furnace.**—See X. **Precipitation apparatus for gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Physico-chemical characterisation of coal on the basis of its power of absorption of pyridine vapour.** M. CHORAZY (Przemysl Chem., 1931, 15, 233–252, 257–270).—The absorption of pyridine vapour by coal is greatest for lignites and least for anthracites, being inversely proportional to the C:H ratio; where this exceeds 20 absorption is insignificant. The pyridine vapour absorption nos. of vitrains at 15° are: 8.5 for anthracite, 5–5.5 for parabituminous anthracite, 3–4 for bituminous and parabituminous coal, 20–44 for caking gas coals, 50–67 for non-caking and 70–77 for gas-flaming coals. A similar classification of durains may be made on the basis of their pyridine

absorption no., viz., bituminous coal about 4, gas coal 40, and gas-flaming coal 50, but the difficulty of isolation of durain from parabituminous and anthracite coals renders the characterisation of this variety of little practical value. The same applies to fusains, the large absorptive surface of which renders the results for absorption uncertain. The absorptive power increases in the order fusain < durain < vitrain in more recent gas and gas-flaming coals, whereas a similar regularity is not observed in coal from older formations. The quantity of pyridine vapour absorbed by a given coal is closely proportional to its content of pyridine-extractable substances, as are also the velocities of absorption and of extraction. The residue after pyridine extraction ( $\alpha$ -substance) absorbs more pyridine vapour than does the coal before extraction. The greatest absorptive power is possessed by  $CHCl_3$ -sol.  $\beta$ -substance, that of  $\gamma$ -substance being distinctly smaller. Max. absorption is obtained by preheating coal at 400–450° in an inert atm., and the temp.-velocity of absorption curve runs parallel to the temp.-plasticity curve of a given coal. The determination of the absorptive properties of coals can thus serve as an index of the temps. at which they transiently assume the plastic state.

R. TRUSZKOWSKI.

**The rotary kiln in cement manufacture. VIII—X. [Drying of coal.]** W. GILBERT (Cement, 1931, 4, 26–35, 648–664, 871–881; cf. B., 1931, 22).—VIII. A rotary tube dryer 50 ft. long and 5 ft. in diam. encased in a brickwork chamber reduced the moisture content of coal from 2.2 to 0.8% at the rate of 2.29 tons/hr. with an average loss of 2.43% of volatile matter. The tube was heated externally by waste gases entering at 345° and leaving at 165°. The heat balance shows that 11.4% of the total heat was utilised for evaporating moisture, but, against this, the draught was found to be defective.

IX. Calculations based on the vol. of gases passing first around the dryer and then through it amongst the cascading coal show that 15.6% of the heat reaches the coal through the walls and 82% by direct contact of the gases with the coal.

X. The effect of the brickwork chamber was shown to be comparatively small in the above, and the heat balance of an exposed tube dryer gives 56.6% of heat effective in drying on counterflow design and 44% for parallel flow.

C. A. KING.

**Sampling of coal.** W. A. SELVIG (New Internat. Assoc. Test. Mat., Sept., 1931, 9 pp.).—Sampling is described under 3 divisions, viz., collection of a representative gross sample, reduction of the gross sample to one of laboratory size, and preparation of a laboratory sample. The gross sample, taken by a large number of increments, from different parts of the sample, should be not less than 1000 lb., except for slack coal and small sizes of anthracite, when a gross sample of 500 lb. is sufficient. If the coal is high in ash, a gross sample of 1500 lb. or more is required. The gross sample must be crushed, mixed, and systematically reduced in quantity. The method of collecting gross samples and their reduction for analysis as recommended by the A.S.T.M. are outlined.

C. B. MARSON.

**Sampling of coal and coke.** E. S. GRUMELL and J. G. KING (New Internat. Assoc. Test. Mat., Sept., 1931, 33 pp.).—The following are discussed: the work of other investigators, distribution of ash in coal, application of a modified size-weight ratio theory to sampling, factors of safety, experimental evidence in favour of certain recommendations, methods of collection of the bulk sample, and the reduction of this sample to one of laboratory size. C. B. MARSON.

**Sampling of coal, coke, and other fuels, and of clinker and ash.** K. BUNTE (New Internat. Assoc. Test. Mat., Sept., 1931, 13 pp.).—The fundamental principles involved in sampling are described. The methods of sampling in individual cases are discussed and the accuracy is demonstrated by test results.

C. B. MARSON.

**Determination of volatile matter in solid fuels.** R. DE BENEDETTI and G. ROSSI (New Internat. Assoc. Test. Mat., Sept., 1931, 8 pp.).—An electric furnace which enables several determinations to be carried out simultaneously, using either Pt or porcelain crucibles, is described; the temp. can be maintained between 1000° and 1100° and special means are taken to prevent oxidation. The furnace can be used also for determining the ash and its fusibility. The results obtained with this and other methods are compared.

C. B. MARSON.

**Determination of the fusion point of coal ash.** F. S. SINNATT (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—Following a summary of the literature on the subject, four methods for the determination, which incorporate certain distinct principles, are given. The possible influence of the heterogeneity of the coal substance on the fusion point of any particular sample is also discussed. Studies of the fusion point of ash from different layers constituting a seam of coal suggest that the value obtained from an average sample must be interpreted with caution.

C. B. MARSON.

**Determining the melting of [fuel] ash.** K. BUNTE (New Internat. Assoc. Test. Mat., Sept., 1931, 6 pp.).—Current methods are reviewed, and Bunte and Baum's method (B., 1928, 322) is described. Reproduction of results in various laboratories, the relationship of m.p. and softening points to the m.-p. curve, the similarity and variability of the m.-p. curves of certain ashes, and the sources of error are discussed. C. B. MARSON.

**Desulphurisation of coke.** J. S. GURARJI (Ukrain. Chem. J., 1931, 6, [Tech.], 49—83).—The best desulphurising agent for coke is  $H_2$ , which at 1000° removes up to 54% of the total S. Steam at 800°, under optimal conditions, does not remove more than 11—16%, whilst  $C_2H_2$  removes up to 17%; the action of  $C_2H_2$  is due to  $H_2$  liberated by its decomposition. The action of  $CH_4$  at 1000° is similar to that of  $H_2$ , but the velocity of reaction is smaller; about 40% of the  $CH_4$  is decomposed in the process. In factory practice coal gas should be used, 45% of coke-S being removed under appropriate conditions at 1000°. In general, the velocity of reaction is a function of the degree of comminution of the coke.

R. TRUSZKOWSKI.

**Occurrence of nitrogen oxides in coal gas.** H. A. J. PIETERS (Brennstoff-Chem., 1931, 12, 285—286).

—N oxides, which occur in coal gas as a result of the suction of flue gases into the retorts, may combine with the unsaturated hydrocarbons of the gas to form resinous, and possibly also explosive, deposits. They may be determined by adding excess of  $O_2$  to the gas, to convert the NO into  $NO_2$ , and then determining the latter colorimetrically with *m*-phenylenediamine. In one coke-oven plant it was found that the concentration of NO in the gas rose in passing through the saturator (from 7.6 to 13.2 c.c./cu. m.) as a result of using  $H_2SO_4$  containing NO; it fell again during the subsequent benzol washing and gas purification to 1.7 c.c./cu. m.

A. B. MANNING.

**Thermal decomposition of low-temperature tar constituents.** I. Reaction products, II. Reaction mechanism, of the thermal decomposition of higher phenols of low-temperature tar. Y. KOSAKA (J. Soc. Chem. Ind., Japan, 1931, 34, 241—242 B, 243—244 H).—I. The tar acid fraction (85.3% of total) boiling above 210°, decomposed by trickling down a  $SiO_2$  tube at 700°, 800°, and 850°, gave 76%, 52%, and 30% of condensate, 20%, 38%, and 50% of gas, and 4%, 10%, and 15% of C. The decomp. products qualitatively resemble those from cresols.

II. There is an optimum temp., about 800°, for the dealkylation and reduction of higher phenols through cresols and  $C_6H_6$  homologues to PhOH, PhMe, and finally  $C_6H_6$ . Diphenyls result from dehydrogenation of hydrocarbons rather than from phenols, whilst  $C_{10}H_8$  and  $C_{14}H_{10}$  seem to be condensation products of monocyclic phenols.

C. HOLLINS.

**Berginisation of coal and tar.** N. A. ORLOV (Ukrain. Chem. J., 1931, 6, [Tech.], 1—12).—A review of work published by various authors on the low-temp. treatment of coal and tar for the production of liquid hydrocarbons. The best yields are obtained from bituminous coals.

R. TRUSZKOWSKI.

**Catalytic desulphurisation and hydrogenation of a primary tar fraction.** J. M. PERTIERRA (Chim. et Ind., 1931, 26, 9—14).—The S in a fraction, b.p. 180—300°, of a commercial low-temp. tar exists in the form of  $H_2S$  (0.17%, expressed as S in the oil), mercaptans (0.09%), thioethers (0.04%), thiophen derivatives (0.14%), and other compounds (0.26%). Attempts to desulphurise the oil by passing the vapour, in the presence of  $H_2$  at the ordinary pressure, over metallic catalysts reduced the total S content by not more than 60%. By passing the vapour subsequently over a hydrogenating catalyst, *e.g.*, Ni, the tar acids in the oil were reduced by 13% and the bases by 64%. Hydrogenation under pressure ("berginisation") in the presence of an  $Fe_2O_3-NH_4$  molybdate catalyst yielded 14% of light spirit, b.p. 50—180°, and reduced the tar acids by 74%. The product was completely sol. in  $Et_2O$ . Hydrogenation removed 70% of the total S, whilst the residual 30% was easily eliminated by further treatment.

A. B. MANNING.

**Testing of bituminous materials.** P. HUBBARD and C. S. REEVE (New Internat. Assoc. Test. Mat., Sept., 1931, 5 pp.).—The twenty tests adopted by the American Society for Testing Materials are classified under consistency, heat, solubility, and miscellaneous

tests. Other tests dealing with physical properties of paving mixtures, weathering tests, and refinements in the ductility and distillation tests are projected. The application of the tests in American practice is briefly described.

T. A. SMITH.

**Constituents of petroleum.** J. VON BRAUN (Z. angew. Chem., 1931, 44, 661—664).—Recent work on the isolation, separation, identification, and properties of the naphthenic acids present in petroleum from the principal oil fields of the world is briefly outlined.

A. R. POWELL.

**Pyrolysis of propane.** G. O. EBREY and C. J. ENGELDER (Ind. Eng. Chem., 1931, 23, 1033—1035).—Pyrolysis of  $C_3H_8$  in  $SiO_2$  tubes at 600—830° gives varying amounts of  $H_2$ ,  $CH_4$ ,  $C_2H_6$ ,  $C_2H_4$ , and  $C_3H_6$ ; the amount of  $H_2$  increases with rise in temp., whilst unsaturated hydrocarbon formation is a max. at 770°. C, tar fog, and tarry oil are produced at temp. above 600°, 650°, and 700°, respectively. With catalysts containing Ni, Co, Cr, or Fe, increased production of  $H_2$  and  $CH_4$  occurs. The primary change in the pyrolysis is considered to be the formation of Me, Et, and Pr radicals and H atoms; these subsequently decompose, inter-react, and undergo dehydrogenation. The results show that a C-C linking undergoes readier scission than a C-H linking. A technique for analysing gaseous hydrocarbons by low-temp. fractionation supplemented by absorption and combustion methods is developed.

H. BURTON.

**Utilisation of acid sludge from benzol washing.** G. A. ELLIS (Gas World, 1931, 95, Coking Sect., 88—90).—The operation of a plant according to the Still process (B.P. 277,619; B., 1928, 220) is described. This process consists in mixing the acid sludge with mother-liquor from the  $(NH_4)_2SO_4$  plant, at 60—70°, allowing to settle, running off the acid liquor to the mother-liquor well, and dissolving the remaining resins in hot crude tar, from which any benzol entrapped in the acid sludge is distilled off, washed free from acid fumes, and recovered. It was found preferable to run off the acid sludge without allowing settling time, to prevent the formation of clots of thick sludge. The mother-liquor used must be of high sp. gr. to aid separation, and of low acidity to avoid charring the resin and tar. Difficulties in separation which arose occasionally were overcome by using a light tar instead of the ordinary crude tar. The plant is operated with low steam consumption and gives a tar product which is mixed with the crude tar without disadvantage.

A. B. MANNING.

**Artificial ageing of special mineral oils.** M. VAN RYSSSELBERGE (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—An effort has been made to standardise a normal method of artificial deterioration to determine the value of such oils. The  $O_2$  absorption at 110° is determined in a sealed vessel fitted with a Hg manometer. The quantity of  $O_2$  absorbed is greatest in the case of refined oils. The absorption in the presence of metals varies with the nature of the metal, and consequently ageing tests should be carried out in the presence of the metal with which the oil will be in contact when in use. Owing to the selective

action of Cu, transformer oils are best tested in the absence of metals.

T. A. SMITH.

**Some properties and compositions of the [68] gasoline fractions of [7] representative Japanese crude petroleum.** II. M. MIZUTA (J. Soc. Chem. Ind. Japan, 1931, 34, 240—241 B).

**Grading aggregates. Ageing of materials.**—See I. Catalytic gas reactions.—See III.  $(NH_4)_2SO_4$ . Detergents.—See VII.  $Me_3BO_3$  in analysis.—See XIII. Microporous rubber [for oil lubrication].—See XIV. Furfuraldehyde.—See XV. Pine-tar oil for orchards etc.—See XVI. Pyrethrum extracts. Coal-tar distillery water.—See XXIII.

#### PATENTS.

**Coal-cleaning apparatus.** H. L. McLEAN, ASST. to G. W. WILMOT and F. H. BLATCH (U.S.P. 1,792,179, 10.2.31. Appl., 9.5.28).—The coal is fed on to an inclined perforated plate supported in a tank in such manner that water under pressure can be passed continuously through the perforations and bring about a separation of the coal and slate. The coal is carried over adjustable inclined guide plates spaced suitably from the perforated plate. The coal and slate are deposited in separate compartments in the lower part of the tank, from which they are removed by means of conveyors.

A. B. MANNING.

**Coke ovens and the like.** C. STILL (B.P. 354,664, 9.12.30. Ger., 11.12.29. Addn. to B.P. 351,872; B., 1931, 871).—The separate sections of the tubes which are used for leading away the volatile distillation products are made of the same external cross-section and are connected by butt- or cone-shaped joints. The openings through which the gases pass into the tubes are shielded internally to prevent particles of fuel being carried into the tubes. The arrangement is such that the tubes can be forced from outside into the previously charged fuel.

A. B. MANNING.

**Coal-carbonising and gas-enriching apparatus.** E. A. DIETERLE (U.S.P. 1,790,745, 3.2.31. Appl., 21.1.24).—Powdered coal, together with the gas which is to be enriched, is injected into the top of a vertical chamber with converging ends, and passes through vertical metal tubes which are arranged in parallel within the chamber, and are heated by the circulation around them of hot gases from a combustion chamber. The residual material is collected in a hopper at the bottom of the chamber, and can be removed without interrupting the continuous operation of the plant. Finely-divided particles and tarry matter entrained with the gas are removed in an electrical precipitator.

A. B. MANNING.

**Operation of carbonising chambers.** N. V. SILICA EN OVENBOUW MIJ., and W. HIBY (B.P. 353,998, 12.6.30).—Towards the end of the carbonising period in intermittently operated coking chambers, water-gas is produced by introducing from one or both ends of the chamber tubes with downwardly directed outlet openings into the cleavage formed in the middle of the coke cake, and leading  $H_2O$  or steam into the chamber through these openings. Instead of  $H_2O$ , gas liquor, or  $H_2O$  mixed with tar or oil, can be used.

A. B. MANNING.

**Retort for destructive distillation of solid carbonaceous material.** E. L. SCHULTZ (B.P. 352,487, 9.4.30).—A vertical annular retort has an inner fixed shell, an intermediate rotary shell, and an outer rotary shell; the fuel is passed down through the heating space between the first and second shells, the space between the second and third forming a gas chamber through which the volatile distillation products pass to suitable outlets. The inner shell has a core of refractory material designed to direct the heating gases from a fire-box or burner against the inner side of the shell. The material to be distilled is forced through the heating space of the retort by a tier of overlapping rings, to which spiral worm-edges, acting as scrapers, may be attached; these rings are rotated, and also given a shaking motion, with the intermediate shell of the retort. The spent material is discharged into a rotary water trough and thence conveyed to a receiver. The rotating shells of the retort are so suspended that they operate continuously irrespective of the expansion or contraction of their parts.

A. B. MANNING.

**Gasification of solid fuel.** A. J. A. HERENG (B.P. 352,271, 18.7.30. Fr., 18.7.29).—The apparatus comprises a vertical retort which is supported within a vertical chamber, a horizontal fuel gasification chamber, and an endless conveyor which forms the bottom of both the retort and the gasification chamber, and conveys the fuel from the former to the latter. An adjustable damper limits the height of the layer of fuel admitted to the gasification chamber. Provision is made for supplying air and steam at the bottom of the gasification chamber. The hot gases produced in the latter pass through the lower part of the retort and thence through the space between the retort and the surrounding chamber, thus serving to heat the retort externally. A part of the gases may be passed through the retort and withdrawn with the distillation gases. Provision is made for recirculating the latter through the gasification chamber, if desired. A. B. MANNING.

**Utilisation [distillation] of bituminous fuels.** R. H. CARR and C. B. WATSON, Assrs. to PURE OIL CO. (U.S.P. 1,793,838, 24.2.31. Appl., 29.10.27. Cf. U.S.P. 1,698,907; B., 1929, 587).—The fuel is conveyed continuously up through a cylindrical chamber counter-current to a gaseous heating medium, *e.g.*, steam, which is introduced at the top of the chamber, and, after passing through the fuel, is withdrawn with the volatile products of distillation at the bottom. The upper layer of the fuel is continuously agitated to render it pervious to the heating medium. The coke is discharged in a compact form by a spiral conveyor. A. B. MANNING.

**Production of [distillation] products from vegetable substances.** E. L. RINMAN (B.P. 353,665, 8.7.30. Swed., 26.5.30. Addn. to B.P. 334,724; B., 1930, 1138).—Digestion is effected without using an excess of alkaline-earth bases, and then a further addition of these bases is made to the product before dry distillation.

A. J. HALL.

**Manufacture of fuel.** R. L. RODGERS (U.S.P. 1,793,014, 17.2.31. Appl., 27.4.28).—The C residue of petroleum distillation or cracking is pulverised, mixed with 10–25% of a liquid petroleum derivative, *e.g.*,

crude petroleum or a liquid residuum, and carbonised to give a solid coherent fuel. A. B. MANNING.

**Manufacture of fuel briquettes.** D. S. ANDREWS (U.S.P. 1,793,833, 24.2.31. Appl., 28.4.30).—A mixture of anthracite "culm" (85–90%), or of anthracite "culm" (45%) and "steam-size" anthracite (45%), with petroleum residuum asphalt (5–10%) and pulverised low-volatile bituminous coal (5%) is briquetted. The asphalt is heated to a temp., *e.g.*, 180–190°, at which it flows freely, and is mixed with part (the "steam-size") or all of the anthracite, preheated to the same temp.; the remainder of the anthracite and the bituminous coal are then added and the whole is thoroughly mixed. A. B. MANNING.

**Calorific improvement of charcoal and other porous fuels.** H. K. MARUSCHEK (B.P. 353,929, 23.4.30. Austr., 29.3.30).—The charcoal is dehydrated and then subjected to the action of compressed air under 4–6 kg./sq. cm. and either at 70–73° or alternately at 70–73° and 5–10°. Under these conditions O<sub>2</sub> is taken up and retained in the pores of the fuel.

A. B. MANNING.

**Gasification process.** E. A. DIETERLE (U.S.P. 1,792,632, 17.2.31. Appl., 7.4.25).—The apparatus consists of a cylindrical shell, the lower part of which constitutes the generator or producer, whilst the upper part contains a central retort surrounded by a fixing chamber comprising a number of vertically arranged elongated passages. Air and steam are passed intermittently through a hot bed of solid carbonaceous fuel in the generator. Powdered fuel is injected intermittently down the central retort, which is heated by the hot blow gases etc. which are passed through the fixing chamber. The carbonised fuel falls on to the heated mass in the generator while the evolved gases and vapours mix with the gases from the generator and pass together therewith over the hot surfaces in the fixing chamber, and thence to the gas outlet.

A. B. MANNING.

**Apparatus for manufacture of combustible gas.** H. O. LOEBELL, Assr. to H. L. DOHERTY (U.S.P. 1,790,823, 3.2.31. Appl., 21.9.23).—The fuel, preferably a mixture of coal and coke, is passed down a vertical shaft, the coal being distilled to form coal gas, and thereafter the coke is alternately blasted with air and steam to produce blue water-gas. The water-gas and coal gas both pass upwardly through the fuel column, the mixed gas leaving the top of the shaft through a suitable outlet. A comparatively deep zone of high temp. is maintained in the fuel bed, and the blast gases, relatively high in CO, are burned in regenerators, which are used for preheating the air for and generating and superheating the steam used in the process. During the gas-making operation H<sub>2</sub>O is admitted to the bottom of the fuel column to quench the fuel and to form additional steam for the process. A. B. MANNING.

**Gas producer.** A. K. BRADLEY, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,795,162, 3.3.31. Appl., 27.9.26).—A gas producer constructed so as to secure a continuous discharge of ashes from the bottom of the producer during the gas-making operation is described.

A. B. MANNING.

**Gas-generating apparatus.** H. F. SMITH, Assr. to GAS RESEARCH CO. (U.S.P. 1,794,478, 3.3.31. Appl., 19.3.28).—A gas producer suitable for household use is provided with automatic means for passing the gas into the household system only when it possesses the requisite calorific val., and with means for controlling its operation from a distance. A. B. MANNING.

**Gas generators.** T. A. LAPIERRE (B.P. 351,494, 26.3.30).—A gas generator for the distillation of lignite, sawmill waste, etc. comprises an inclined distillation chamber, communicating at its lower end with a primary combustion chamber, and separated from a secondary combustion chamber by the inclined wall over which the carbonaceous material flows. The heat for distillation is provided either by passing the gases from the combustion of the carbonised material in the primary combustion chamber through the secondary combustion chamber, or by burning the distillation gases in the latter. The distillation chamber may be divided by transverse walls into a number of separate retorts sealed from one another by the layer of material. The flow of the material is assisted by suitably driven agitators. A. B. MANNING.

**Water-gas generators.** J. PINTSCH A.-G. (B.P. 351,828, 11.9.30. Ger., 11.9.29).—The generator is provided with a waste-heat boiler producing steam at a higher pressure, and a steam jacket producing steam at a lower pressure. During the "blow" both sources of steam are utilised in the first and second stages respectively of a two-stage steam turbine. During the "run" the high-pressure steam is used in the first stage of the turbine, leaving it at a pressure equal to that of the low-pressure steam, with which it is then mixed and both are passed through the fuel bed of the generator. A. B. MANNING.

**Production of heating and illuminating gas.** A. F. KUNBERGER, Assr. to UNITED GAS IMPROVEMENT CO. (U.S.P. 1,792,172, 10.2.31. Appl., 13.6.25).—A mass of coal, containing a small proportion of Fe or  $\text{Fe}_2\text{O}_3$ , is raised to incandescence by blasting it with air in a generator, and the blow gases are passed through a regenerator chamber containing a mixture of refractory material and Fe, and thence to a waste-heat boiler. Steam from the latter is then passed through the regenerator chamber, wherein part of it is decomposed, and the mixture of  $\text{H}_2$  and superheated steam is introduced into the generator, together with a further amount of coal containing  $\text{Fe}_2\text{O}_3$ , and is maintained therein under a pressure of 30–300 lb./sq. in. for sufficient time to bring about combination of the  $\text{H}_2$  and C to form  $\text{CH}_4$  and other gaseous hydrocarbons. The gas produced has a calorific val. of not less than 400 B.Th.U./cu. ft. A. B. MANNING.

**Production of non-poisonous town gas.** H. COHN (B.P. 352,864, 7.11.30. Ger., 7.11.29).—The CO of town gas is converted into  $\text{CH}_4$  or  $\text{CO}_2$  ( $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$ , or  $\text{CO} + \text{H}_2\text{O} = \text{H}_2 + \text{CO}_2$ ) by passing the gas at a suitable temp. over a catalyst formed by depositing Ni, Fe, Cr, metals of group VIII, rare-earth metals, their oxides, or mixtures thereof, on  $\text{SiO}_2$  gel. A. B. MANNING.

**Manufacture of mixed hydrocarbon gas and water-gas.** HUMPHREYS & GLASGOW, LTD., Assees. of J. A. PERRY (B.P. 354,650, 13.11.30. U.S., 19.12.29).—Hydrocarbon gas from an extraneous source, e.g., natural gas, is cracked in an ignited fuel bed, and water-gas is produced by steaming the fuel bed. The gases are passed through a carburettor wherein the re-formed hydrocarbon gas is carburetted with oil so that the stiff pitch from the gas becomes mixed with fluid tar from the oil, the mixture being easily removable from the wash-boxes or other accessory apparatus. A. B. MANNING.

**Manufacture of acetylene by pyrogeneration of methane gas.** SOC. INDUSTR. DES HYDROCARBURES & DÉRIVÉS, Assees. of INTERNAT. INDUSTRIAL & CHEM. CO., LTD. (B.P. 352,688, 17.6.30. Fr., 19.6.29).—The process is carried out under the following conditions: (1) temp. 950–2000°, (2) abs. or partial pressure of  $\text{CH}_4$  varying from 760 mm. for 950° down to 25 mm. for 2000°, (3) duration of heating decreasing from about 15 sec. at 1000° to 0.01 sec. at 1500°, (4) continuous withdrawal by suction of the gases produced, (5) the ratio of surface to vol. of the apparatus increasing with rise of temp., being preferably 10:1 at about 1500°. A halogen or halogen compound may be used as a catalyst.  $\text{H}_2$ ,  $\text{C}_2\text{H}_4$ , C black, and liquid hydrocarbons are formed as by-products. A. B. MANNING.

**Performing endothermic gas reactions [e.g., production of acetylene by pyrolysis of methane].** T. S. WHEELER, D. BINNIE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 353,913, 26.2.30).—In the pyrolysis of gaseous hydrocarbons at high temp. and high space velocities the necessary heat is largely supplied by a simultaneous and independent exothermic reaction *in situ*, consisting of the formation of HCl from  $\text{H}_2$  and  $\text{Cl}_2$ . A. B. MANNING.

**Purification of gas and manufacture of fertilisers.** L. MELLERSH-JACKSON. From SOC. BELGE D'ELECTRO-SYNTHESE "SOBELSYN" SOC. ANON. (B.P. 354,302, 25.2.30).—Gas liquor is treated with  $\text{FeSO}_4$  and  $\text{K}_2\text{CO}_3$  or  $\text{K}_2\text{SO}_4$ , and, after heating, if desired, to drive off  $\text{NH}_3$  and  $\text{CO}_2$ , is used for washing the crude gas. The spent washing liquor is treated with sufficient  $\text{FeSO}_4$  to ppt. the sulphides therein, and, after decantation, is electrolysed in the anode compartment of an electrolytic bath. The anode liquor, which contains compounds of N, K, and P, per-salts, and synthetic humic acid, is utilised as a liquid fertiliser. The precipitated sulphides are roasted to regenerate  $\text{FeSO}_4$ ; the  $\text{SO}_2$  thereby evolved is supplied to the anode compartment of the bath to form  $\text{H}_2\text{SO}_4$ . A. B. MANNING.

**Gas purification process.** D. L. JACOBSON, Assr. to KOPPERS CO. (U.S.P. 1,792,097, 10.2.31. Appl., 9.10.26).—The gas is washed with an alkaline suspension of a compound of Fe, Ni, or Co, containing in solution an aromatic OH-compound, e.g., up to 2% of PhOH. The org. compound retards the formation of thiosulphates, and accelerates the oxidation of the metal sulphide by air during the revivification process. A. B. MANNING.

**Removal of sulphur from illuminating, coke-oven, and like gases.** J. Y. JOHNSON. From I. G.

FARBENIND. A.-G. (B.P. 351,975, 24.12.29).—The  $\text{H}_2\text{S}$  in the gas is catalytically converted, in known manner, into  $\text{SO}_2$ , which is then absorbed in aq.  $\text{NH}_3$  to form  $(\text{NH}_4)_2\text{SO}_3$ . The latter is heated in solution under pressure with the formation of  $(\text{NH}_4)_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{S}$ . The solution is then boiled or blown with steam to decompose the sulphide, the  $\text{NH}_3$  and  $\text{H}_2\text{S}$  evolved being returned wholly or in part to the gas, or worked up in any suitable manner.

A. B. MANNING.

**Treatment of gases containing hydrocarbons.** T. S. WHEELER, W. FRANCIS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,994, 29.3.30).—The gases are mixed with a minor proportion of S or  $\text{H}_2\text{S}$  and passed through a reaction zone at a high temp., *e.g.*, about  $1000^\circ$ , and with a high space velocity, *e.g.*, over 100 reciprocal min., under conditions giving high yields of  $\text{C}_6\text{H}_6$  and  $\text{CS}_2$ . Natural gases containing S or reactive S compounds may be similarly treated.

A. B. MANNING.

**Treatment of gas mixtures containing saturated hydrocarbons.** SOC. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTR., Assees. of E. VOITURON (B.P. 352,280, 29.7.30. Ger., 29.7.29. Addn. to B.P. 349,067; B., 1931, 832).—The process of the prior patent is applied to gases which are relatively poor in saturated hydrocarbons, and gases containing also  $\text{H}_2$ , *e.g.*, coke-oven gases.

A. B. MANNING.

**Dehydration of gas.** F. W. SPERR, JUN., ASSR. to KOPPERS Co. (U.S.P. 1,791,086, 3.2.31. Appl., 11.10.26).—The gas is compressed, cooled to room temp., and treated while under pressure with a hygroscopic material, *e.g.*,  $\text{H}_2\text{SO}_4$  or  $\text{CaCl}_2$  solution. When  $\text{H}_2\text{SO}_4$  is used the diluted acid is utilised in removing  $\text{NH}_3$  from the gas.

A. B. MANNING.

**Apparatus for detecting and indicating the presence of inflammable gases in air.** C. MCLUCKIE (B.P. 352,267, 15.7.30).—A spiral bimetallic strip is supported by one end above the flame of a miner's lamp. In the presence of inflammable gases or vapours which burn in the region of the flame the additional heat causes the metal to expand, and the free end of the strip to move and make contact with a pin, thereby completing an electric circuit which operates a suitable warning device.

A. B. MANNING.

**Determination and indication at a distance of the presence and amount of combustible gases in atmospheric air.** W. W. TRIGGS. From VULKANWERKE GES.M.B.H. (B.P. 352,817, 20.9.30).—The air is passed over a contact substance, formed by precipitating Pt or Pd on  $\text{SiO}_2$  gel, thereby bringing about the oxidation of the combustible gases. The heat developed operates a differential air thermometer, and the movement of a thread of liquid in the capillary tube thereof serves to indicate the presence and amount of the combustible gases. By using a Hg thread and having fused-in contact wires in the capillary the apparatus may be made to close an electric circuit operating an alarm device.

A. B. MANNING.

**Distillation of tar.** BARRETT Co., Assees. of S. P. MILLER (B.P. 354,591, 18.9.30. U.S., 25.9.29).—The gas collector main of a coal-distillation plant is flushed

with tar, and at the same time the walls of the main are sprayed with tar, whereby the accumulation of pitch and coke within the main is prevented. The gases are removed from the main at a temp. sufficiently high to carry with them the vapours of high-boiling constituents originally present in the gas as well as those distilled from the tar. The gases leaving the main are cleaned at a high temp. in an electrical precipitator or scrubber, and are then cooled to separate clean oils therefrom.

A. B. MANNING.

**Manufacture of bituminous emulsions.** L. R. MacKENZIE (U.S.P. 1,795,226, 3.3.31. Appl., 30.10.30).—The molten bitumen and the emulsifier, at a temp. slightly below the b.p. of the latter, are fed continuously into a mixing chamber to form an emulsion with bitumen as the external phase; this emulsion is removed immediately from the zone of formation and agitated to form an emulsion with bitumen as the internal phase, the relative vols. of the materials remaining const. The emulsion may then be subjected to grinding action between close converging surfaces, and is finally centrifuged and cooled.

A. B. MANNING.

**Emulsification of asphalt.** R. K. PAINTER, ASSR. to BITUTECH, INC. (U.S.P. 1,793,957, 24.2.31. Appl., 1.2.30).—The asphalt is dissolved in a solvent, *e.g.*,  $\text{CCl}_4$ , and to the solution is added an aq. solution of colloidal clay. A small amount of  $\text{AcOH}$  or other org. acid is added, and the asphalt is precipitated in the presence of the colloidal clay, *e.g.*, by the addition of  $\text{COMe}_2$ , while the whole is agitated. The process is then continued by introducing melted asphalt together with a dil. aq. solution of an org. acid into the mixture and drawing off the emulsified asphalt.

A. B. MANNING.

**Production of dispersions [of bituminous substances].** J. M. FAIN, ASSR. to FLINTKOTE Co. (U.S.P. 1,793,918, 24.2.31. Appl., 21.11.28).—Relatively stable emulsions are produced by making an aq. suspension of a paste-forming colloidal material, *e.g.*, bentonite, adding thereto a  $\text{H}_2\text{O}$ -sol. agent, *e.g.*, soap or saponin, which reduces the surface tension of the medium, and finally adding the material to be emulsified, *e.g.*, bitumen, and dispersing it by relatively light agitation.

A. B. MANNING.

**Distillation of shale and other oleiferous minerals.** H. WERNER (B.P. 354,962, 14.7.30).—Oil shale etc., in the form of grains of various sizes, are compressed into a retort or perforated container fitting into a retort, and channels, which may be filled with coarser material, are formed by withdrawable cores, in order that the gaseous products of distillation are not overheated by contact with the retort walls. A utilisable coke may be obtained by adding at least 50% of fuel to the material.

D. K. MOORE.

**Distilling apparatus and method [for mineral oils etc.].** E. O. BENJAMIN (U.S.P. 1,784,956, 16.12.30. Appl., 28.7.24).—Oil (or other liquid) is passed through a succession of retorts in each of which it is maintained at temps. higher and pressures lower than those of the preceding retort. In each retort a rapid oil circulation is maintained, independent of inter-retort circulation by convection currents, by providing a separate relatively

cool downflow path from the upper to the lower part of the retort. The vapours from each retort pass to an associated fractional condenser maintained at considerably lower temps. and pressures. The heavier fractions are sprayed by the injector action of higher-pressure gases into a succeeding retort at a higher temp. and lower pressure than that from which they originate, in order to effect rapid cracking, the differences both in pressure and in temp. being substantial and continuously maintained during normal operation. Uncondensed gases are returned to the vapour space of each retort separately, thereby maintaining intercondenser vapour flow in a direction countercurrent to that of the inter-retort oil flow. H. S. GARLICK.

**Tube still for oil distillation and cracking.** H. THOMAS, Assr. to SUN OIL Co. (U.S.P. 1,788,213, 6.1.31. Appl., 14.6.27).—A still consisting of an upright gas-combining and heating chamber with a bank of oil tubes in the upper part and a series of laterally disposed Dutch ovens communicating with the lower part of the chamber below the tubes is described. Cooled gases from the upper part of the chamber are removed through a series of pipes communicating with a stack connected to a conduit along which extend ducts communicating with the rear ends of the Dutch ovens, and through which the cooled gases are forced by means of fans to a main stack. A fresh-air conduit is in heat-exchange relation with the duct conveying hot gases to the main stack, and communicates with the forward ends of the ovens. H. S. GARLICK.

**Cracking of mineral oil.** A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL Co. (U.S.P. 1,786,315, 23.12.30. Appl., 24.2.26).—Oil is preheated and continuously passed through a fractionating zone (A) to a cracking zone (B), from which it is circulated through a second cracking zone (C) back to (A). Hg vapours are passed in heating relation with (C) in order to effect condensation and thereby impart sufficient heat to the oil to decompose the higher-boiling constituents and to continue the cracking in (B). Vapours from (B) are then fractionated and the light vapours separated and withdrawn. The heavier vapours are condensed and the condensate is returned with the inflowing preheated oil to the cracking system. H. S. GARLICK.

**Pyrolysis [of hydrocarbon oils].** N. E. LOOMIS, A. H. TOMLINSON, and F. A. HOWARD, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,785,274, 16.12.30. Appl., 31.5.23).—A stream of hydrocarbon oil, after traversing a heating coil under such conditions of temp., pressure, and time as to give substantial cracking and vaporisation, is passed through a digesting zone in which the oil is held for a relatively longer time and into which sufficient cool oil is introduced to maintain the temp. lower than that in the heating coil, but not below an effective cracking temp. All the fluid products are removed together from the digestion zone. H. S. GARLICK.

**Oil-refining process and apparatus.** I. B. FUNK (U.S.P. 1,789,173, 13.1.31. Appl., 2.10.26).—A mixture of light and heavy hydrocarbons, e.g., absorption oil containing natural-gas gasoline, is vaporised and mixed with steam. The mixture is fractionated to eliminate heavy hydrocarbons and the light hydrocarbons and

steam are condensed in two stages. A portion of the condensate is continuously cycled through the vapours of the light hydrocarbons in the first condensing stage, from which the  $H_2O$  is removed from admixture with the reflux, the final condensation taking place at below  $100^\circ$ . H. S. GARLICK.

**Recovery of gasoline from gas.** N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,789,470, 20.1.31. Appl., 25.11.25).—Natural gas, still gas, etc. is cooled and continuously passed into an absorption tower, maintained under a pressure of 30 lb. abs., into which a suitable preheated absorption liquid is introduced at an intermediate point. Gas and vapour flow upward countercurrent to the absorption stock, only the vapours being absorbed owing to the preheating of the oil. In the upper half of the tower the rising gas and vapours are washed by a stream of fresh absorption oil. The charged oil from the tower is passed to a steam still for recovery of gasoline constituents, and the stripped oil passed through heat exchangers and coolers and returned to the absorption tower. H. S. GARLICK.

**Reclamation of [petroleum] residues.** A. E. BECKER and R. G. SLOANE, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,785,242, 16.12.30. Appl., 28.11.23).—The heavy fluid residue resulting from the distillation of petroleum containing org. acid compounds in the presence of NaOH is thinned out with naphtha or other relatively low-boiling solvent, washed with  $H_2O$  to remove sol. naphthenates (which are subsequently recovered from the wash-water), and the remaining residue treated in the presence of the solvent with 50 lb. of dil.  $H_2SO_4$  (d 1.33) per barrel. After settling and removing the acid sludge, the resultant product is washed with aq. NaOH and  $H_2O$ , and the solvent distilled off. H. S. GARLICK.

**Dewaxing of petroleum oil.** L. D. JONES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,784,525, 9.12.30. Appl., 7.10.25).—Oil containing cryst. and amorphous wax, hard asphalt, and naturally occurring "amorphising" impurities is subjected to non-cracking distillation. The distillate has a lower hard-asphalt content than the original oil and contains sufficient "amorphising" impurities to cause all of the wax precipitated on reduction of temp. to be in the amorphous form. The distillate is subjected to mild acid treatment to remove asphaltic impurities that interfere with wax precipitation, and the temp. of the acid-treated distillate lowered sufficiently to precipitate the wax therefrom, which is removed by centrifugal means. The dewaxed oil is then treated by known means to remove remaining impurities and colour. H. S. GARLICK.

**Production of hydrocarbons by destructive hydrogenation.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,181, 28.4. and 12.5.30).—Hydrocarbon oils, e.g., heavy benzines, the boiling ranges of which lie principally within the limits  $60^\circ$  and  $325^\circ$ , are hydrogenated in the vapour phase and in the presence of catalysts, under conditions which maintain the partial pressure of the desired products (b.p. below  $200^\circ$ ) greater than 10% of the total pressure. The process is carried out at  $500$ – $650^\circ$  and under pressures of at



least 50 atm. The initial oil is preferably subdivided into fractions of relatively narrow boiling range, *e.g.*, 20–30°, and each fraction is treated separately. The catalyst, *e.g.*, molybdc or tungstic acid mixed with ZnO and/or MgO, may be activated by the addition of a small proportion of Re or one of its compounds.

A. B. MANNING.

**Manufacture of lubricating oils.** STANDARD OIL Co., Assecs. of F. W. SULLIVAN, JUN., and V. VOORHEES (B.P. 355,214, 18.2.30. U.S., 15.3.29).—Hydrocarbon material containing 30% or more of hydrocarbon wax, preferably a crude wax product containing > 80% of hydrocarbon wax, is subjected to vapour-phase cracking at 400–600° under pressure (< 100 lb./sq. in.). The cracked products are fractionated to obtain a substantially wax-free distillate, which is subjected to the action of a polymerising agent, preferably  $\text{AlCl}_3$ , at a suitable temp., *e.g.*, by agitation at 37–121° for 15–20 hr. The lighter distillates are separated from the polymerised product and the remainder is distilled to produce lubricating oils of remarkably low cold test, low Conradson C content, and temp.-viscosity characteristics approximating those of Pennsylvania oils.

H. S. GARLICK.

**Refining of crude petrol.** IMPERIAL CHEM. INDUSTRIES, LTD., W. R. MADEL, and E. W. FAWCETT (B.P. 355,212, 18.2.30).—Petrol containing phenols, *e.g.*, that obtained from the hydrogenation of carbonaceous materials, is cooled until it separates into two layers (–40°), and the lower layer rich in phenols, bases, gummy matter, and associated impurities is removed. The best separation is obtained when the petrol is rich in paraffins, and the cooling may advantageously be conducted in the presence of porous materials to absorb the gummy matter. Alternatively, the crude petrol may be distilled and the fraction boiling at 170–200°, which contains the greater part of the phenolic constituents, subjected to cooling as above. H. S. GARLICK.

**Purification of crude hydrocarbons or their derivatives.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,164, 17.5.30).—The crude hydrocarbons, *e.g.*, benzols, are refluxed or heated under pressure with 1–3% of maleic acid or its anhydride.

A. B. MANNING.

**Manufacture of wax compositions.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 354,782, 6.5.30).—Waxes and creams suitable for floor polishes may be obtained by melting wax, *e.g.*, montan, carnauba, paraffin, with or without the addition of wax-like substances prepared according to B.P. 296,145 and B.P. 324,631 (B., 1928, 779; 1930, 519), and adding a hydroxyalkylamine and/or its derivatives, an org. solvent, *e.g.*, turpentine, and a liquid in which the waxy substance is insol., *e.g.*,  $\text{H}_2\text{O}$ .

D. K. MOORE.

**[Levelling bars for] coke ovens.** GAS CHAMBERS & COKE OVENS, LTD., and N. J. BOWATER (B.P. 356,036, 12.11.30).

**Apparatus for cooling of coke [by quenching].** R. WAGNER (B.P. 355,558, 29.9.30. Ger., 7.10.29).

**[Generator ash-door locking device for] water-gas apparatus.** HUMPHREYS & GLASGOW, LTD., and E. C. MENGEL (B.P. 352,411, 7.4.30).

**Generating acetylene.** IMPERIAL CHEM. INDUSTRIES, LTD., and J. SAVAGE (B.P. 355,875, 24.6.30).

**Atomisation of oils.** S. D. ROSS and C. F. EDWARDS (B.P. 355,255, 12.3.30. Can., 11.6.29).

**Chemical reactions with arc discharges.** Separation of solids by flotation. Centrifugal separators. Prefiltering means. Separation of mixed liquids.—See I. Bituminous pulps. Cardboard.—See V. Decolorising agent for gasoline.—See VII. Bitumen mixtures. Materials for roads etc.—See IX. Water-purifying material. Impregnating gas.—See XXIII.

### III.—ORGANIC INTERMEDIATES.

**Catalytic gas reactions in a liquid medium.** F. FISCHER and K. PETERS (Brennstoff-Chem., 1931, 12, 286–293).—The catalytic hydrogenation and polymerisation of  $\text{C}_2\text{H}_2$ , and other gas reactions, have been carried out in an inert liquid medium, *e.g.*, paraffin oil of high b.p., in which the catalyst was suspended, the gas mixture being bubbled through the medium contained in an electrically-heated, vertical reaction tube. With  $\text{C}_2\text{H}_2 : 2\text{H}_2$  mixtures and a Ni-kieselguhr catalyst the temp.-vol. contraction curves pass through a max. at about 200°, corresponding with practically complete hydrogenation and/or polymerisation of the  $\text{C}_2\text{H}_2$ . The curves are similar to those obtained with a "dry" catalyst, but have much sharper maxima, due to the greater uniformity of temp. which can be maintained in a liquid medium. Below 200° the principal products are  $\text{C}_2\text{H}_6$  and  $\text{C}_2\text{H}_4$ ; at higher temps. appreciable quantities of higher hydrocarbons are formed. With a Ni-Th-Al-kieselguhr catalyst the yield of liquid hydrocarbons is much higher, corresponding at 200° with almost 80% of the  $\text{C}_2\text{H}_2$ . With a Cu catalyst a high proportion of the  $\text{C}_2\text{H}_2$  is converted into cuprene. Other reactions which have been carried out successfully in a liquid medium with the Ni-Al-Th catalyst are the hydrogenation (almost quant.) of  $\text{C}_2\text{H}_4$  to  $\text{C}_2\text{H}_6$ , and the reduction of CO by  $\text{H}_2$  to  $\text{CH}_4$  and higher gaseous paraffins.

A. B. MANNING.

**Preparation of ethylene dichloride.** E. A. DOROGANEVSKAJA (J. Chem. Ind. Russ., 1931, 8, 857–860).— $\text{Cl}_2$  does not combine with  $\text{C}_2\text{H}_4$  in the absence of a catalyst, such as glass. The reaction in glass vessels is greatly accelerated by the addition initially of  $\text{C}_2\text{H}_4\text{Cl}_2$ , which acts as a solvent for the substrates. Under suitable conditions of temp., concentration of gases, velocity of flow of gases, etc. 93·6% yields are obtained; these conditions differ for each apparatus used.

R. TRUSZKOWSKI.

**Preparation of tetrachloroethane and trichloroethylene.** M. J. KRAFT and B. A. ALEXEEV (J. Chem. Ind. Russ., 1931, 8, 861–863).—A mixture of  $\text{C}_2\text{H}_2$  and  $\text{Cl}_2$  is passed up a column the upper part of which is packed with  $\text{Fe}_2\text{O}_3$  grains (15 mm. diam.) and down which flows a current of  $\text{C}_2\text{H}_2\text{Cl}_4$  containing 0·01% of  $\text{SbCl}_5$ , heated at 30°. Under these conditions very pure  $\text{C}_2\text{H}_2\text{Cl}_4$  is obtained, without risk of explosion; only traces of Sb are needed.

R. TRUSZKOWSKI.

**Catalysts for synthesis of methyl alcohol.** V. N. IPATIEV and B. N. DOLOV (J. Chem. Ind. Russ., 1931,

8, 825—829).—The velocity of decomp. of MeOH at 350° in the presence of a Zn—Cr carbonate catalyst is inversely proportional to the rate at which the vapour is passed. The catalytic action of  $\text{Zn}(\text{HCO}_3)_2$  with or without Cr carbonate increases with the temp. at which it was precipitated. The action of ZnO obtained by precipitation of  $\text{ZnCO}_3$  with  $\text{K}_2\text{CO}_3$  or  $\text{Na}_2\text{CO}_3$  and subsequent heating in a reducing atm. is greater at above 350° than is that of ZnO prepared by similar treatment of  $\text{Zn}(\text{OH})_2$  precipitated by KOH or NaOH. More active catalysts are obtained by precipitation by more dil. than by more conc.  $\text{Na}_2\text{CO}_3$  solutions. The most active ZnO catalysts are obtained by precipitation on asbestos. The activity of ZnO catalysts falls with keeping. The addition of small quantities of U, Th, or Ce increases the activity of Zn and Zn—Cr catalysts.

R. TRUSZKOWSKI.

**Refraction of aqueous ethyl and methyl alcohols of sp. gr. approximately 0.94.** R. H. FIELD, M. W. FAIRN, and J. M. MACOUN (J.S.C.I., 1931, 50, 283—286 r).—Curves have been prepared correlating the sp. gr. and refractometer readings (as shown by the immersion refractometer) of aq. EtOH and of aq. MeOH over a narrow range of sp. gr. (0.9380—0.9400), the temp. range being from 18° to 22°, taking intervals of 1°. A similar curve has also been prepared for the temp. 15–6°. The degree of accuracy claimed is the highest of which the instrument used is capable, the object of the work being to furnish data for routine official and commercial analyses.

**Determination of ethyl and methyl alcohols in aqueous solutions by the immersion refractometer.** J. M. MACOUN (J.S.C.I., 1931, 50, 281—283 r).—A weighed distillate containing only  $\text{H}_2\text{O}$ , EtOH, and MeOH having been obtained, it is brought, by addition of the calc. quantity of alcohol or of  $\text{H}_2\text{O}$ , as closely as possible to a concentration of 40% total alcohol by wt. The sp. gr. and refractive power, as measured by an immersion refractometer, are then determined. From these data the proportion of each constituent of the original distillate is determined with the help of a table (see preceding abstract).

**Determination of small proportions of butyl chloride and ethyl phthalate in ethyl alcohol.** R. W. HOFF (J.S.C.I., 1931, 50, 244 r).—A 100-c.c. sample is slowly distilled till 70 c.c. have passed over, carrying all the BuCl. Et phthalate is determined in the residue by its "ester value." The BuCl in the first distillate is separated and measured as in the Babington—Tingle method (B., 1919, 509 A). This method has application in the analysis of a certain Canadian officially "denatured" alcohol.

**Determination of small proportions of hydrocarbon in alcohol containing acetone.** R. W. HOFF (J.S.C.I., 1931, 50, 242—244 r).—The acetone is first converted into relatively non-volatile derivatives prior to distillation of the hydrocarbon from the diluted mixture. Different treatments are necessary according to the volatility of the hydrocarbon present. For higher-boiling hydrocarbons, e.g., benzene and solvent naphtha, a known volume (100 c.c.) of the mixture is digested under a reflux with Al—Hg in presence of

alkali. The hydrocarbon is then separated and measured as in the Babington—Tingle method for  $\text{C}_6\text{H}_6$  (B., 1919, 509 A). For a lower-boiling hydrocarbon, e.g.,  $\text{C}_6\text{H}_6$ , 100 c.c. of the mixture are treated with  $\text{NH}_2\text{OH}$  in presence of alkali. After dilution, the hydrocarbon is separated and measured as before. These methods have application in the analysis of certain Canadian officially "denatured" alcohols.

**anti-Diazotates and their use for the preparation of azo dyes in substance and on the fibre.** H. T. BUCHERER and E. MÖHLAU (J. pr. Chem., 1931, [ii], 131, 193—258).—The rate of coupling of *anti*-diazotates with phenols is influenced by substituents in the same direction and to the same degree as the rate of isomerisation of the *syn*- to the *anti*-forms, and of the latter to the nitrosoamine. Dyestuff formation takes place in a solution which never becomes acid, and consequently must be due to reaction between the phenol and the *anti*-diazotate (and not the nitrosoamine, or *syn*-form). A mixture of *anti*-diazotate and phenolic component, stabilised by excess NaOH, can be printed on cotton, the colour being developed either by addition of an alkali-binding substance to the solution shortly before printing, or by previously steeping the cloth in the alkali-binding substance. In the former process reaction is hastened by steaming the printed cloth. The second method is, however, preferable, because there is no tendency for dyestuff to form in the solution on keeping, and because it is easier to control.  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{OAc}$ , or  $\text{NaHCO}_3$  can be used for removal of excess alkali, excess of the salts being subsequently washed out. Gypsum or  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  is unsatisfactory. Under suitable conditions, *anti*-diazotates give excellent dyeings with naphthols and aminonaphthols (pure hydroxyazo dyes being produced in the latter case), but reaction with naphthylamines is rather slow. Na *p*-nitrobenzene-*anti*-diazotate (I) with K-acid gives a pure *o*-hydroxyazo dye, with  $\gamma$ - and J-acids mixtures of *o*- and *p*-hydroxyazo dyes (the proportions being variable according to the acidity of the solution), and with  $\beta$ -naphthol-1-sulphonic acid (II) the *o*-hydroxyazo dye derived by replacement of the  $\text{SO}_3\text{H}$  group by the azo component. Diazotised *p*-nitro-*o*-anisidine reacts with (II) in  $\text{NaHCO}_3$  solution in the same way as does (I), an unstable salt being formed between the phenolic OH group and the azo group, passing into the dyestuff by replacement of the  $\text{SO}_3\text{H}$  group by the azo component. Diazotised  $\text{NH}_2\text{Ph}$  reacts slower, but possibly similarly with (II). The speed of reaction of *p*-nitrobenzenediazonium chloride (III) with naphtholsulphonic acids is greater in  $\text{NaHCO}_3$  or  $\text{NaOAc}$  than in HCl solution. (I) reacts more slowly than (III), but quantitatively and fairly fast at 90° in  $\text{Na}_2\text{CO}_3$  or  $\text{NaOAc}$  solution; in the presence of a large excess of alkali the reaction does not go to completion even on warming. (I) couples with  $\beta$ -naphthol-6-sulphonic acid faster than with the 8-sulphonic acid, and with  $\beta$ -naphthol-3:6-disulphonic acid faster than with the 6:8-disulphonic acid. (II) couples with (I) only slowly in hot alkaline solution, elimination of the  $\text{SO}_3\text{H}$  group, which is necessary for coupling of this acid, proceeding only slowly unless acid is present. The dyes from (I) and naphthol-7-sulphonic acids are yellowish-red to red *o*- or *p*-hydroxyazo dyes (the former

being less sol.), which give a red solution in  $\text{H}_2\text{SO}_4$ , becoming yellow on dilution. This colour change is not shown by the dyes from (I) and naphthylaminesulphonic acids. (III) reacts with ten naphthylaminesulphonic acids fastest in dil. acid solution, but still only slowly with  $\beta$ -naphthylamine-8-mono- and -4:7-di-sulphonic acids owing to steric hindrance; *o*- or *p*-aminoazo dyes are formed in these cases, except with  $\beta$ -naphthylamine-4:8-disulphonic acid, which forms a diazoamino-compound, decomposed by 80%  $\text{H}_2\text{SO}_4$  into its components. Naphthylaminemonosulphonic acids couple faster than the disulphonic acids with (III). (I) couples only slowly with naphthylaminesulphonic acids. Aminonaphtholsulphonic acids (8 examples) couple with (I) in strongly alkaline solution to give hydroxyazo dyes, and with (III) in strongly acid solution to give aminoazo dyes, mixtures being usually formed in solutions of intermediate acidity. With these acids also (I) couples more slowly than (III). The stability of aq. solutions of (I) is decreased by  $\text{Na}_2\text{CO}_3$  or  $\text{NaOAc}$ , more so by  $\text{NaHCO}_3$ ,  $\text{CaCl}_2$ , or  $\text{MgCl}_2$ , but is increased by  $\text{NaOH}$ . Only  $\text{MgCl}_2$  affords more than a trace of *p*-nitroaniline.  $\text{HNO}_2$  is formed only in aq. solution. When the aq. solution is heated some *p*-nitrophenol is formed, whereas with cold  $\text{NH}_4\text{Cl}$  solution much *p*-nitroaniline, together with (possibly) some diazoamino-compound, is obtained (cf. A., 1909, i, 272). (I) is stable for 8 days in a solution containing 1.8% of  $\text{NaOH}$ .  $\beta$ -Naphthol, naphthol AS, and naphthol AS-G couple normally with (I) in neutral solution at room temp. or  $90^\circ$ , and with (III) in dil.  $\text{AcOH}$  at room temp., the first-mentioned naphthol most readily with (III), the last most rapidly with (I).  $\alpha$ -Naphthol couples normally with (III), but with (I) gives a reddish-violet substance (changed to blue by  $\text{HCl}$ ), which on warming with  $\text{NaOH}$  passes into the normal blue salt (changed to reddish-brown by  $\text{HCl}$ ). 2-Aceto- $\alpha$ -naphthol couples normally, though slowly, with (III), but with (I) at  $90^\circ$  rapidly gives a dirty bluish-violet substance, unchanged by boiling with  $\text{NaOH}$ . *p*-Phenylenediamine catalytically decomposes (III), but with (I) in hot or cold neutral solution gives brown crystals with a bluish reflex. Whereas a solution of  $\text{NaOH}$  saturated with  $\beta$ -naphthol oxidises on keeping, excess of  $\text{NaOH}$  delays decomp., which is then not brought about by addition of  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ , or  $\text{NH}_4\text{Cl}$ .  $\beta$ -Naphthol is thus suitable for the printing process. The rate of coupling of (I) with  $\beta\text{-C}_{10}\text{H}_7\cdot\text{ONa}$  in 2% solution is decreased by addition of  $\text{NaOH}$ , 1.6% of the latter rendering the mixture stable for 1 day. This corresponds approx. with commercial "Rapidecht-Bordeaux B," which is, however, slightly less stable. Larger concentrations of  $\text{NaOH}$  are necessary to confer stability at  $90^\circ$ . Mixtures of *p*-nitroanisyl-anti-diazotate (IV) and  $\beta\text{-C}_{10}\text{H}_7\cdot\text{ONa}$  or naphthol AS behave similarly, 2 and 2.5%, respectively, of  $\text{NaOH}$  being necessary to confer stability for 1 day at room temp. The violet dye formed from (IV) in the presence of alkali is anomalous, being only slowly affected by  $\text{HCl}$ . Dyes from (IV) are best prepared by printing on cotton previously impregnated with  $\text{MgCl}_2$ . The monoazo dye from diazotised naphthionic acid and *m*-phenylenediamine couples with (III) in the presence of  $\text{NaOAc}$  to give a disazo dye, and slowly a trisazo

dye. The polyazo dyes can be separated by 10%  $\text{NaCl}$  solution, in which the monoazo dye only is sol.; the trisazo dye only is insol. in hot  $\text{H}_2\text{O}$ . The monoazo dye couples slowly with (I) in presence of  $\text{Na}_2\text{CO}_3$ , the mixture being stabilised by 1%  $\text{NaOH}$ . The disazo dye is best printed from neutral solution on cotton previously impregnated with  $\text{NaHCO}_3$ . A method is described for identifying nitrogen B (the condensation product of 1:8-diaminonaphthalene-4-sulphonic acid and  $\text{COMe}_2$ ), which is best purified through its toluidine salt. This acid couples readily with (III) in  $\text{NaHCO}_3$  solution at room temp. to give successively a sol. mono- and an insol. dis-azo dye, distinguishable by their reddish-violet and green colours, respectively, in  $\text{H}_2\text{SO}_4$ . In dil.  $\text{HCl}$  only a monoazo dye is formed. Mixtures of nitrogen B and (I) are stabilised for 1 day at room temp. by 0.7–1% of  $\text{NaOH}$ . Even without excess  $\text{NaOH}$  and at  $90^\circ$  formation of the disazo dye from (I) is slow and incomplete. The disazo dye can best be printed from neutral solution on cotton impregnated with  $\text{NaHCO}_3$ , and is a satisfactory deep black dye. (II) reacts rapidly with (III) in  $\text{NaHCO}_3$  solution, the  $\text{SO}_3\text{H}$  group being replaced by the diazo component, and a reliable method of determining (II) is based on this reaction. (I) reacts in the same way as (III), but not smoothly. Benzenediazonium chloride rapidly, and *p*-nitroanisyl-diazonium chloride slowly, give similar dyes, the product in the former case being unstable.

R. S. CAHN.

**Friedel-Crafts reaction. Preparation of 2-amino-anthraquinone from phthalic anhydride and bromobenzene.** P. H. GROGGINS, A. J. STIRTON, and H. P. NEWTON (Ind. Eng. Chem., 1931, 23, 893–899).—A semi-technical investigation. The use of pure instead of technical  $\text{AlCl}_3$  offers no advantages in the prep. of 4'-bromo-*o*-benzoylbenzoic acid from phthalic anhydride and  $\text{PhBr}$ ; the lower yields obtained compared with those of the corresponding  $\text{Cl}$ -derivative appear to be due to the conversion of  $\text{PhBr}$  into  $\text{C}_6\text{H}_6$  and  $\text{C}_6\text{H}_4\text{Br}_2$ . The yield of 2-bromoanthraquinone obtained by cyclisation of the ketonic acid with conc.  $\text{H}_2\text{SO}_4$  varies inversely, and its purity varies directly, with the final degree of dilution during precipitation. The conversion of 2-bromo- into 2-amino-anthraquinone is best carried out with a large excess of aq.  $\text{NH}_3$  at  $165^\circ$  in presence of  $\text{Cu}$  or a  $\text{Cu}$  compound as catalyst, and proceeds much more readily than with 2-chloroanthraquinone. The use of  $\text{Ni}$ ,  $\text{Co}$ , or  $\text{Ag}$  salts as catalysts is impracticable and leads to highly impure products, but  $\text{PhNO}_2$  equal in amount to that of 2-bromoanthraquinone taken is almost equal in efficiency to  $\text{Cu}$  compounds.

H. A. PIGGOTT.

**Low-temp. tar constituents. Pyrolysis of  $\text{C}_3\text{H}_8$ .**—See II. Cyanide.—See VII. Furfuraldehyde.—See XV.  $\text{PhOH}$  from coal-tar distillery water.—See XXIII.

#### PATENTS.

**Separation of aliphatic amines.** I. G. FARBERMIND. A.-G. (B.P. 350,539, 12.3.30. Ger., 12.3.29).—An aromatic aldehyde ( $\text{PhCHO}$ ) is added to the mixture of amines, *sec*- and/or *tert*-amine is distilled off, and the primary amine is recovered from its arylidene compound.

C. HOLLINS.

**Production of acetic acid [from acetaldehyde].** C. F. BOEHRINGER & SÖHNE G.M.B.H. (B.P. 350,874, 10.11.30. Ger., 15.11.29).—In the catalytic oxidation of liquid  $\text{MeCHO}$  by  $\text{O}_2$ , the  $\text{MeCHO}$  in the  $\text{AcOII}$  produced is oxidised by a countercurrent of  $\text{O}_2$  in presence of catalyst, whilst the  $\text{MeCHO}$  vapour from the main reaction vessel is similarly oxidised by the  $\text{O}_2$  mixed with it, the whole being thus effected in a series of catalytic reaction vessels in which  $\text{O}_2$  passes in countercurrent to liquid  $\text{MeCHO}$  and with  $\text{MeCHO}$  vapour.

C. HOLLINS.

**Preparation of granular anhydrous citric acid.** R. PASTERNAK and F. G. BREHMER, Assrs. to C. PFIZER & Co. (U.S.P. 1,792,657, 17.2.31. Appl., 26.9.29).—The cryst. acid is heated in layers of about 1 in. in a current of air at 35–50°, preferably raised slowly from 35° to 45° during the process.

L. A. COLES.

**Manufacture of anhydrides of aliphatic halogen acids.** I. G. FARBENIND. A.-G. (B.P. 350,867, 20.10.30. Ger., 19.10.29).—A solution or suspension of a salt of the acid (Na mono- or tri-chloroacetate, Ca di- or tri-chloroacetate) in liquid ester or acid anhydride ( $\text{EtOAc}$ ,  $\text{EtOBz}$ ,  $\text{Et oxalate}$ ,  $\text{Ac}_2\text{O}$ ) is treated at 15–50° with  $\text{SO}_2\text{Cl}_2$ , or  $\text{SO}_2$  and  $\text{Cl}_2$ , or  $\text{Cl}_2$  and a carrier, or other acid chlorides ( $\text{COCl}_2$ ).

C. HOLLINS.

**Dehydration of organic liquids [alcohol].** Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 350,473, 6.3.30. Fr., 26.8.29).—Dil.  $\text{EtOH}$  (etc.) is distilled with an entraining liquid ( $\text{C}_6\text{H}_6$ -petrol) through a column comprising an extraction zone, I, a concentrating zone, II, and an azeotropic distillation zone, III. Between II and III the  $\text{EtOH}$  condenses at greater concentration than that of the azeotropic  $\text{EtOH-H}_2\text{O}$  mixture and a portion is here withdrawn to a small column with surface-heated plates, where the small content of  $\text{H}_2\text{O}$  and entrainer is removed and 97.5–99.5%  $\text{EtOH}$  recovered. At the top of III the condensate approaches the ternary mixture,  $\text{EtOH-H}_2\text{O}$ -entrainer; the upper layer is decanted and refluxed to the top of III, the lower layer being returned at an appropriate point to II. Suitable apparatus is described.

C. HOLLINS.

**Purification of alcohols.** IMPERIAL CHEM. INDUSTRIES, LTD., J. W. ARMIT, and G. E. WAINWRIGHT (B.P. 350,502, 13.12.29).—The alcohol is distilled with alkali to remove acids, a primary amine of b.p. above that of the alcohol mixture is added to combine with aldehydes and ketones, together with  $\text{H}_3\text{PO}_4$  or  $\text{H}_2\text{SO}_4$  to hydrolyse acetals, and the mixture is fractionated. Brine may be added with the alkali, and preferred amines are  $\text{NH}_2\text{Ph}$  and sulphanilic acid.

C. HOLLINS.

**[Manufacture of] nitrated esters of polyhydric alcohols.** C. M. A. STINE and C. E. BURKE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,792,515, 17.2.31. Appl., 18.10.24. Renewed 22.3.29).—The compounds consist of nitrated esters of lactic acid and a polyhydric alcohol having 2–4 OH groups, e.g., glycerol or ethylene glycol.

W. J. WRIGHT.

**Manufacture of thiuram monosulphides.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 350,564, 14.3.30).—A dithiocarbamate or mixture of amine,  $\text{CS}_2$ , and alkali is treated with  $\text{COCl}_2$ , whereby thiuram

monosulphide,  $\text{COS}$ , and metal chloride are produced without any disulphide. The prep. of tetramethyl-, bis-pentamethylene-, and dicyclohexyldiethyl-thiuram monosulphides is described.

C. HOLLINS.

**Production of disubstituted tetrazoles.** KNOLL A.-G. CHEM. FABR. (B.P. 350,782, 18.7.30. Ger., 20.5.30).—Chlorinated tetrazoles, produced by successive action of  $\text{PCl}_5$  and  $\text{N}_3\text{H}$  on lactams (e.g.,  $\zeta$ -leucine lactam) are reduced, e.g., with Zn and  $\text{AcOH}$ , to give Cl-free tetrazoles.

C. HOLLINS.

**Purification [increasing the sp. resistivity] of nitrobenzene.** S. BOYER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,793,304, 17.2.31. Appl., 19.2.27. Renewed 16.2.29).— $\text{PhNO}_2$ , or other liquid material used for insulation, is filtered successively through fuller's earth and a basic oxide ( $\text{Al}_2\text{O}_3$ ) and is then distilled.

L. A. COLES.

**Distillation products from vegetable substances.**  $\text{C}_2\text{H}_2$  from  $\text{CH}_4$ .—See II.

#### IV.—DYESTUFFS.

**Precipitation of black sulphur-dye waste.** W. RUDOLFS and W. H. BAUMGARTNER (Ind. Eng. Chem., 1931, 23, 906–908).—For the clarification of spent liquors from Sulphur Black dyebaths before discharge into the sewers,  $\text{FeCl}_3$  or, better,  $\text{FeCl}_3$  followed by sufficient  $\text{HCl}$  to produce a final  $p_{\text{H}}$  of about 6.6–7.7, preferably at raised temp., appears to cause rapid and complete clarification and sedimentation without evolution of  $\text{H}_2\text{S}$  or precipitation of free S. Addition of  $\text{FeSO}_4$  and  $\text{H}_2\text{SO}_4$  is likewise effective, and activated sludge is of limited application.

H. A. PIGGOTT.

**Azo dyes derived from homophthalimide.** A. MEYER and R. VITTENET (Compt. rend., 1931, 193, 344–346).—Homophthalimide is coupled with diazotised sulphanilic, *m*-xylyldinesulphonic,  $\beta$ -naphthylamine-6 : 8-disulphonic, naphthionic, and 1-amino- $\beta$ -naphthol-4-sulphonic acids yielding the corresponding azo dyes (*Na* salts). Substantive dyes (shades on cotton given) are prepared by coupling tetrazotised benzidine, tolidine, and dimethoxybenzidine first with 1 mol. of an amino-naphthol or a naphtholsulphonic acid in acid or alkaline media, and then with 1 mol. of homophthalimide in alkali.

H. BURTON.

**anti-Diazotates.**—See III. Sewage and industrial wastes.—See XXIII.

#### PATENTS.

**Manufacture of a stable reduction compound of a vat dye.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 350,862, 9.10.30. Addn. to B.P. 334,878 and 334,919; B., 1930, 1144; 1931, 14).—The process of the prior patents is applied to 5 : 5'-dichloro-4 : 4' : 7 : 7'-tetramethylthioindigo.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 350,575, 15.3.30).—2 : 4-Dichloro-6-phenyl-1 : 3 : 5-triazine is condensed with 2 mols. of an aminoanthraquinone, e.g., 1-aminoanthraquinone (yellow), 1-amino-5-benzamidoanthraquinone (golden-orange), 1-amino-4-benzamidoanthraquinone (red), or 1-amino-4-methoxyanthraquinone (orange).

C. HOLLINS.

**Manufacture of vat dyes from dibenzanthrone and isodibenzanthrone.** S. PERCIVAL. From AZIENDE CHIMICHE NAZIONALI ASSOCIATE (A.C.N.A.), and BELLINO & COLLI (B.P. 350,457, 10.12.29. Cf. B.P. 341,229; B., 1931, 386).—Dibenzanthrone or isodibenzanthrone in  $\text{PhNO}_2$ ,  $\text{C}_6\text{H}_5\text{Cl}_3$ , xylene, or naphtha is treated, e.g., at  $120^\circ$ , with  $\text{AlCl}_3$  and a chlorinating chloride such as  $\text{FeCl}_3$ ,  $\text{CrCl}_3$ ,  $\text{CuCl}_2$ ,  $\text{AsCl}_3$ ,  $\text{PCl}_5$ ,  $\text{SbCl}_5$ . Reddish-blue vat dyes are produced. C. HOLLINS.

**Dyes and dyeing [with enolic ester salts of indanthrones].** D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 350,499, 9.12.29).—For the development of leuco-indanthrone ester salts on the fibre,  $\text{FeSO}_4$  is used in conjunction with acid  $\text{Fe}_2(\text{SO}_4)_3$ , over-oxidation being thereby avoided. C. HOLLINS.

**Manufacture of vat dyes [of the anthraquinone-acridone series] containing halogen.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 350,501, 13.12.29).—A 1-arylaminoanthraquinone, carrying  $\cdot\text{CHO}$  *ortho* to the  $\text{NH}$ , is treated with halogen or halogenating agents. 1-*o*-Aldehydoanilino-, 1-anilino-2-aldehydo-, 1-(2':4':5'-trichloroanilino)-2-aldehydo-, 1:5-di-(*o*-aldehydoanilino)-, and 6(7)-chloro-1-anilino-2-aldehydo-anthraquinone give red to violet vat dyes when treated with  $\text{Cl}_2$  (or  $\text{SO}_2\text{Cl}_2$ ) or  $\text{Br}_2$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  or  $\text{PhNO}_2$ . C. HOLLINS.

**Production of azo dyes [for cellulose esters or ethers].** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 350,577, 9.1.30. Cf. B.P. 346,751; B., 1931, 716).—1-Phenyl-3-methyl-5-pyrazolone is coupled with a diazotised arylamine, containing already or subsequently a  $p\text{-NH}_2$  or  $p\text{-OH}$  group, to give dyes especially suitable for discharge effects with  $\text{SnCl}_2$ . Examples are:  $p$ -aminophenol (golden-yellow),  $p$ -phenylenediamine (orange),  $p$ -aminodimethylaniline (red),  $p$ -aminoacetanilide (golden-orange), 4-dimethylamino-*m*-anisidine (red). C. HOLLINS.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Rapid determination of the moisture content of textile fibres [silk].** E. EINECKE (Z. anal. Chem., 1931, 85, 45—50).—The increase in length of the silk on drying is proportional to the moisture content for the same silk, but varies for different silks. 25 parallel strands of silk are passed through an electrically heated horizontal tube and fixed at one end to a stationary clamp, the other end being passed over a small pulley attached to a lever and tied to a 10-g. wt. The extension of the silk threads moves the lever over a scale on which the extension is magnified in the ratio 60:1. The drying tube is heated to  $135^\circ$  over a period of 5 min., and from the observed extension the  $\text{H}_2\text{O}$  content is obtained by reference to a graph previously constructed from observations made on the same silk with different moisture contents. A. R. POWELL.

**Viscosity of cellulose esters. III. Viscosity of cellulose nitrate solutions in mixed solvents.** K. ATSUKI and M. ISHIWARA (J. Cellulose Inst., Tokyo, 1931, 7, 191—195; cf. B., 1931, 479).—Mathematical expressions are deduced to show the effect of concentration of the cellulose nitrate on the relations between the viscosity of the solution and the composition of the mixed

solvent ( $\text{COMe}_2\text{-H}_2\text{O}$ ). Results of viscosity measurements on 2% solutions of a nitrocellulose containing 12.18% N in solvents of various compositions of  $\text{COMe}_2$  and  $\text{H}_2\text{O}$  are in good agreement with the values calc. from these equations. B. P. RIDGE.

**Beating of wood pulp in laboratory and mill. I. Determination of beaten strength. II. Experimental.** H. A. HARRISON (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 11, 272—303, 303—338).—I. An attempt has been made to continue the work of the Pulp Evaluation Committee of the P.M.A., and a tentative method for measuring the beaten strength of wood pulps is described. Representative samples of pulp are taken under the same conditions as samples for moisture determination. In the case of air-dry pulps, at least four lots of pulp, each exactly equivalent to 24 g. of bone-dry pulp, are soaked for 4 hr. Moist pulps are not soaked. Each batch is made up to 2 litres at  $15\text{--}20^\circ$ , disintegrated by the standard method, conc. to 800 c.c. (3% consistency), and beaten in a Lampen mill (10-kg. ball) for 15, 30, 60, and 120 min. (sulphite pulps) or 30, 60, 120, and 240 min. (kraft pulps), the mill being run at 300 r.p.m. in each case. Each sample of beaten pulp is cleared in the standard disintegrator for 7500 revs. after dilution to 2 litres followed by dilution to 10 litres. 500 c.c. of diluted stock (1.2 g. of bone-dry fibre) are used to make each test sheet, employing the standard procedure for the British standard sheet machine, 6 sheets being made from each sample of beaten stock. Sheets conditioned at 65% R.H. are tested for burst, tensile, and tearing strengths, basis wt., and apparent sp. gr. Burst, tensile, and tear ratios are calc. from the corresponding strength factors obtained by the standard method, having in each case bone-dry wt. in lb. D.C. as denominator. The strength factor for the pulp at each stage of beating is obtained from the formula:  $S.F. = 2B.F. + B.L./100 + T.F./5$ , where  $B.F.$ ,  $B.L.$ , and  $T.F.$  are burst factor, breaking length, and tear factor, respectively. For the comparison of pulps for a particular purpose, the strength factor at the stage of beating corresponding to  $40^\circ$  Schopper-Riegler has been arbitrarily chosen. Comparison with mill practice may be facilitated by calculating strength factors for mill-made papers in a similar way. Details of experiments made in the investigation of the factors involved are given.

II. The use of the Lampen mill in conjunction with the British standard sheet machine in general paper-making investigations is discussed. The effect of cooking, ageing, and heating on the behaviour of pulps on beating has been investigated. Overcooked kraft pulps reach max. strength on beating more rapidly, but final strength is not so high as that of properly cooked pulps. Storage of pulp under normal conditions is found to result in lowering the strength. Heating kraft pulp at  $100^\circ$  for 20 hr. followed by soaking for 4 hr. and beating for 30 min. causes reduction of burst and tensile strengths, and increase in tear. The development of strength during beating is illustrated by examples and photomicrographs. T. T. PORTS.

**Wood pulp testing for fine paper mills.** S. R. H. EDGE (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 11, 227—237).—12.5 g. of pulp are soaked in 2 litres of  $\text{H}_2\text{O}$ ,

disintegrated, conc. to 500 c.c., and beaten in a Lampen mill for 13,500 revs. Sheets are made in the British standard sheet machine. A "general strength factor" (G.S.F.) is calc. from the burst, tear, fold, and break (tensile) of the test sheets, the several strengths being reduced in the ratios 25/100 for burst, 25/250 for tear, 25/30 for break, and 25/3000 for fold. The results are added together to obtain the G.S.F. A similar factor is calc. for the initial (unbeaten) strength. The ratios are so chosen that, for the majority of pulps available, the max. obtained G.S.F. is 100. Numerical examples are given, particularly for bleached sulphite pulps. Determinations of colour, fastness to light, and cleanliness are made on sheets prepared from disintegrated but unbeaten pulp.

T. T. POTTS.

**Bursting tester standardisation [for paper].** G. F. UNDERHAY (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 11, 247—264).—The Mullen and Schopper bursting strength testers have been critically examined. The Schopper instrument is preferred, being the more capable of standardisation. The mechanism for controlling the rate of flow of air, and, hence, the rate of application of pressure to the test piece, is found to be considerably improved by the use of Dewrance valves in conjunction with accurate pressure gauges on the compressed air reservoir. The effect of diaphragm bulge has been investigated, thin red rubber diaphragms being found to give the best results.

T. T. POTTS.

**Ageing of materials.**—See I. Effect of light on nitrocellulose films.—See XIII. Furfuraldehyde.—See XV. Paper-mill waste as spray material.—See XVI.

## PATENTS.

**Treatment of hard vegetable fibres [horsehair substitute].** E. ELÖN (B.P. 354,347, 30.4.30. Ger., 1.5.29. Addn. to B.P. 308,720; B., 1930, 813).—Fibres other than those of coconut, *e.g.*, agave fibre, after removal of embedding substances, are treated in a closed vessel with dil. alkali at 120—150°.

F. R. ENNOS.

**Degumming of textile plants.** U. J. L. THUVAU (B.P. 354,897, 31.5.30. Fr., 15.6.29).—The fibres are treated in the hot or cold with a solution of simple non-substituted naphthalenesulphonic acids of appropriate concentration, washed with H<sub>2</sub>O, and neutralised with a solution of soap and/or alkali.

F. R. ENNOS.

**Treating [deodorising, sterilising, and bleaching], fibrous materials.** F. W. BRODERICK (U.S.P. 1,792,805, 17.2.31. Appl., 22.8.27).—The material, *e.g.*, sawdust, is moistened with 0.5—1% aq. Na<sub>2</sub>CO<sub>3</sub>, heated by steam to 38°, and treated with sufficient Cl<sub>2</sub> to form NaOCl *in situ*. The material is finally washed and dried.

D. J. NORMAN.

**Manufacture of solutions of silk fibroin.** I. G. FARBERIND. A.-G. (B.P. 355,161, 23.12.30. Ger., 23.12.29).—A solution obtained by dissolving fibroin in anhyd. liquid NH<sub>3</sub> at -77° is mixed with H<sub>2</sub>O, after which the NH<sub>3</sub> is expelled.

F. R. ENNOS.

**Treatment [saccharification] of carbohydrates.** H. DREYFUS (B.P. 354,820, 15.5.30).—Cellulosic materials are impregnated with a non-volatile acid (H<sub>2</sub>SO<sub>4</sub>) sufficiently dil. to prevent hydrolysis, and are then sub-

jected to a vac. or gas blast (*e.g.*, of N<sub>2</sub> or CO<sub>2</sub>) to concentrate the residual acid with formation of dextrins; further hydrolysis to sugars is effected by dilution with H<sub>2</sub>O and heating to 100°.

F. R. ENNOS.

**Production of paper stock.** H. P. BASSETT (U.S.P. 1,792,351, 10.2.31. Appl., 3.6.25).—Hydrated stock is obtained from, *e.g.*, cotton linters without prolonged or severe beating by conducting the beating operation in a weakly acid medium, *e.g.*, 0.05—1% H<sub>2</sub>SO<sub>4</sub>, at 20—100°, *e.g.*, 80°, for 0.5—4 hr. The material is then washed, treated with dil. alkali to remove sol. org. salts, and, if desired, again treated with 0.05—1% H<sub>2</sub>SO<sub>4</sub> at 20—100°. Treatment with dil. aq. NaOH may in certain circumstances precede the acid beating treatment. The degree of hydration increases the more conc. is the acid, the higher the temp., and the longer the duration of treatment.

D. J. NORMAN.

**Forming feltable bituminous pulps.** C. L. KELLER, ASSR. to RICHARDSON CO. (U.S.P. 1,792,098, 10.2.31. Appl., 20.5.29).—The pulp is first converted into half-stuff and is then thickened and mixed with thermoplastic materials, preferably in a mixer of the Werner-Pfleiderer or Banbury pressure type. The stock is then broken down with H<sub>2</sub>O and beaten to the required degree in a hollander.

D. J. NORMAN.

**Method of wood disintegration.** W. O. MITSCHERLING, ASSR. to A. M. JOHNSON (U.S.P. 1,793,711, 24.2.31. Appl., 27.12.28).—Wood logs are subjected to a vac. first at room temp. for about 2 hr. to remove air and easily volatile constituents and then at 80—90° for about 1 hr. to remove heavy resin oils etc. The vac. is then reduced gradually and the pressure raised to and held at 100—150 lb./sq. in. for about 0.5 hr., after which it is gradually raised to 600 lb./sq. in. After 1 hr. the pressure is suddenly released so that the wood becomes disrupted into fibres by the air in the capillaries. The resulting fibres are in an unbroken condition, are easily penetrated by cooking liquors, and require less chemical for digestion.

D. J. NORMAN.

**Preparation of ground wood pulp.** R. S. HATCH, R. B. WOLF, and R. P. HILL (U.S.P. 1,794,174, 24.2.31. Appl., 31.1.29).—Wood is ground on a relatively coarse stone to give 10—40% or more of slivers. These are separated by screening and then treated in an apparatus in which circulation can be maintained and at a pulp concentration of about 10% with, *e.g.*, 4.5% of Na<sub>2</sub>SO<sub>3</sub> (on the wt. of slivers) at 82° until separation of the fibres is substantially complete (2 hr. or more). After further screening to remove insufficiently softened slivers a long-fibred pulp is obtained which, after admixture with the short-fibred pulp already obtained, can be used for the manufacture of paper without addition of chemical pulp. A saving in steam and power is thereby effected.

D. J. NORMAN.

**Specific digestion procedure and apparatus for use in the production of chemical wood pulp.** C. E. PETERSON, ASSR. to CHAMPION FIBRE CO. (U.S.P. 1,792,703, 17.2.31. Appl., 12.8.29).—In the process for producing easy-bleaching sulphate pulp by digesting coniferous wood with sufficient of a Na<sub>2</sub>S—NaOH liquor containing 70—80 g. of total active chemical per litre

to give 28–36% of active chemical (calc. as  $\text{Na}_2\text{CO}_3$  in each case) on the wt. of wood, the time required to heat the liquor to the cooking temp. by ordinary, external, indirect heating means is considerably increased owing to the relatively large quantity of liquor required. This is obviated by introducing direct steam into the bottom of the digester while maintaining external circulation of the liquor from the bottom of the digester to the top, until the required cooking temp. is reached, after which the external indirect heater is employed. The dilution caused by injecting steam must be allowed for in preparing the cooking liquor, so that the final cooking conditions are as above.

D. J. NORMAN.

**Treatment [beating] of paper pulp.** W. V. KNOLL, Assr. to MID-WEST MACHINE Co. (U.S.P. 1,793,095, 17.2.31. Appl., 4.10.26).—The stock is beaten in a small, totally enclosed beater comprising a fast-running roll, e.g., 2 ft. in diam. and 3 ft. long, revolving at more than 300 r.p.m. No back-fall is provided, the stuff being fed in by gravity and returned to the gravity tank by a centrifugal pump, which is so positioned as to exert a small suction effect on the output side of the roll. The bearings for the roll are fixed, the beating pressure being controlled by adjusting a movable bed plate by means of a wedge-type mechanism.

D. J. NORMAN.

**Cooking of sulphite cellulose.** S. I. TSCHUVIKOVSKY and G. I. LIACHOVETZKY (U.S.P. 1,793,264, 17.2.31. Appl., 8.11.29. U.S.S.R., 7.8.28).—The hot spent liquor is run direct from the digesters to an evaporating plant where it is conc. at a high temp. by fresh steam. The secondary steam and gases resulting from this operation are fed back into the digesters to assist in the cooking of a fresh charge.

D. J. NORMAN.

**Production of moulded cellulose composition.** F. H. SMYSER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,792,254, 10.2.31. Appl., 22.10.29).—After digestion of cellulosic material (sawdust or bagasse) with alkali, the mass is acidified to ppt. resinous substances on and around the fibres. The filtered material is then washed until neutral, mixed with 5–40 wt.-% of  $\text{Ca}(\text{OH})_2$ , powdered, and moulded under heat and pressure.

F. R. ENNOS.

**Apparatus for producing cellulose acetate.** J. ALTWEGG, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,792,059, 10.2.31. Appl., 10.10.29).—A vertical cylinder with an inlet at the top and an outlet at the bottom has a central shaft to which are attached a number of rotatable stirring elements, which vary in form and increase in size from inlet to outlet according to the texture of the material to be treated thereby.

F. R. ENNOS.

**Manufacture of esters of derivatives of cellulose or other carbohydrates.** I. G. FARBENIND, A.-G. (B.P. 355,144, 26.11.30. Ger., 26.11.29. Addn. to B.P. 301,036; B., 1930, 504).—Esterification is conducted in liquid  $\text{SO}_2$  by means of a free carboxylic acid or mixture of acids, under pressure higher than that due to the  $\text{SO}_2$  and/or in presence of a catalyst or oxidising agent.

F. R. ENNOS.

**Manufacture of cellulose derivatives esterified by residues of partly esterified polybasic acids.**

DEUTS. CELLULOID-FABR. (B.P. 355,172, 17.2.31. Ger., 24.2.30).—Cellulose or a derivative thereof containing one or more OH groups is treated with a chloride of an acid ester of a polybasic acid, which may be added as such or formed in the reaction mixture from the acid ester, e.g., Bu H phthalate, and a catalyst ( $\text{SOCl}_2$ ,  $\text{SO}_2\text{Cl}_2$ ,  $\text{PCl}_3$ , or  $\text{POCl}_3$ ).

F. R. ENNOS.

**Manufacture of mixed esters of cellulose.** DEUTS. CELLULOID-FABR. (B.P. 354,221, 25.3.30. Ger., 25.3.29).—A cellulose fatty acid ester containing free OH groups is dissolved in diacetyl-*o*-nitric acid, either previously made or in a nascent state, and treated with a dehydrating agent ( $\text{Ac}_2\text{O}$ ).

F. R. ENNOS.

**Spinning of artificial silk threads.** COMPTOIR DES TEXTILES ARTIFICIELS, SOC. ANON. (B.P. 355,155, 4.12.30. Fr., 12.12.29).—A method of winding the threads into a spinning pot is described.

F. R. ENNOS.

**Manufacture of artificial silk [of reduced lustre].** KIRKLEES, LTD., A. BENNETT, and W. TONG (B.P. 355,015, 11.8.30).—A completely dissolved viscose solution is intimately mixed with a paraffin-protein emulsion. An emulsion of liquid paraffin with casein or blood-albumin is suitable.

F. R. ENNOS.

**Maintaining the composition of viscose precipitating baths containing magnesium sulphate.** J. J. STÖCKLY and R. BARTUNEK, Assrs. to VEREIN. GLANZSTOFF-FABR. A.-G. (U.S.P. 1,793,649, 24.2.31. Appl., 27.11.28. Ger., 15.12.27).—Liquors containing  $>220$  g. of  $\text{MgSO}_4$  per litre are cooled to 8–10°, at which temp. part of the  $\text{MgSO}_4$  crystallises out. The mother-liquor from this is diluted by about 6–10% and cooled to –5° to –10°, whereby part of the  $\text{Na}_2\text{SO}_4$  crystallises out. The remaining liquor is conc. and used in conjunction with the requisite quantity of recovered  $\text{MgSO}_4$  for concentrating the precipitating baths. Liquors containing  $<170$  g. of  $\text{MgSO}_4$  per litre are cooled direct to –5° to remove part of the  $\text{Na}_2\text{SO}_4$  and are then conc. and used as above. Liquors containing 170–220 g. of  $\text{MgSO}_4$  per litre are worked up either after addition of sufficient  $\text{MgSO}_4$  to give a total  $\text{MgSO}_4$  content  $>220$  g. per litre, or after dilution with  $\text{H}_2\text{O}$  to a  $\text{MgSO}_4$  content  $<170$  g. per litre.

D. J. NORMAN.

**Manufacture of pyroxylin or other cellulose ester sheet material.** FIBERLOID CORP., Assees. of G. J. ESSELEN, JUN. (B.P. 354,339, 4.4.30. U.S., 1.11.29).—A pyroxylin mix of stiff, dough-like consistency is extruded by high pressure through an elongated orifice as a continuous, self-sustaining sheet, from which the solvent is removed by means of hot air or a suitable liquid.

F. R. ENNOS.

**Manufacture of cardboard.** P. B. BURCHARDT (B.P. 354,829, 16.5.30. Swed., 16.11.29).—A number of layers of cellulose wood pulp in sheet form and paper, with outermost layers of the latter, are compounded by pressure after application of asphalt, tar, or similar moisture-proof material.

F. R. ENNOS.

**Stencil sheet.** S. HORII (U.S.P. 1,792,095, 10.2.31. Appl., 26.7.28).—Yoshino paper is coated with a solution containing esters of polysaccharides in association with naphthenic glycerides and a heavy hydrocarbon oil free



from solid paraffin, *e.g.*, liquid paraffin of *d* 0.903—0.875. D. J. NORMAN.

[Sized] paper product. W. C. LODGE, Assr. to OSWEGO FALLS CORP. (U.S.P. 1,792,382, 10.2.31. Appl., 28.1.27).—Paper with max. H<sub>2</sub>O-resistance compatible with good strength is obtained by sizing with 2—3% of resin and 1% of paraffin wax. The wax emulsion is added after the resin size and the whole precipitated with alum. The wax increases the degree of retention of the resin, apart from its waterproofing action.

D. J. NORMAN.

Treating [cross-wound] cheeses [of artificial silk] with liquids. ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 355,645, 18.2.31. Holl., 18.2.30).

Non-smooth films for use as sound or talking films. A. A. P. RICHARD (B.P. 355,259, 16.5.30. Fr., 22.2.30).

Filter [for "white water"].—See I. Dissolving xanthates. Alkalising waste pulp-liquors.—See VII. Laminated glass.—See VIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Application of a photo-element to the determination of the degree of bleaching of fabrics. G. E. MUCHIN, VOJCIECHOVSKI, and L. J. KURTZ (Ukrain. Chem. J., 1931, 6, [Tech.], 17—21).—A photo-element which is more convenient and gives more exact results than the step-photometer is described.

R. TRUSZKOWSKI.

Dyeing of cellulose. I. Dyeing with basic dyes. Facts consistent with the electrical adsorption theory of dyeing. K. KANAMARU (J. Cellulose Inst., Tokyo, 1931, 7, Abs. p. 42).—A theoretical explanation is given in this preliminary paper for some observed facts relating to the dyeing properties of cellulosic substances (*e.g.*, normal cellulose, hydro- and oxycelluloses, cellulose hydrate, regenerated cellulose, cellulose esters, etc.) for basic dyes, based on data obtained with respect to the  $\zeta$ -potentials on such substances. The effects of electrolytes in the dye bath and especially the  $p_H$  of the latter are also considered from the viewpoint of the electrical adsorption theory of dyeing.

A. J. HALL.

Weighting of knitted woollen goods. S. R. TROTMAN and W. SAVORY (J.S.C.I., 1931, 50, 299—303 T).—Dyed woollen hose and half-hose lose wt. in dyeing, owing to protein hydrolysis and diminution of "regain." The authors describe experiments made with the object of making good this loss. Hygroscopic agents are used sometimes, and of these diethylene glycol is the best. Double-bath processes are inconvenient and expensive, but a moderate amount of weighting may be obtained by means of gelatin tannate, metallic salts of algin, wool protein, and metallic oxides. Wool has a natural affinity for sulphonic acids of C<sub>10</sub>H<sub>8</sub>. This affinity is increased by the presence of OH groups and diminished by NH<sub>2</sub> groups. These acids may be added to the ordinary acid dyebath after most of the dyestuff has been taken up without deleterious effect on the colour, tensile strength, or handle of the goods. Synthetic tans act in a similar manner. An increase in wt. of from 2 to 3 oz. per dozen pairs of hose may be

obtained by adding 10% of Diaclar S (on the wt. of the goods) to the nearly exhausted dyebath and completing dyeing in the usual manner.

*anti-Diazotates*.—See III. *Detergents*.—See VII. *Rubber and textiles etc.*—See XIV. *Sewage and industrial wastes*.—See XXIII.

## PATENTS.

Printing on cotton by means of vat dye preparations. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,955, 30.11.29).—Hydroxyanthraquinones or their leuco-compounds are added to vat dye printing pastes, with or without a hydrotropic agent. Examples are: vat dye, glycerin, 2-hydroxy- or 1-chloro-2-hydroxy anthraquinone, thickening, KOH, and formaldehyde-sulphoxylate; vat dye, anthraquinone, 2-hydroxyanthraquinone, glycerin, aq. NH<sub>3</sub>, hyposulphite, and, after reduction, PhSO<sub>3</sub>Na; vat dye, anthraflavin, glycerin, urea, conc. to 10.85% dye. Stronger dyeings are obtained.

C. HOLLINS.

Production of plaited fabric. C. DREYFUS (U.S.P. 1,793,915, 24.2.31. Appl., 15.7.27).—Permanent plaited effects are obtained on fabrics consisting of thermoplastic derivatives of cellulose (*e.g.*, cellulose acetate, methyl cellulose) by passing them through a Chandler machine after being softened by impregnation or spraying with a suitable agent such as a 25% solution of COMe<sub>2</sub> or other suitable solvent.

A. J. HALL.

Composition of matter. [Fireproof binding for fibrous materials.] C. H. BROWN (U.S.P. 1,793,357, 17.2.31. Appl., 19.4.28).—Such a composition, especially suitable for use with redwood bark, consists of an NH<sub>4</sub> salt, such as (NH<sub>4</sub>)<sub>3</sub>PO<sub>4</sub>, H<sub>3</sub>BO<sub>3</sub>, and an amorphous constituent such as dextrin.

A. J. HALL.

Protective treatment [fireproofing] of materials. A. ARENT, Assr. to A. ARENT LABORATORIES, INC. (U.S.P. 1,792,457, 10.2.31. Appl., 8.7.26).—For fireproofing cellulose ester materials (*e.g.*, kinema film, artificial silk) a solution of SbCl<sub>3</sub> in EtOAc, containing urea, is employed.

J. LEWKOWITSCH.

Dry-cleaning plant [with solvent recovery]. BRIT.-AMERICAN LAUNDRY MACHINERY CO., LTD. From AMERICAN LAUNDRY MACHINERY CO. (B.P. 355,809, 29.5.30).

Dyeing with indanthrone derivatives.—See IV. Bleaching fibrous materials.—See V.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Absorption of oxides of nitrogen by sulphuric acid (formation of nitrosyl compounds), and certain factors in denitration. E. V. GERBURD-GEIBOVITSCH, E. S. KABANOV, and S. A. KRIVOSCHEEV (Ukrain. Chem. J., 1931, 6, [Sci.], 121—134).—The absorption of NO is in practice so small as to be negligible. Better absorption is obtained by oxidising NO to NO<sub>2</sub> than to N<sub>2</sub>O<sub>3</sub>, as both NO·SO<sub>2</sub>·OH and HNO<sub>3</sub> are formed; for this purpose air is introduced into the chambers. The optimal concentration of H<sub>2</sub>SO<sub>4</sub> is *d* 1.77 at 40°. The velocity of formation of NO compounds varies inversely with the temp. and directly with the concentration of

SO<sub>2</sub>. Pure NO·SO<sub>2</sub>·OH is not decomposed at its b.p., but in the presence of HNO<sub>3</sub> it begins to decompose at 40°, below which temp. loss of N owing to this reaction is in factory practice negligible. The NO compound obtained contains exclusively N<sup>III</sup>. R. TRUSZKOWSKI.

**Determination of phosphoric acid.** A. F. DA SILVA (Rev. Chim. Pura Appl., 1930, [iii], 5, 69—94).—A critical review of available methods, particularly as regards their applicability for soil and fertiliser analysis. Results for various soils are discussed in relation to the assimilation of P by plants. H. F. GILLBE.

**Manufacture of synthetic ammonia.** G. FAUSER (Giorn. Chim. Ind. Appl., 1931, 13, 361—367).—The most economical pressure is about 200 atm. The reacting gases are introduced in the catalysing chamber at 600° and then the temp. is gradually lowered to 450° as the % NH<sub>3</sub> formed increases. The velocity of reaction is thus accelerated by means of a temp. exchanger which consists of a set of tubes immersed in the catalysing material. The fresh gases are circulated and gradually heated in these tubes before coming in contact with the catalyst; the temp. reaches 600° at this point, and then the outgoing gases undergo a gradual cooling by the countercurrent action of the gases travelling in the tubes. O. F. LUBATTI.

**Chemical control of manufacture of crystalline sodium carbonate.** J. POSPIŠIL (Chem. Listy, 1931, 25, 298—302).—A description of the operations involved. R. TRUSZKOWSKI.

**Preparation of colourless crystalline sodium sulphide.** V. LENCHOLD and S. SOLOVIEV (J. Chem. Ind. Russ., 1931, 8, 738—739).—The yellow coloration of technical Na<sub>2</sub>S is due to colloidal FeS and to Na<sub>2</sub>S<sub>2</sub>. NaCN (0.8%) decolorises the product by forming Na<sub>4</sub>Fe(CN)<sub>6</sub> and NaCNS. R. TRUSZKOWSKI.

**Detergent value of sodium metasilicate.** C. L. BAKER (Ind. Eng. Chem., 1931, 23, 1025—1032).—The detergent properties of Na<sub>2</sub>SiO<sub>3</sub> have been compared with those of NaOH, Na<sub>2</sub>CO<sub>3</sub>, and Na<sub>3</sub>PO<sub>4</sub> in respect of wetting, emulsification, deflocculation, and dissolving power. Na<sub>2</sub>SiO<sub>3</sub> is more effective in wetting glass or in displacing petroleum from a glass surface than are the other reagents examined. Solutions of Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O and Na<sub>3</sub>PO<sub>4</sub>·12H<sub>2</sub>O emulsify a light motor oil better than do those of Na<sub>2</sub>CO<sub>3</sub> and NaOH, and the deflocculating power of Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O for bone black is equal to that of the other reagents. Na<sub>2</sub>SiO<sub>3</sub> yields solutions of higher *p*<sub>H</sub> than do the other alkalis, with the exception of NaOH, and a larger proportion of the Na<sub>2</sub>O is available at a *p*<sub>H</sub> effective in dissolving fats than in any other alkali salt considered. Solutions of Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O can be used for washing tin-plated articles which would be attacked by the other reagents. The foaming power of a soap solution is related to its power to suspend solid dirt. Solutions of Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O give a larger vol. of foam with Na stearate at 60° than the other alkalis at all concentrations, excepting a very small concentration range where NaOH is superior. A smaller amount of soap is required to wash clothes when the *p*<sub>H</sub> is maintained above 10.5. E. S. HEDGES.

**[Manufacture of] sodium ferrocyanide.** V. N. KURTZKOV (J. Chem. Ind. Russ., 1931, 8, 835—837).—

Leather waste, horn, etc. and scrap Fe are added to a fused mixture of 2 pts. of calcined soda to 1 pt. of charcoal, and the product is extracted with H<sub>2</sub>O at 80—100° during 16—20 hr.; Ca(OH)<sub>2</sub> is then added and the mixture filtered. The filtrate, containing NaOH, Na<sub>4</sub>Fe(CN)<sub>6</sub>, NaCNO, NaCNS, and Na<sub>2</sub>S, is evaporated to *d* 1.24 at 95° and allowed to crystallise. The mother-liquor is evaporated to dryness, leaving a residue of "black soda." On repetition of the process the original soda-charcoal mixture is replaced by one containing 5 kg. of calcined soda, 16 kg. of "black soda," and 5 kg. of charcoal, when slightly higher yields of Na<sub>4</sub>Fe(CN)<sub>6</sub> are obtained at a lower cost.

R. TRUSZKOWSKI.

**"Electro-nitrogen" process [for the production of ammonium sulphate].** B. WAESER (Brennstoff-Chem., 1931, 12, 293—295. Cf. Tern, B.P. 338,869; B., 1931, 331).—A mixture of air and SO<sub>2</sub> obtained by roasting spent gas-purification material is subjected to a high-tension electric flame, under the influence of which the SO<sub>2</sub> is oxidised to SO<sub>3</sub>, and the latter is then mixed with NH<sub>3</sub>, obtained from gas liquor, and the requisite quantity of H<sub>2</sub>O vapour, in an electrostatic precipitation apparatus, wherein (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> is separated in a dry form. Tern has assumed that the oxidation of the SO<sub>2</sub> is facilitated catalytically by the N oxides formed in the electric flame. The process works best with gases of low concentration, e.g., 0.6—1.5 vol.-% of SO<sub>2</sub>. If the product is to be used as a fertiliser the removal of CO<sub>2</sub> from the NH<sub>3</sub> is unnecessary. A large plant has been constructed by the Thuringian gasworks and is in operation near Leipzig. The process appears to be suitable for works already provided with oxide purifiers and the necessary apparatus for treating gas liquor; for new plants, and for utilising the S in coke-oven gases for binding synthetic NH<sub>3</sub> as sulphate, the washing methods are to be preferred.

A. B. MANNING.

**Extraction of potash from polyhalite. II. Production of syngenite and by-product magnesite.** H. H. STORCH and N. FRAGEN (Ind. Eng. Chem., 1931, 23, 991—995; cf. B., 1930, 1026).—When calcined polyhalite is leached with H<sub>2</sub>O at 25° the solid phase consists of syngenite and CaSO<sub>4</sub>. The CaSO<sub>4</sub> may be kept in solution by adding K<sub>2</sub>SO<sub>4</sub> and thus practically pure syngenite may be prepared. The residual liquor contains MgSO<sub>4</sub>, which may be reclaimed as MgO by treatment with CO<sub>2</sub> and NH<sub>3</sub> and subsequent thermal decomp. of the precipitated MgCO<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>·4H<sub>2</sub>O. E. S. HEDGES.

**[Alleged fire risks in handling] commercial calcium nitrate.** C. MATIGNON (Compt. rend., 1931, 193, 329—330; cf. B., 1931, 675).—In a recent fire in Oslo, burning beams etc. fell on several tons of commercial Ca(NO<sub>3</sub>)<sub>2</sub> without causing any specially active combustion or hindrance in the extinction of the fire.

C. A. SILBERRAD.

**Storage of calcium cyanamide.** J. Z. ZALESKI (Przemysl Chem., 1931, 15, 271—274).—CaCN<sub>2</sub> decomposes when kept in moist air, the dicyanodiamide content increasing in saturated air from 0.1% of total N to 1.3% after 10 days, 4% after 20 days, and 61.4% after 80 days. The NH<sub>3</sub> content increases to 2% after

20 days, whilst after 30 days loss of N amounts to 7%. Under ordinary hygroscopic conditions, the N content falls by about 14% during a year, and the dicyanodiamide content rises to about 21% total N. The presence of  $\text{CO}_2$  in the air does not affect  $\text{CaCN}_2$ . The presence of  $\text{CaCl}_2$  accelerates decomp.,  $\text{MnO}_2$  without action, whilst mineral oil retards decomp.  $\text{CaCN}_2$  is best stored in stacks, covered with a layer of sand, and kept in dry magazines. R. TRUSZKOWSKI.

**Present status and uses of cyanamide-process cyanide.** G. H. BUCHANAN (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 235—254).—Aero Brand cyanide is made by fusing  $\text{CaCN}_2$  with C and NaCl in an electric furnace and cooling rapidly; it contains 25.4% CN', 19.8% Cl, 32.1% Ca, 12.1% Na, 0.9%  $\text{CN}_2$ ", and 1.9%  $\text{CaC}_2$ . The "Aerocase" method of case-hardening comprises immersing the Fe or steel in a bath of molten neutral salts to which 0.5—1%  $\text{Ca(CN)}_2$  is added every hr. The depth and rate of penetration of C into the Fe are greater and more uniform in this bath than in one containing 30—90% NaCN; this difference is attributed to the strong tendency for  $\text{Ca(CN)}_2$  to revert to  $\text{CaCN}_2$  with the liberation of C in an active form. Finely-powdered  $\text{Ca(CN)}_2$  is rapidly decomposed by moisture, yielding HCN gas, and is therefore useful as a fumigator and insecticide. The uses of HCN in org. synthesis and in the prep. of ferrocyanides is indicated. A. R. POWELL.

**Cyanides in metallurgy.** M. R. THOMPSON (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 263—273).—The manufacture of alkali cyanides and their uses in the treatment of Au and Ag ores, in electroplating, and in case-hardening are briefly discussed. A. R. POWELL.

**Determination of traces of chloride in bromides.** I. E. ORLOV (J. Chem. Ind. Russ., 1931, 8, 630).—The bromide (5 g.) is dissolved in  $\text{H}_2\text{O}$ , 50 c.c. of 20%  $\text{H}_2\text{SO}_4$  and 3 g. of freshly prepared  $\text{MnO}_2$  are added, and the mixture is diluted to 150 c.c. and boiled for 30 min. to expel free Br. The residue is diluted to 200 c.c., filtered, and Cl determined by Volhard's method in 100 c.c. of filtrate. R. TRUSZKOWSKI.

**Grading aggregates.** See I. Catalysts for MeOH. —See III. Ag and Au plating baths.—See X. Lithopone.—See XIII. Testing  $\text{H}_2\text{O}_2$ . Determination of Hg in preps.—See XX.

#### PATENTS.

**Manufacture of nitrogen-oxygen compounds [nitric acid].** GUTEHOFFENUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 354,736, 6.2.30. Ger., 6.2.29).—Moist air is passed through the electric field produced between solid electrodes by a high-tension a.c. (frequency >100,000 periods per sec.) at >50,000 volts. Catalytically active bodies, e.g., Cu, preferably as intermediate electrodes, are placed in the high-frequency field. A. R. POWELL.

**Purification of acid waste [from steel-pickling vats] and recovery of values therefrom.** J. T. TRAVERS, Assr. to OHIO SANITARY ENG. CO. (U.S.P. 1,793,342, 17.2.31. Appl., 17.10.27).—After cooling, the waste is treated with a 4 : 1 mixture of a solid waste product from the alkali plant in the manufacture of

$\text{CaOCl}_2$  and  $\text{Ca(OH)}_2$ . The resultant sludge is adapted for purifying waste liquors containing org. matter.

W. J. WRIGHT.

**Catalytic oxidation of ammonia.** N. CARO and A. R. FRANK (B.P. 355,178, 26.2.31. Ger., 3.3.30).—A mixture of 1 vol. of  $\text{NH}_3$  and 2—3 vols. of  $\text{O}_2$  is passed over Pt or Rh-Pt gauzes cooled by means of metal plates or tubes which themselves are cooled by boiling  $\text{H}_2\text{O}$ , so that the catalyst is maintained at 700—1000°. A 95% conversion into  $\text{N}_2\text{O}_4$  of high concentration is claimed. A. R. POWELL.

**Apparatus for oxidation of nitrites to nitrates.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 355,092, 13.10.30).—The alkaline liquors obtained by the absorption of  $\text{N}_2\text{O}_4$  in  $\text{Ca(OH)}_2$  or NaOH solution in the catalytic oxidation of  $\text{NH}_3$  are mixed with  $\text{HNO}_3$  and the mixture is blown with air (enriched to contain 50—90%  $\text{O}_2$ ) upwards under pressure through a tower packed with a refractory filler or a series of sieves to break up the gas stream. The effluent from the top of the tower contains no nitrous compounds and the waste gases contain no combined N. A. R. POWELL.

**Machine for dissolving xanthates.** R. THURM and E. SCHMIERER, Assrs. to BAKER PERKINS CO., INC. (U.S.P. 1,794,214, 24.2.31. Appl., 20.1.30).—The apparatus comprises an upright cylinder within a bowl. The cylinder is divided into compartments by horizontal grids above which rotate propellers which force the material downwards through the grids; the height of the compartments and the thickness of the slices cut by the propellers diminish downwardly, and the lowest compartment has its cylindrical wall in the form of a grating (forming the outlet to the bowl), the propeller within which is designed to give great eddying effect with but little propulsion. B. M. VENABLES.

**Crystallisation of borax from solution.** R. B. PEET, Assr. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,792,863, 17.2.31. Appl., 16.4.29).—A colloidal emulsion (e.g., laundry soap, oleic acid) is added to the super-saturated solution to prevent aggregation of crystals and, instead, induce crystallisation of single crystals. Cooling may be accelerated by reducing the pressure, and the solution may be agitated during cooling, seed crystals being added, if desired. W. J. WRIGHT.

**Preparation of hydrogen peroxide-phosphate compounds.** G. SCHOENBERG (B.P. 355,016, 11.8.30. Austr., 10.8.29).— $\text{Na}_2\text{HPO}_4$  (1 mol.) or  $\text{Na}_4\text{P}_2\text{O}_7$  (0.5 mol.) is mixed with 30%  $\text{H}_2\text{O}_2$  (1 mol.) and the mixture dried at 30° in vac. The dry product is comminuted and again gently heated in vac. until  $\text{H}_2\text{O}$  ceases to be evolved.  $\text{Na}_2\text{O}_2$  may replace  $\text{H}_2\text{O}_2$ , but a more acid phosphate must be used to combine with the extra Na'. A. R. POWELL.

**Production of triammonium phosphate from potassium phosphates.** I. G. FARBENIND. A.-G. (B.P. 355,111, 27.10.30. Ger., 2.11.29).—A 30% solution of  $\text{K}_2\text{HPO}_4$  is treated with 20 wt.-% of  $\text{NH}_3$  and with sufficient  $\text{CO}_2$  to convert the K into  $\text{K}_2\text{CO}_3$ . On cooling, 98% of the  $\text{P}_2\text{O}_5$  present separates as  $(\text{NH}_4)_3\text{PO}_4 \cdot 3\text{H}_2\text{O}$ . A. R. POWELL.

**Production of ammonium sulphate.** KUNST-DUNGER-PATENT-VERWERTUNGS A.-G. (B.P. 355,098,

17.10.30. Swed., 9.11.29).— $\text{CaSO}_4$  and  $(\text{NH}_4)_2\text{CO}_3$  are fed continuously into a conc. solution of  $(\text{NH}_4)_2\text{SO}_4$  at  $35^\circ$  and the reaction products are continuously removed from the reaction vessel so that the mixture therein remains of practically const. composition as regards  $\text{CaCO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$ . In this way the  $\text{CaCO}_3$  ppt. is obtained in a coarsely cryst. form which filters readily.

A. R. POWELL.

**Alkalisising the waste liquors from the soda- or sulphate-pulp manufacture for the purpose of dry-distilling the same.** E. L. RINMAN (B.P. 354,951, 8.7.30. Swed., 23.5.30. Addn. to B.P. 335,371; B., 1930, 1150).—A mixture of powdered  $\text{BaO}$  or  $\text{SrO}$  with  $\text{CaO}$  is added to the conc. hot waste liquor, whereby a powdery mass is obtained;  $\text{BaO}_2$  is simultaneously or subsequently added to convert S both in the waste liquor and in the added oxides into sulphates.

F. R. ENNOS.

**Treatment of seaweeds.** M. MALLET (B.P. 355,223, 13.5.30. Fr., 15.5.29).—Seaweed, contained in vats with perforated bottoms, is extracted with, e.g.,  $\text{Na}_2\text{CO}_3$  solution which is collected in tanks below each vat, whence it is sprayed into the charge in the previous vat of the series. The vats are moved forward periodically, the first one being removed for recharging and returned to the other end of the series. The Na alginates in the solution are precipitated by the addition of EtOH and removed, and the EtOH and salts are recovered from the residual liquor.

L. A. COLES.

**Production of decolorising material [for gasoline].** W. S. BAYLIS, ASSR. to FILTROL CO. (U.S.P. 1,792,625, 17.2.31. Appl., 22.5.26).—Decolorising clay is dehydrated by heating at  $149^\circ$ , pulverised, and mixed with 4–8% of conc.  $\text{H}_2\text{SO}_4$ .

W. J. WRIGHT.

**Formation of moulded products [from magnesium oxide].** L. T. RICHARDSON and E. M. SCHMIEL, ASSRS. to CUTLER-HAMMER, INC. (U.S.P. 1,793,830, 24.2.31. Appl., 23.5.25).—Granular fused  $\text{MgO}$  is packed into a container capable of withstanding considerable pressure, wetted, and heated in an autoclave. The  $\text{MgO}$  becomes hydrated and expands, filling all voids. The moulded article is then heated to redness to re-form  $\text{MgO}$ .

C. A. KING.

**Production of commercial antimony oxide.** ANTIMON BERG U. HÜTTENWERKE A.-G. (B.P. 355,613, 1.12.30. Ger., 29.11.29).—A mixture of Sb "concentrate" (purified Sb ore) and 20–40% of "fusible oxide" (the residue from a previous oxidation) is oxidised by air or  $\text{O}_2$  in a furnace. The Sb oxide is recovered in the usual way by volatilisation.

H. ROYAL-DAWSON.

**Zeolite-regenerating process.** W. H. GREEN, ASSR. to PERMUTIT CO. (U.S.P. 1,792,220, 10.2.31. Appl., 13.12.26).—The zeolite is passed through a solution of NaCl countercurrent to the flow of regenerating solution so that the conc. NaCl first meets zeolite from which most of the Ca and Mg has been removed by the more dil. NaCl solution.

A. R. POWELL.

**Production of carbon dioxide ice.** G. D. G. CRIBB (B.P. 355,602, 18.11.30. Addn. to B.P. 314,371; B., 1929, 1015).—The  $\text{CO}_2$  ice or snow in the expansion chamber is made to undergo periods of drenching with

liquid  $\text{CO}_2$  which alternate with the periods of downward suction.

H. ROYAL-DAWSON.

**Apparatus for condensing sulphur.** SULPHUR & SMELTING CORP., ASSCES. OF E. W. WESCOTT (B.P. 355,272, 17.2.30. U.S., 5.6.29).—S vapour or a gas mixture containing it is passed through a series of condensers comprising header sections and connecting tubes surrounded by a jacket containing  $\text{H}_2\text{O}$  or steam at a temp., e.g.,  $115$ – $150^\circ$  or  $240$ – $250^\circ$ , such that the S is deposited in a freely fluid form. The tubes contain, e.g., baffles, helical strips, perforated plates, and a mist-collecting chamber may be connected with the last condenser of the series.

L. A. COLES.

**Chemical reactions with arc discharges. Absorbent for refrigerators. Separation of gases.**—See I. Gas and fertilisers. S from gases.—See II. Acid-resisting enamels.—See VIII. Fertilisers.—See XVI.

## VIII.—GLASS; CERAMICS.

**Restoration of solarised ultra-violet-transmitting glasses by heat treatment.** A. Q. TOOL and R. STAIR (Bur. Stand. J. Res., 1931, 7, 357–374).—The ultra-violet transmission of "vita" and "helio" glass before and after artificial solarisation at room temp., and after heat treatment in the temp. range  $200$ – $600^\circ$ , is discussed. The greatest transmission is shown after heat treatment at  $500^\circ$ . Treatment at higher temps. caused surface deterioration, whilst treatment below this temp. produced a partial restoration only. Colorations produced by the artificial solarisation disappear with heat treatment at  $200$ – $300^\circ$ . The power to emit thermoluminescence and the coloration seem to disappear simultaneously. As the temp. is raised, the intensity of the thermoluminescence increases but its duration diminishes.

W. R. ANGUS.

**Analysis of clays.** O. REBUFFAT (Giorn. Chim. Ind. Appl., 1931, 13, 368–369).—When subjected to the action of a deflocculating liquid a mixture of  $\text{SiO}_2$  gel and Al hydrosilicates, the  $\text{SiO}_2$  is dispersed in a larger proportion and the suspended portion becomes increasingly richer in Al hydrosilicates. The opposite occurs during flocculation. The progress of separation may be ascertained by determining the  $\text{H}_2\text{O}$  of hydration of the fractions obtained; when flocculated  $\text{SiO}_2$  yields its  $\text{H}_2\text{O}$  of hydration at  $110^\circ$ , whilst the Al hydrosilicates at the same temp. retain it completely or almost completely.

O. F. LUBATTI.

## PATENTS.

**Giving coloured glasses a reddish tinge.** DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 354,566, 26.8.30. Ger., 12.9.29).—Glass coloured by the addition of oxides of Pr or Nd may be tinted to a large range of colours by incorporating a small quantity of a red colloid colouring matter, e.g., Au, Se, or  $\text{Cu}_2\text{O}$ .

C. A. KING.

**Manufacture of laminated materials [glass, wood, etc.].** NEWTEX SAFETY GLASS CO., LTD., and W. C. DOUGAN (B.P. 355,604, 20.11.30).—The materials, e.g., alternating sheets of glass and cellulose acetate, are assembled in a solution of an initial glycerol-phthalic acid condensation product in, e.g., a mixture of Et and

Bu lactates and  $\text{CH}_2\text{Cl}_2$ , and, after removal, are consolidated by compression at 300 lb./sq. in. at 90–130°.

L. A. COLES.

**[Acid-resisting] enamels.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 354,362, 7.5.30. Ger., 21.5.29).—High acid-resistance is obtained without loss of brilliance by substituting the  $\text{B}_2\text{O}_3$  by a eutectic mixture of  $\text{Li}_2\text{O}$  and  $\text{SiO}_2$  in addition to  $\text{Pb}$  (as  $\text{Pb}_3\text{O}_4$ ).

C. A. KING.

**Glass-melting furnaces.** L. MELLERSH-JACKSON. From HARTFORD-EMPIRE CO. (B.P. 355,555, 27.9.30).

**[Revolving pot-furnace for] manufacture of glassware.** UNITED GLASS BOTTLE MANUFACTURERS, LTD., and E. A. COAD-PRYOR (B.P. 355,929, 12.8.30).

**Method and apparatus for treating the edges of laminated glass.** B. L. GREGG, Assoc. of F. N. CAMPBELL, J. MYATT, and F. M. GREGG (B.P. 355,541, 16.9.30. U.S., 17.9.29).

## IX.—BUILDING MATERIALS.

**Setting and hardening of Portland cement.** A. C. DAVIS (Cement, 1931, 4, 893–899).—The theories of colloidal and cryst. setting are discussed. Whilst it was thought previously that the max. formation of tricalcium silicate was necessary to develop the greatest strength in a reasonable time, the recently introduced aluminous cements rather negative this view. The addition of gypsum, or in some instances preferably plaster of Paris, can be more relied on for a slow and uniform set than “weathering” or steaming the clinker, though the latter method is useful in conjunction with the addition of gypsum. The use of “hot” cement fresh from the grinding plant has been found to possess no disadvantage in practice.

C. A. KING.

**Influence of “tripel” on the mechanical properties of Portland cement.** P. P. BUDNIKOV, L. G. GULINOV, and V. I. TOKAREV (Ukrain. Chem. J., 1931, 6, [Tech.], 13–16).—The addition of “tripel” (hydrated silicic acid) to Portland cement in certain cases increases its mechanical strength.

R. TRUSZKOWSKI.

**Chemical action of aggressive waters on cement.** J. O. ROOS AF HJEBMSÄTER (New Internat. Assoc. Test. Mat., Sept., 1931, 22 pp.).—Concrete deteriorates chiefly because of the dissolution of free  $\text{Ca}(\text{OH})_2$  and combined  $\text{CaO}$  by percolating water, the rate of dissolution depending on the  $\text{CO}_2$  or  $\text{CaHCO}_3$  content of the water. Different Portland cements proved to be practically alike in extraction tests. Carbonation of the surface of the concrete or the addition of certain acid compounds and pozzuolanas containing reactive  $\text{SiO}_2$  slightly reduced the extraction of  $\text{CaO}$ .

C. A. KING.

**Dolomitic Roman cements and hydraulic dolomite.** E. ROSENSTEIN (Latvij. Univ. Raksti, 1931, 2, 25–144).—The tensile strength of dolomitic Roman cement is not directly proportional to the  $\text{Al}_2\text{O}_3$  content or the content of  $\text{SiO}_2$  in the mortar, but there is an optimum kiln temp. for each type of raw material. Heterogeneous specimens exhibit signs of sintering at 1100° and are readily “burnt.” In nearly all cases addition of  $\text{CaSO}_4$  (up to 3%) produces an increase of

strength. After air-curing, mortars prepared with incompletely ignited dolomite are of comparable strength with those prepared with the fully ignited material; curing in moist air or  $\text{H}_2\text{O}$  results in a weaker mortar. Mortars of hydraulic dolomite or dolomitic Roman cement are stronger after 7 days’ air-curing than after considerably longer moist curing, and the 28-day strength of the air-cured hydraulic mortars alone is greater than that of the moist-cured specimens. The strength of moist-cured mortars consisting of partly sintered dolomitic limestone increases gradually and finally exceeds that of the air-cured mortars. In presence of  $\text{CaSO}_4$  crystallisation always takes place: the tetragonal form of  $\text{Ca}(\text{OH})_2$  appears only after a considerable period, and is therefore probably the more stable form.

H. F. GILLBE.

**Suggested investigations of high-alumina cements.** P. H. BATES (New Internat. Assoc. Test. Mat., Sept., 1931, 6 pp.).—Recommended investigations are: the correlation of laboratory and field strength results, equilibria of the constituents, conditions under which heat is generated during setting, optimum  $\text{H}_2\text{O}$ : cement ratio, determination of thermal and moisture dilatation, resistance to action of saline waters, and determination of moduli of elasticity.

C. A. KING.

**Grading aggregates. II. Application of mathematical formulæ to mortars.** F. O. ANDEREGG (Ind. Eng. Chem., 1931, 23, 1058–1064; cf. B., 1931, 950).—Continuous grading gives the best results for mortars, including masonry, stucco, and cast stone mixes, because of the remarkable workability combined with good packing. Intermittent grading, modified slightly to improve workability, gives good results and is being applied commercially in making certain concrete products.

E. S. HEDGES.

**Designing concrete for high strength, low permeability, and low shrinkage.** W. A. SLATER (New Internat. Assoc. Test. Mat., Sept., 1931, 16 pp.).—Requirements to obtain these properties are reduced to combining the min. quantity of  $\text{H}_2\text{O}$  with cement and suitable aggregates so as to give a workable mixture. The max. size of a well-graded coarse aggregate should be as large as conditions permit; the fine aggregate should be  $< \frac{1}{4}$  in. diam. and have high density (100 lb./cu. ft.), and the proportion of sand should be somewhat less than that which gives the max. wt. per cu. ft. of mixed material. Curing should be under conditions which prevent the evaporation of  $\text{H}_2\text{O}$  for as long as possible.

C. A. KING.

**Durability and strength properties of Swedish coniferous timber.** R. SCHLYTER (New Internat. Assoc. Test. Mat., Sept., 1931, 20 pp.).—During forest storage, timber should always be decorticated to minimise rot. Poles of pine and spruce erected in a green condition showed greater resistance to rot than seasoned poles. Greater resistance was observed when the bottom of the pole was surrounded by stones rather than by soil or sand or concrete. Good results were obtained by providing the underground portions with a poison bandage, e.g.,  $\text{HgCl}_2$ ,  $\text{ZnCl}_2$ ,  $\text{CuCl}_2$ ,  $\text{NaCl}$ . Paraffin or creosote increased the life, though not to a degree commensurate with the cost of treatment, but coal tar

in all cases increased the damage from rot. The strength of timber for aircraft purposes is discussed, including fatigue bending strength. C. A. KING.

**Development of the mechanical testing of timber in Great Britain, with a note on preservative treatments.** C. J. CHAPLIN (New Internat. Assoc. Test. Mat., Sept., 1931, 18 pp.).—The methods of testing used in the Forest Products Research Laboratory are described and the necessity for matching and grading timber when assuming an average strength is emphasised. Variations in the strength of clear timber are due largely to differences in forest conditions, cellular structure, chemical composition, and moisture content, and to the method of seasoning. Methods of impregnation against dry rot and fire are noted.

C. A. KING.

**Apparatus for testing corrosion- and erosion-resistance of constructional materials.** W. DENECKE (Chem. Fabr., 1931, 4, 358—359).—A propeller is formed of the material and is used to circulate and agitate vigorously the corrosive fluid. Air or other gases, or solid particles, may readily be introduced into the liquid, and the temp. may be adjusted to any required value. The formation of a protective film is prevented by the rapidity of the stirring.

H. F. GILLBE.

**Universal standardisation of tests for mortar and cement concrete.** I. VANDONE (New Internat. Assoc. Test. Mat., Sept., 1931. 5 pp.).

**More important properties of concrete and their significance and application in practice.** O. GRAF (New Internat. Assoc. Test. Mat., Sept., 1931. 12 pp.).

**Change in dimensions of concrete under compression.** F. EMPERGER (New Internat. Assoc. Test. Mat., Sept., 1931. 11 pp.).

**Testing of woods.** M. MONNIN (New Internat. Assoc. Test. Mat., Sept., 1931. 31 pp.).

**Technical methods for testing wood.** K. RYSKA (New Internat. Assoc. Test. Mat., Sept., 1931. 28 pp.).

**Mechanical properties of various woods.** A. PERFETTI (New Internat. Assoc. Test. Mat., Sept., 1931. 5 pp.).

**Asphalt and bitumens, their suitability for use in building and road-construction.** H. SUIDA and W. JANISCH (New Internat. Assoc. Test. Mat., Sept., 1931. 12 pp.).

**Grading aggregates. Ageing of materials.**—See I. Rotary kiln in cement manufacture. **Testing of bituminous materials.**—See II. Defective tiles.—See X.

## PATENTS.

**Sintering and calcining of cement and like materials.** METALLGES. A.-G. (B.P. 355,564, 7.10.30. Ger., 16.11.29).—The material is heated firstly with sufficient fuel to expel  $\text{CO}_2$  and  $\text{H}_2\text{O}$ , and then in a blast-furnace to cause sintering. The first operation may be effected in a blast furnace or in a rotary tube furnace, in which case the addition of fuel is unnecessary, and 1—5% of clinker screenings, blast-furnace slag, etc. is added prior to the second operation, to assist the sintering. L. A. COLES.

**Burning of slurry-like material.** E. RØNNE (B.P. 354,565, 25.8.30).—Slurry is dried and burned in two or more independently rotating tubes arranged in succession. The drying portion, which may be non-uniform in cross-section, is furnished with a quantity of heat-transmitting bodies, e.g., chains, rings, or wire netting. C. A. KING.

**Cement.** T. M. CAVEN, Assr. to F. R. HAZARD (U.S.P. 1,791,591, 10.2.31. Appl., 9.2.27).—A monolithic furnace lining is formed by setting silica bricks with a cement containing approx. 5% of colloidal  $\text{SiO}_2$ , 10% of diatomaceous  $\text{SiO}_2$ , and 85% of relatively coarse  $\text{SiO}_2$ . In an example, quartzite is used as the starting material. C. A. KING.

**Feeding of cement material to kilns.** P. T. LINDHARD, Assr. to F. L. SMITH & Co. (U.S.P. 1,793,499, 24.2.31. Appl., 5.11.27).—In a kiln in which the slurry is injected in an atomised condition, the dust escaping with the gases is collected and fed by screw conveyors through peripheral openings in the kiln wall at a point at which the section of the spray cone practically fills the kiln. C. A. KING.

**Manufacture of Portland cement.** J. H. COLTON, Assr. to PACIFIC PORTLAND CEMENT Co. (U.S.P. 1,791,800, 10.2.31. Appl., 4.2.26).—Portland cement clinker is pulverised, mixed with a raw batch, and re-burned. C. A. KING.

**Portland cement.** A. LUNDTEIGEN and G. O. GARDNER, Assrs. to ASH GROVE LIME & PORTLAND CEMENT Co. (U.S.P. 1,794,526, 3.3.31. Appl., 11.7.27).—A quick-hardening cement is formed by grinding cement clinker with < 1% of a mixture of  $\text{NaCl}$  or  $\text{CaCl}_2$ , the salt being in large excess, and an insol. soap, which acts as a water-repellent material. A small quantity of  $\text{CaO}$  may be incorporated. C. A. KING.

**Portland cement modifying composition.** M. TOCH, Assr. to STANDARD VARNISH WORKS (U.S.P. 1,793,143, 17.2.31. Appl., 23.9.27).—An additive colouring and hardening composition consists of a dry mixture of  $\text{CaCl}_2$  and a colloiddally precipitated inorg. salt, e.g., Al silicate, Ca aluminates, or  $\text{BaSO}_4$ . Thus a mixture containing 60 pts. of bright red  $\text{Fe}_2\text{O}_3$ , 30 pts. of  $\text{CaCl}_2$ , 5 pts. of colloidal Al silicate precipitated in presence of  $\text{NH}_4$  tannate, and 5 pts. of  $\text{BaCl}_2$  is suitable. C. A. KING.

**Composition of matter [for addition to Portland cement].** C. E. MILLER (U.S.P. 1,791,630, 10.2.31. Appl., 8.3.26).—A mixture of  $\text{CaCl}_2$  and diatomaceous earth is finely ground and added to concrete batches. Such a mixture is non-deliquescent. C. A. KING.

**Manufacture of cement.** J. F. GODDARD, and SUPER CEMENT, LTD. (B.P. 354,502, 27.6.30).—Portland cement is rendered more impermeable in use by incorporating gypsum containing tannin, the latter being not more than 0.05 wt.-% of the clinker. C. A. KING.

**Making cement impervious.** H. E. POTTS. From CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER, A.-G. (B.P. 355,583, 29.10.30).—Solutions containing K Al hydroxide (or a mixture of it with Na Al hydroxide), prepared by dissolving  $\text{Al}(\text{OH})_3$  in KOH solution, are used for working up the cement. L. A. COLES.

**Chlorine-treated cement.** H. M. OLSON (U.S.P. 1,792,755, 17.2.31. Appl., 16.1.29).— $\text{Cl}_2$  is "mixed" with cement during the last grinding operation. The cement absorbs a crit. quantity of  $\text{Cl}_2$ ; the product is non-hygroscopic and has rapid set and early strength.

C. A. KING.

**Manufacture of dental cement.** J. E. THOMSEN (U.S.P. 1,792,200, 10.2.31. Appl., 20.9.28).—Dry mixtures containing 145 pts. by wt. of a silicate cement and 40–60 pts. of  $\text{H}_3\text{PO}_4$ , or 150 pts. of a basic metal oxide cement and 100–150 pts. of a phosphoric acid with a ratio  $1\frac{1}{2}$ –2 mols.  $\text{H}_2\text{O} : 1\text{P}_2\text{O}_5$  are claimed. For use,  $\text{H}_2\text{O}$  is added to form a paste.

C. A. KING.

**Making plaster.** F. D. SNELL (U.S.P. 1,792,661, 17.2.31. Appl., 30.3.29).—The suction of a wall plaster is reduced without interfering with the porosity by adding about 0.16 lb. of lime soap per cu. yd. of wet plaster.

C. A. KING.

**Manufacture of porous building materials.** E. C. BAYER (U.S.P. 1,794,272, 24.2.31. Appl., 23.3.28. Denm., 23.2.27).—A tenacious foam is mixed with a  $\text{CaO}$ -cement mortar such as is formed from 6.3% of  $\text{CaO}$ , 63.5% of sand, 2.5% of aluminous cement, 5% of ground quartz, and 22.7% of  $\text{H}_2\text{O}$ . A suitable foam consists of 2% of resin soap, 0.4% of gelatin, and 97.6% of  $\text{H}_2\text{O}$ . After 12–36 hr. the moulded material is cured in steam at 8–10 atm. pressure for 8–10 hr.

C. A. KING.

**Manufacture of coloured granules.** R. L. ATKINSON, Assr. to A. D. LITTLE, INC. (U.S.P. 1,792,058, 10.2.31. Appl., 13.1.30).—Argillaceous shale either raw or artificially coloured with oxide pigments is ground and the product (passing 8-mesh, but held by 35-mesh) is heated in a controlled atm. to a temp. below the sintering but above the hardening point of the shale. Heating is carried out in a rotary kiln through which air is passed if a red to brown colour is desired, and reducing gases for grey to black. The product may be incorporated into roofing tiles, cement blocks, etc.

A. R. POWELL.

**Manufacture of (A) bitumen-concrete mixtures and sand-bitumen mixtures, and (B) tar-bitumen-concrete mixtures and tar-bitumen-sand mixtures.** H. MILKE and J. OBERBACH (B.P. 354,433–4, 26.5.30. Ger., [A] 14.10.29, [B] 9.12.29).—(A) Heated mineral substances, which have been compounded according to the min. hollow-space principle, are mixed with heated bitumen (natural or artificial), of penetration 60–300 on the Richardson scale, and a filler (ground natural asphaltic limestone). The filler is heated in admixture with the other mineral substances (to 100–200°) before admixture with the bitumen. (B) Instead of bitumen, mixtures of coal tar and bitumen may be used.

A. B. MANNING.

**Materials for use in construction of roads, roofs, etc.** RUBBERPHALTE, LTD., and C. M. C. HUGHES (B.P. 353,919, 25.4.30. Cf. B.P. 330,440; B., 1930, 771).—Peat moss, coconut fibre, or like fibrous materials, and stone or sand, are mixed together with a binding compound consisting of a mixture of fuel oil, bituminous pitch (petroleum residue of m.p. about 120°), and a native bitumen.

A. B. MANNING.

**Charging of kilns.**—See I. Bituminous dispersions.—See II. Fireproof binding.—See VI. Laminated wood.—See VIII. Coating compositions. Floor coverings.—See XIII. Cellular material for plants.—See XVI.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Retention of flue dust in the blast furnace by the Eichenberg process.** P. GEIMER, A. WAPENHIENSCH, and A. SPONHEUER (Stahl u. Eisen, 1931, 51, 1073–1077).—In the Eichenberg process,  $\text{H}_2\text{O}$  and gas are sprayed into the throat of the furnace in such a way that a rotary motion is imparted to the rising gases, which are cooled by the evaporation of the  $\text{H}_2\text{O}$  and their velocity is thus reduced, whereby the dust is deposited in the upper layers of the charge. With a correct arrangement of the  $\text{H}_2\text{O}$  jets at least 50% of the normal dust losses can be retained in the furnace without disturbing the regular running of the furnace or increasing fuel consumption.

A. R. POWELL.

**Modification of Penny's method for determining iron in ores.** R. P. HUDSON (Chemist-Analyst, 1931, 20, No. 3, 6–7).—Titration using  $\text{NiPh}_2$  in presence of  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$  is recommended.

CHEMICAL ABSTRACTS.

**Production of pig iron with a high alumina content of the charge.** M. PASCHKE and E. JUNG (Arch. Eisenhüttenw., 1931–2, 5, 1–8).—Pig Fe has been successfully produced over long periods by smelting scrap Fe with limestone, ferruginous bauxite, and coke in the usual type of Fe blast furnace using a blast preheated to 550°. The charge contained 23–55% of scrap Fe and 27.30% of coke and the fluxes were so adjusted that the slag contained 45% ( $\text{Al}_2\text{O}_3 + \text{TiO}_2$ ), 40%  $\text{CaO}$ , 7%  $\text{SiO}_2$ , and 2–3%  $\text{FeO}$ ; the Fe averaged 5% C, 0.3% Si, 0.5% Mn, 0.13% P, 0.02% S, and 0.02% Al. The high C content of the Fe is attributed to the high viscosity and m.p. (about 1550°) of the slag, the low Si content to the small amount of  $\text{SiO}_2$  in the charge, and the low S content to dissociation of the sulphides at the high temp. necessary to melt the slag properly. The slag itself has only a low desulphurising action, and for all practical purposes may be considered to be a neutral and not a highly basic slag as its composition would suggest. After grinding the slag may be used directly as ciment fondu.

A. R. POWELL.

**Effect of low temperature on the shock-resisting properties of new wrought-iron chain.** H. J. GOUGH and A. J. MURPHY (Proc. Inst. Mech. Eng., 1930, 1159–1223).—Impact tests on smooth and notched bars of best Yorkshire chain-iron over temps. ranging from 200° to –80° have shown that smooth specimens suffer little change of shock-resistance over the range, and that notched specimens become very brittle at the lower temps., the greatest rate of loss of strength taking place between 20° and 0°. Impact tensile tests on new welded chain of high quality and workmanship over the range –80° to 20° show considerably reduced strength at the lower temps., the decrease being attributed to the notch effect arising at the scarf formed in welding, from which part of the links the fractures usually originate.



The loss in shock-resistance does not persist when a chain or notched bar is reheated to room temp.

E. H. BUCKNALL.

#### Structure and mechanical properties of cast iron.

J. G. PEARCE (New Internat. Assoc. Test. Mat., Sept., 1931, 8 pp.).—Correlation of results on mechanical tests with composition can be made only for one set of melting conditions, as neither the temp. nor time of melting is sufficient to clear the graphite. Under const. conditions the strength of similar cast irons decreases as the section increases, and in the grey range decreases as Si increases. Difference in mechanical properties of irons of the same composition denotes difference in melting conditions.

C. A. KING.

#### Testing of cast iron.

E. DÜBI (New Internat. Assoc. Test. Mat., Sept., 1931, 34 pp.).—Numerous tests on various types of cast Fe have shown that the separately cast test piece is satisfactory in enabling conclusions to be drawn as to the mechanical properties of the castings of which it is a specimen. Bending and tensile tests are sufficient for the evaluation of cast Fe; the hardness is a linear function of the tensile strength.

A. R. POWELL.

#### Cast iron and methods of testing it.

E. PIVOVARSKY (New Internat. Assoc. Test. Mat., Sept., 1931, 24 pp.).—Recent work on the structure and the physical and mechanical properties of cast Fe is critically reviewed.

A. R. POWELL.

#### Endurance limit of the [German] constructional

steel St. 52. H. BUCHHOLTZ and E. H. SCHULZ (Stahl u. Eisen, 1931, 51, 957—961).—The mechanical properties, with especial reference to fatigue, of the standard German constructional steels 37 and 52 (composition not stated) have been compared with those of a 3% Ni steel. In static and dynamic tests of round rods the strength of steel 52 is 50% above that of steel 37. The flow stress of both steels is increased to 2.4 times its normal value by very deep notches, whilst the resistance to alternating stresses is reduced to less than half its normal value, and the relative superiority of 52 over 37 in structures with sharp edges and recesses is about 30%. The fact that the 3% Ni steel suffers a similar and sometimes greater reduction in resistance to alternating stresses by notching and yet has proved of great value in practice shows that these tests cannot be translated directly to the practical scale. The higher yield point of steel 52 under static loads is probably the cause of the superior endurance strength of perforated rods of this steel compared with steel 37; both steel 52 and the 3% Ni steel are 30% superior to steel 37 in endurance bending tests, and on the whole there is little to choose between the two first-named steels.

A. R. POWELL.

#### Rapid determination of the creep limit of steel.

A. POMP and W. ENDERS (New Internat. Assoc. Test. Mat., Sept., 1931, 17 pp.).—A more detailed account of work the results of which have been already published (B., 1931, 443).

A. R. POWELL.

Advances in [ultra-violet] microscopy [as applied to steel]. F. F. LUCAS (New Internat. Assoc. Test. Mat., Sept., 1931, 24 pp.).—Improvements in technique reveal minute quenching cracks in martensitic needles of hardened steel, of the order of 25 atom-diams. in

width and 1000 in length. Their possibilities as sources of fatigue failure are discussed.

C. W. GIBBY.

#### Case-carburising [of iron and steel] in fused cyanides.

H. B. NORTHRUP (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 275—279).—The rate of decomp. of fused NaCN is appreciably reduced by admixture with NaCl and Na<sub>2</sub>CO<sub>3</sub> without impairing its case-hardening efficiency. The best practice comprises starting a bath with a mixture of 30% NaCN, 35% NaCl, and 35% Na<sub>2</sub>CO<sub>3</sub> and determining by analysis the rate of loss of NaCN hourly over a period. When this is established, the requisite quantity of NaCN is added every hr. to maintain the bath at about 25% NaCN.

A. R. POWELL.

#### Mechanism of the galvanising process. Solubility of pure iron in molten zinc.

H. GRUBITSCH (Stahl u. Eisen, 1931, 51, 1113—1116).—The solubility of Armco Fe in pure Zn is <0.5% below 470°, but above 480° it rises rapidly to a max., depending on the time of contact, at 495° and then falls to a min. at 515—540°, according to the time of contact. With further rise in temp. the solubility rises again almost linearly and becomes much less dependent on the time.

A. R. POWELL.

#### Determination of silicon in ferrosilicon.

W. HARTMANN (Z. anal. Chem., 1931, 85, 185—187).—The sample is decomposed by fusion in an Fe crucible with NaOH followed by addition of Na<sub>2</sub>O<sub>2</sub> when the fusion is quiet. The product is dissolved in HCl, the solution evaporated to dryness, and the residue heated at 130—140° to dehydrate the SiO<sub>2</sub>. The analysis is then finished as usual, the first filtrate being again evaporated for the recovery of more SiO<sub>2</sub>.

A. R. POWELL.

#### Determination of oxygen and nitrogen in irons and steels by the vacuum fusion method.

H. C. VACHER and L. JORDAN (Bur. Stand. J. Res., 1931, 7, 375—401).—The apparatus and technique for determining H<sub>2</sub>, N<sub>2</sub>, and O<sub>2</sub> in ferrous materials are described. Results reproducible to 0.002% have been obtained for N<sub>2</sub> and O<sub>2</sub>. The presence of Al<sub>2</sub>O<sub>3</sub> and Mn oxide interferes with the accuracy of the O<sub>2</sub> determination, but not with the N<sub>2</sub> determination.

W. R. ANGUS.

#### Meaning of the notched-bar impact test for investigation and for acceptance test purposes.

R. H. GREAVES (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—The essential feature of the test is concentration of stress by the notch, which may lead to a shear with considerable deformation or a tensile break with little deformation. A number of tests should be made before consideration. A bad impact figure always indicates a decided liability to fail under localised stress, and the utility of the test as an exclusion test has been largely due to its capacity for detecting temper brittleness in Ni-Cr and other steels.

C. A. KING.

#### Impact tests on notched bars.

R. ZOJA (New Internat. Assoc. Test. Mat., Sept., 1931, 9 pp.).—The phenomena of ageing of steels can be studied by a modification of the notched-bar impact test. The specimen is first deformed by allowing the pendulum to fall from a height insufficient to cause rupture, and the work consumed ( $L_1$ ) in causing a certain deformation

is measured. Then, after ageing for the desired time, the specimen is completely broken in the usual way and the work ( $L_2$ ) and deformation are again measured.  $L_1$  and  $L_2$  are then expressed as percentages of  $L_0$  (the value obtained in the usual impact test), and the deformations corresponding with  $L_1$  and  $L_2$  are similarly expressed as percentages of the deformation corresponding with  $L_0$ . A characteristic graph for every steel is then obtained by plotting  $L_1$ ,  $L_2$ , and  $L$  ( $= L_1 + L_2$ ) against the corresponding deformations, all expressed as percentages.

A. R. POWELL.

**Impact tests—standard test pieces.** M. SCHMIDT (New Internat. Assoc. Test. Mat., Sept., 1931, 7 pp.).—The Mesnager type of test piece with a sharp notch is recommended.

A. R. POWELL.

**Mechanism of deformation in tensile test pieces.** • N. DAVIDENKOV and G. SAJZEV (Stahl u. Eisen, 1931, 51, 1116—1117). W. TAFEL (*Ibid.*, 1117).—A criticism of the work of Tafel and Scholz (Arch. Eisenhüttenw., 1929—30, 3, 545) and a reply.

A. R. POWELL.

**Fatigue [of metals].** P. LUDWIK (New Internat. Assoc. Test. Mat., Sept., 1931, 17 pp.).—The resistance to alternating stresses of metals with polished surfaces is dependent on the resistance to deformation and not on the capacity for deformation. For most metals and alloys, except cast Fe, V2A steel, scleron, and bronzes, the resistance to alternating bending stresses  $\sigma_b$  (for  $10^7$  cycles) is about  $\frac{1}{10}$  the sum of the ball hardness (in kg./sq. mm.) and the ultimate shear stress. For steels,  $\sigma_b$  is equal to 0.3 times the sum of the elastic limit (0.2% elongation) and the tensile strength. Endurance bending and torsion tests on notched bars of various steels show that notches are particularly dangerous in very hard steels, and that no simple relationship exists between the damping effect and sensitivity to notching. From prolonged corrosion tests in sea-water it appears that, even by heat treatment, the corrosion fatigue limit for  $10^7$  cycles of bending stress cannot be raised above 7 kg./sq. mm. for light metals and 14 kg./sq. mm. for steels. The fatigue fracture, under corrosive conditions, of pure Al and soft Fe is generally of an intercryst. nature. The resistance to alternating torsion stresses of all the metals tested except cast Fe is about 0.575 times the resistance under alternating bending stresses.

A. R. POWELL.

**[Strength of] metals at high temperatures.** J. GALIBOURG (New Internat. Assoc. Test. Mat., Sept., 1931, 9 pp.).—Tensile tests on metals at high temps. show that there is neither a true elastic limit at all temps., nor a limit towards which flow tends to occur under a given load; hence it appears that the strength of a metal at any given temp. and under a given load can be defined only by the parameter of a flow-time curve. The limit at which first signs of flow appear may be determined by means of the Martens mirror elasticity meter, which has a sensitivity of 0.001 mm.

A. R. POWELL.

**First report of the Corrosion Committee.** IRON AND STEEL INDUSTRIAL RESEARCH COUNCIL (Iron and Steel Inst., 1931. Advance copy, 250 pp.).—The scope of a proposed extensive research into the nature

and causes of the corrosion of Fe and steel is described in detail. The work comprises exposure of numerous types of Fe and steel in widely different atms. and a series of correlated laboratory tests. The intermittent salt-spray test carried out under carefully controlled standardised conditions has been found to give comparative results in good agreement with those obtained in field tests. As an index of the extent of corrosion the wt. increment is considered to be sufficiently accurate for industrial purposes provided that care be taken that the corrosion products do not drop off. A critical discussion of replies received from railway and shipping companies, engineering firms, and Fe and steel manufacturers to a questionnaire as to their experiences on corrosion is included.

A. R. POWELL.

**Corrosion of iron pipes. Defective tiles.** C. LEPIERRE (Rev. Chim. Pura Appl., 1930, [iii], 5, 95—99).—Extensive corrosion of underground Fe pipes was caused by the presence of traces of alkali and alkaline-earth chlorides and nitrates in the soil and to the existence of minute electric currents. The appearance of efflorescence on red tiles was due, not to the raw materials which had been used, nor to the action of  $H_2O$ , but to the formation of  $Al_2(SO_4)_3$  from  $SO_2$  produced during combustion of the coal used in their manufacture, which contained a high proportion of pyrites.

H. F. GILLBE.

**Life of domestic supply [water-]pipes in relation to the material, diameter, and nature of the water.** F. MEYER (Gas- u. Wasserfach, 1931, 74, 703—709).—From 1874 to 1880 cast Fe pipes of 25 mm. internal diam. were used to connect the street mains with the domestic supply of the houses, from 1881 to 1912 similar pipes of 40 mm. internal diam., and since 1913 wrought Fe pipes of 50 mm. internal diam. Curves are given showing the percentage of replacements annually during these periods; with the first type of pipes the curves rise much more rapidly than with the second type, showing that the wider pipes have the longer life. Wrought Fe pipes have so far proved superior to those of cast Fe as no replacements have been required during the past 20 years. Analyses of the raw  $H_2O$  from which the town's supplies are drawn and of the same  $H_2O$  after purification by the  $O_3$  process are tabulated for 5 years and for 2 yrs. after purification with  $Cl_2$ .

A. R. POWELL.

**Relation between pinholes, electrical conductivity, and the protective value of a pipe coating.** S. EWING (Gas J., 1931, 195, 488—489).—The possibility of determining the protection afforded by a pipe coating by conductivity measurements is demonstrated. Thin coatings are inferior to heavier coatings, and there is no combination of costs and circumstances which justifies the use of coatings of 0.01 in. or less in thickness.

C. B. MARSON.

**Corrosion of metal articles in storage. Influence of alkaline surface films.** W. E. COOPER (Ind. Eng. Chem., 1931, 23, 999—1002).—An efficiently protective film for storage purposes is given to steel articles by washing them in soap solution and roughly drying. A good drying atm. has the effect of dehydrating and partly disrupting the protective alkaline film, and a

wet atm. dilutes the film and renders the metal accessible to corrosion. Dust obtained from wrapping papers is a serious factor favouring corrosion. E. S. HEDGES.

**Combating the corrosion problem with lubricated valves.** G. F. SCHERER (Ind. Eng. Chem., 1931, **23**, 986—989).—The adaptability of Fe and steel valves of the plug-cock design to pressure lubrication is discussed, particularly with regard to increased resistance to corrosion. Factors affecting the selection of a lubricant are pointed out. R. S. HEDGES.

**X-Ray investigations of the constitution of alloys.** A. WESTGREN (New Internat. Assoc. Test. Mat., Sept., 1931, 16 pp.).—Examples are given and the nature of intermetallic compounds is discussed. C. W. GIBBY.

**Röntgenographic determination of the solubility of magnesium in aluminium.** E. SCHMID and G. SIEBEL (Z. Metallk., 1931, **23**, 202—204).—The solid solubility of Mg in Al increases in a smooth curve from 2.95% at 150° to 15.35% at the eutectic line (451°). The results are in close agreement with those of Dix and Keller (Amer. Inst. Min. Met. Eng., Tech. Publ., 1929, No. 187), except that the increase in solubility with rise in temp. is slightly greater. A. R. POWELL.

**Effect on ageing [of aluminium alloys] of rolling after quenching.** W. FRAENKEL (Z. Metallk., 1931, **25**, 172—176).—Tests on a light alloy of the duralumin type, scleron with 6—8% Zn and 1% Li, and Al with 4% Cu and 1% Si shows that cold-work after quenching retards ageing especially in the case of alloys of the scleron type. The rates of increase of electrical resistance and hardness of duralumin during ageing in particular are greatly reduced by cold-work, but the max. in each case is greater than that attained by the unworked alloy. The hardness of the Cu—Si—Al alloys which have been cold-rolled after quenching falls during the first hr. after rolling, increases rapidly during the next 2 hr., and thereafter more slowly. Ageing of duralumin under load appreciably reduces the electrical resistance and the rate of increase of resistance with time of ageing. A. R. POWELL.

**Heat treatment and method of cooling cast tin bronzes.** H. IMAI and M. HAGIYA (Mem. Ryojun Coll. Eng., 1931, **4**, 99—110).—Cast Sn bronzes undergo an irreversible change in length on annealing at 500—650°. With increasing Sn content cast rods increase in length by increasing amounts to a max. of 0.115% with 15% Sn; further addition of Sn causes a sudden contraction to occur with 17% Sn, which increases to 0.102% with 25% Sn. These results are attributed to segregation owing to uneven cooling, and it is possible to produce in one rod zones which contract and zones which expand on annealing; this produces internal stress which may cause cracking on quenching, but may be overcome by prolonged annealing at 600° until a homogeneous structure is obtained. A. R. POWELL.

**Lead blast-furnace operations at the works of the Broken Hill Associated Smelters' Proprietary, Ltd., Port Pirle, S.A.** O. H. WOODWARD (Bull. Inst. Min. Met., 1931. Advance copy. No. 321, 27 pp.).—With a view to subsequent recovery of the Zn from the

slag, the charge at Port Pirie is adjusted so as to obtain a slag with 18% Zn; this high Zn content has a tendency to form accretions in the furnace which result in the development of a hot top and the consequent loss of Zn in the form of objectionable fume. To prevent these troubles it is essential that a uniform smelting rate be maintained, which is possible only if the coke is of such size and porosity that a proportion of it regularly reaches the tuyère zone and that no accumulations of coke occur above this zone so as to produce an excessively reducing zone in which the Zn is distilled out of the charge. In a good slag the  $(\text{ZnO} + \text{CaO}) : (\text{FeO} + \text{MnO})$  ratio should be 8 or less with 20%  $\text{SiO}_2$ , 9—9.5 with 22%  $\text{SiO}_2$ , and 7—7.5 with 18%  $\text{SiO}_2$ . The proportion of Pb in the slag is decreased by lengthening the tapping interval; this has been rendered possible by increasing the height of the tuyères from 12 to 24 in. above the bottom of the jacket, whereby the tapping interval has been increased from 10 to 30 min. and the Pb content of the slag reduced from 2% to <1.5%. An average charge consists of 6000 lb. of Dwight-Lloyd sinter (8.27%  $\text{SiO}_2$ , 11.2% FeO, 2.8% MnO, 6% CaO, 2.7%  $\text{Al}_2\text{O}_3$ , 10.6% Zn, 3.1% S, 47% Pb), 1000—1200 lb. of old slag, 660—720 lb. of coke, and 60—150 lb. of ironstone. The slag fall is 1.57 tons per ton of Pb and assays 19.8%  $\text{SiO}_2$ , 28.6% FeO, 5.8% MnO, 13%  $(\text{CaO} + \text{MgO})$ , 5.8%  $\text{Al}_2\text{O}_3$ , 17.5% Zn, 1.9% Pb, and 2.3% S. A. R. POWELL.

**Determination of lead ores containing barium.** L. GUZELJ (Arh. Hemiju, 1931, **5**, 204—211).—When such ores are treated, the dissolution of  $\text{PbSO}_4$  by  $\text{NH}_4\text{OAc}$  according to the ordinary technique is incomplete, owing to adsorption of Pb on  $\text{BaSO}_4$ . In such cases the sulphate ppt. should be boiled with excess of  $\text{NH}_4\text{OAc}$ . R. TRUSZKOWSKI.

**Physical chemistry of flotation.** H. FREUNDLICH (Trans. Amer. Electrochem. Soc., Sept., 1931, **60**, 27—32).—The phenomena involved in flotation are interpreted on the basis of capillary action, particular emphasis being given to differences in the ease of wetting of mineral and gangue. The function of adsorption phenomena, the formation of unimol. surface layers, and the time factor in flotation are discussed in the light of recent research. A. R. POWELL.

**Physico-chemical problems of flotation.** A. M. GAUDIN (Trans. Amer. Electrochem. Soc., Sept., 1931, **60**, 305—313).—The physico-chemical problems associated with the production of gas bubbles, modification of the surface of the solids, mechanics of gas-solid attachment, and the economic elimination of solid to solid interfaces are briefly outlined in the light of recent research. A. R. POWELL.

**Flocculation and froth quality in flotation.** O. C. RALSTON and L. M. BARKER (Trans. Amer. Electrochem. Soc., Sept., 1931, **60**, 39—43).—Flocculation of ore minerals is the resultant of the effects of pulp modifiers and of collector reagents. Modern sol. collecting reagents vary between causing the two extremes of "under-flocculation" and "over-flocculation" of the minerals that pass into the froth. Under-flocculated froths are dirty in appearance, voluminous, poorly

mineralised, and less selective, whilst over-flocculated froths are brilliant and metallic in appearance, heavily burdened with mineral, and flat, shallow, and too tough to flow by their own wt. or to be easily raked off. Experiments with the Cu ore of the United Verde mine showed that by a judicious admixture of collectors of each of the above types a good selective froth can be obtained. Aerofloat reagents invariably produced a poor froth of the under-flocculated type containing much  $\text{FeS}_2$ , but substitution of these reagents by Et xanthates with pine oil as frother much improved the results. Amyl xanthates gave a tough over-mineralised froth too heavy to separate cleanly, but a mixture of 0.16 lb. of Et xanthate and 0.08 lb. of amyl xanthate per ton in a CaO circuit gave an excellent froth with a rejection of over 70% of the  $\text{FeS}_2$  in the ore.

A. R. POWELL.

**Factors affecting the flotation of gold in milling ores.** E. S. LEAVER and J. A. WOOLF (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 287—303).—Flotation tests on a number of American Au ores containing various sulphide minerals sometimes in a weathered form indicate that CaO has a depressing effect on the flotation of Au and that any CaO in the circuit will depress the Au if  $\text{CO}_2$  be passed into the pulp. The optimum  $p_{\text{H}}$  range for Au flotation is 7—10, but for some ores free from CaO better results are obtained in a pulp slightly acidified with  $\text{CO}_2$ . The use of  $\text{Na}_2\text{S}$  to sulphidise base-metal minerals for flotation may depress the Au unless much slime is present in the pulp to adsorb the alkali formed. NaCN in the circuit tends to dissolve Au and to retard flotation of sulphide minerals containing Au. When coarse Au is present it should be removed by amalgamation prior to flotation or cyanidation.

A. R. POWELL.

**Physical and mechanical aspects of the cyanide process [for gold].** A. W. ALLEN (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 281—285).—Grinding of a Au ore in cyanide solution may cause loss of Au owing to adsorption of the  $\text{Au}(\text{CN})_2$  ion by colloids present; grinding in a  $\text{Ca}(\text{OH})_2$  solution is therefore recommended as the colloid then adsorbs and is flocculated by the Ca ion. The pulp should be thickened and dewatered before cyaniding. Removal of Au from the leach liquors with activated charcoal has the advantage of not fouling the solution and of yielding a ppt. from which the Au is more readily recovered by smelting;  $\text{Na}_2\text{S}$  also liberates the Au from the charcoal.

A. R. POWELL.

**Cyanides in the metallurgy of gold and silver.** E. M. HAMILTON (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 255—261).—The application of the cyanide process to the treatment of Au and Ag ores with and without a preliminary flotation is outlined.

A. R. POWELL.

**Cyanides in relation to silver and gold[plating] solutions.** E. J. DOBBS (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 33—36).—KCN is preferable to NaCN for the preparation of Au- and Ag-plating baths. Ag deposits from the K baths are smoother, brighter, and softer and can be made at a higher c.d. K baths also have a higher throwing power than Na baths and the Ag more readily covers soldered joints. For

gilding, KCN allows for a greater margin in the plating range without spoiling the colour of the Au deposit.

A. R. POWELL.

**Development of cyanidation with increasing complexity of ores.** E. S. LEAVER (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 15—20).—Au has been successfully extracted from telluride ores with CNBr, but roasting followed by leaching with KCN is usually the more satisfactory process. Complex Ag ores containing  $\text{MnO}_2$  are refractory to cyanidation, but are amenable to treatment after a reducing roast or after leaching with  $\text{SO}_2$  to remove Mn compounds. In many cases removal of sulphides and most of the Au by flotation followed by KCN leaching of the tailings is the most economical method of recovery. The precipitation of the Au and Ag is almost exclusively effected by Zn dust after a pretreatment to remove dissolved  $\text{O}_2$ .

A. R. POWELL.

**Electrolytic manufacture of aluminium, and aluminium plating.** V. O. PLOTNIKOV, M. M. GRACJANSKI, and M. S. FORTUNATOV (Ukrain. Chem. J., 1931, 6, [Tech.], 33—48, and J. Chem. Ind. Russ., 1931, 8, 829—835).—Al cannot be purified by electrolysis of the fused complex  $\text{AlCl}_3 \cdot 3\text{NH}_3$ , as Al is redissolved from the cathode with the production of AlN and  $\text{H}_2$ . Mixtures (2 : 1 or 3 : 2) of  $\text{AlCl}_3$  and NaCl yield highly pure Al, containing only 0.043—0.06% Fe, 0.026% Si, and 0.01% Na. Al anodes containing 36% Fe are used, the c.d. being 5 amp./sq. dm.; as the c.d. is increased from 1 to 50 amp. the deposit changes from white crystals to a grey, spongy mass. The anode sludge is isolated from the electrolyte by an asbestos diaphragm. Al plating is performed in Al tanks, which serve as the anode. Volatilisation of  $\text{AlCl}_3$  was not observed in closed tanks. The thickest plating (0.08 mm.) was obtained at 200° with a cathode c.d. of 1 amp./sq. dm. The resistance of the plating to corrosion by steam or conc. NaCl solution is greater than is that of Kahlbaum's Al sheet. A solid solution of Al in Fe intervenes between the Fe and the Al surfaces. The plating does not scale off on slight bending, but breaks when the plated sheet is bent at an angle of 45°. Al-plated Fe is more resistant to oxidation at 800—1000° than is ordinary Fe.

R. TRUSZKOWSKI.

**Present state of knowledge of fatigue of metals.** H. J. GOUGH (New Internat. Assoc. Test. Mat., Sept., 1931. 21 pp.).

**Fatigue impact and alternating bending test on rails used by Italian Railways.** P. FORCELLA (New Internat. Assoc. Test. Mat., Sept., 1931. 19 pp.).

**Development of fatigue testing in Germany.** E. H. SCHULZ and H. BUCHHOLTZ (New Internat. Assoc. Test. Mat., Sept., 1931. 26 pp.).

**Notched-bar impact test as an acceptance test.** A. STECCANELLA (New Internat. Assoc. Test. Mat., Sept., 1931. 9 pp.).

**Notched-bar impact test problem in Germany.** M. MOSER (New Internat. Assoc. Test. Mat., Sept., 1931. 14 pp.).

**Tests on cast iron—a consideration of test results obtained in Czechoslovakia.** F. PISEK (New Internat. Assoc. Test. Mat., Sept., 1931. 19 pp.).

Progress of knowledge regarding slag inclusions in iron and steel. C. BENEDICKS and H. LOEFQUIST (New Internat. Assoc. Test. Mat., Sept., 1931. 12 pp.).

Progress of metallography. J. L. HAUGHTON (New Internat. Assoc. Test. Mat., Sept., 1931. 12 pp.).

Recent development in research on the constitution of alloys. W. GUERTLER (New Internat. Assoc. Test. Mat., Sept., 1931. 15 pp.).

Elasticity, plasticity, etc. Accuracy of measurement. Behaviour of materials at high temps.—See I. Cyanide.—See VII. Testing resistance of constructional materials.—See IX. Sewage and industrial wastes. Metal surfaces for sewage.—See XXIII.

## PATENTS.

Rotary furnaces [for metals]. E. WEISS (B.P. 353,754, 13.9.30. Ger., 16.9.29).—The burner opening at the upper end of an inclined rotary furnace serves also as a charging opening, the exhaust gas discharging at the lower end. A combustion chamber in front of the burner opening may be jointed to the furnace or be detachable, forming a unit with the charging apparatus.

C. A. KING.

Ore-roasting muffled-retort furnace. H. S. BAILEY and R. A. BENNETT (Assee.) (U.S.P. 1,792,476, 17.2.31. Appl., 30.8.27).—The furnace has several superposed hearths and is rectangular in plan. It is rabbled by rollers of such a form that they effect lifting and turning of the ore, and are carried at their ends by pairs of endless chains. The floor sections can be renewed without disturbing the walls. B. M. VENABLES.

Agglomeration of pulverulent [hæmatite] ores. B. CHRISTIANSEN (U.S.P. 1,792,413, 10.2.31. Appl., 30.10.28. Swed., 8.11.27).—Hæmatite fines are ground finer than 0.04 mm., but not to the colloidal state, and the powder is compressed into blocks which are sintered in the usual way.

A. R. POWELL.

Manufacture of open-hearth steel. G. E. HILLIARD (U.S.P. 1,793,441—2, 17.2.31. Appl., [A] 4.4.28, [B] 26.3.29).—(A) Fine metal scrap is introduced into open-hearth steel furnaces in the form of a freely flowing stream of such vol. and falling at such a rate that the occluded air is consumed by the furnace gases before the metal reaches the molten bath. (B) Pig Fe is melted in an open-hearth furnace and a strongly oxidising flame is passed over the surface of the metal to oxidise the impurities, while finely divided scrap is fed in at one spot at such a rate that it reaches the surface of the bath almost in a molten condition, but is not excessively oxidised by the flame.

A. R. POWELL.

Salt-bath furnaces for heat treatment of steel and other metals. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 351,812, 22.8.30. Ger., 19.9.29. Addn. to B.P. 349,830; B., 1931, 811).—The cylinder forming the second electrode in the furnace is divided into three separate parts which are individually connected to the phases of a 3-phase current supply. The electrodes are provided with lateral projections consisting of flat surfaces so arranged as to form with the electrodes in horizontal cross-section a broken-sided hexagon.

A. R. POWELL.

Production of ferrochrome. K. M. SIMPSON (B.P. 353,406, 17.2.30. U.S., 9.10.29).—A mixture of chrome ore and carbonaceous material, in briquette form, is heated at 1200–1450° until reduction is practically complete. The excess of C is burned out and the Fe-Cr is separated from earthy residues by melting.

C. A. KING.

[Corrosion-resistant] iron, chromium, and nickel alloys. INTERNAT. NICKEL CO., INC., Assees. of (A) N. B. PILLING, (B) N. B. PILLING and D. E. ACKERMAN (B.P. 353,448 and 353,459, 21.1.30. U.S., 22.1.29).—(A) An austenitic steel alloy containing not more than 0.1% C and the proportions of Cr and Ni as represented on a graph is claimed as possessing reduced hardening properties when worked, particularly in the cold state. (B) Alloys containing Cr 8–25%, Ni 12–19%, C < 0.3%, with or without Si or Mn < 5% are claimed. A particular composition is: Ni 15%, Cr 15%, Mn 0.8%, Si 0.2%, C < 0.2%. [Stat. refs.]

C. A. KING.

Preventing corrosion of metallic surfaces. H. SUTTON and L. F. LE BROCC (B.P. 353,415, 14.4.30).—Mg or alloys rich in Mg are cleaned in dil. HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, or NaOH and immersed in a solution (p<sub>H</sub> 5–7) of alkali chromate or dichromate which may also contain NaHSO<sub>4</sub>.

C. A. KING.

Coating metal [iron with a rustproof film]. V. M. DARSEY, Assr. to PARKER RUST-PROOF CO. (U.S.P. 1,791,715, 10.2.31. Appl., 13.5.29).—The Fe article is immersed in a boiling dil. solution of an alkali chromate and then in a solution of H<sub>3</sub>PO<sub>4</sub> containing Cu and Pb salts.

A. R. POWELL.

Copper alloy and its [heat] treatment. M. G. CORSON, Assr. to G. H. COREY (U.S.P. 1,792,146, 10.2.31. Appl., 16.2.29).—A bronze bearing alloy containing Cu with 10.35% Zn and 18.4% Sn is claimed. The heat treatment comprises annealing at 650–850° until homogeneous  $\beta$  with an acicular structure is produced, quenching, reheating at 550–625° for 1–6 hr. to decompose the  $\beta$  into  $\alpha + \beta$  in which the  $\beta$  is regularly distributed throughout the  $\alpha$ , then holding at 450–525° until the  $\beta$  decomposes into a finely-dispersed hard mass of  $\alpha + \delta$ .

A. R. POWELL.

Dezincing of lead. J. O. BETTERTON, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,792,210, 10.2.31. Appl., 1.11.26).—The Pb is circulated at 355–400° through a closed vessel in which a const. atm. of Cl<sub>2</sub> is maintained, whereby the Zn is converted into ZnCl<sub>2</sub>.

A. R. POWELL.

Production of tungsten. I. G. FARBERIND. A.-G. (B.P. 353,815, 15.12.30. Ger., 31.12.29).—W ores, e.g., wolframite, are reduced at 850–900° in an atm. of H<sub>2</sub> and Cl<sub>2</sub>, the proportions of the gases preferably being about 8:1. Carbonaceous material may also be incorporated.

C. A. KING.

Production of metallic [nickel-chromium] coatings. C. G. FINK and L. C. PAU, Assrs. to CHEM. TREATMENT CO., INC. (U.S.P. 1,792,082, 10.2.31. Appl., 13.1.26).—Fe or other metal articles are plated with Ni, then heated to render the Ni plate ductile, and subsequently plated in a Cr bath under such conditions that the nascent H evolved at the cathode reduces the oxide on the Ni surface.

A. R. POWELL.

**Preparation of finely-divided metals [copper] or metal-metal compounds [zinc-copper alloys].** W. C. WILSON, Assr. to W. C. WILSON (U.S.P. 1,792,262, 10.2.31. Appl., 20.3.29).—Zn dust finer than 0.04 mm. is made into a thin paste with  $H_2O$  and the mixture poured with rapid stirring into a solution of  $CuSO_4$ . By suitable adjustment of the proportion of the reactants, Cu,  $Cu_2Zn$ ,  $CuZn$ , or  $CuZn_2$  may be obtained in a finely-divided form. A. R. POWELL.

**Welding [preparations].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 353,671, 12.7.30).—Metal for use in welding is obtained by the thermal decomp. of the corresponding carbonyls. *E.g.*, Fe wires may be made in this way containing 0.2–1% Mn, 0.01–0.15% C, and 0.1–2% of a flux. The usual fluxing agents are used. C. A. KING.

**Soldering materials.** GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 353,758, 17.9.30. Ger., 28.9.29).—Soldering metal is mixed with one or more acid amides or their derivatives; org. substances, *e.g.*, resins, may be added. C. A. KING.

**Plating of aluminium.** L. SCHULTE (U.S.P. 1,791,642, 10.2.31. Appl., 29.5.26).—Al is passivated by immersion in conc.  $HNO_3$ , then plated with a Ni-Fe alloy in a solution containing  $NiSO_4$ ,  $(NH_4)_2SO_4$ ,  $6H_2O$  10–12 oz. and  $FeSO_4$ ,  $(NH_4)_2SO_4$ ,  $6H_2O$  1–2 oz. per gal. together with  $Na_2SO_4$  to increase the conductivity. The article can then be plated with any finishing metal. A. R. POWELL.

**Furnace [for metal sheets]. Continuous-heating furnace. Sintering plant. Large single crystals. Separation of solids by flotation.**—See I. **Pickling-acid recovery.**—See VII. **Fusion-deposition of metals.**—See XI. **Coating compositions.**—See XIII.

## XI.—ELECTROTECHNICS.

**Characteristics of working of the lead accumulator.** E. DENINA and A. FRATES (L'Energia Elettrica, 1931, 8. Reprint, 32 pp.).—The variations of the internal resistance and of the e.m.f. of the Pb accumulator and of the single plates have been studied during the charging and discharging processes and also during the inversion of polarity after prolonged discharge. The internal resistance of the accumulator is very small and is due mainly to the layer of acid which is in contact with the active material in the pores of the plate. Sulphation increases the resistance by preventing access of acid to the pores and by reducing the surface of contact between the active material and the electrolyte. During discharge the internal resistance increases with the decrease in e.m.f., but during charge it drops rapidly to the lowest value, whereas the e.m.f. continues to increase gradually. The variation in the acid concentration at the positive plate has a greater effect on the resistance than that at the negative plate. The various phenomena are not accounted for by assuming  $Pb_2O_5$  to be the active material at the positive plate and also prove the non-existence of  $Pb_2SO_4$ , but they are readily explained by the theory of double sulphation. O. J. WALKER.

**Electrical purification of water.** J. BILLITER (Trans. Amer. Electrochem Soc., Sept., 1931, 60, 45–52).—When  $H_2O$  is purified in the Billiter cell the efficiency of the process and the rate at which pure  $H_2O$  is delivered depend on the diaphragm material, the best results being obtained with a high-grade ceramic diaphragm round the anode and a specially prepared asbestos diaphragm round the cathode. It is claimed that under ordinary working conditions  $H_2O$  containing 600 mg. of solids per litre can be so treated, the product averaging 6–9 mg./litre, at the rate of 1.7 litres per hr. per sq. dm. of diaphragm surface; the purified water has  $pH$  6.8–7.2 and an electrical conductivity as low as 2 to  $10 \times 10^{-7}$ . With special precautions the conductivity can be reduced to  $1.2 \times 10^{-7}$ . The degree of salt removal is directly proportional to the energy consumption until the conductivity falls below  $1 \times 10^{-6}$ , when it increases very rapidly. The current efficiency with a good diaphragm is about 12–15%. A. R. POWELL.

**Resistance thermometers for measurement of relative humidity or small temperature differences.** D. C. ROSE (Canad. J. Res., 1931, 5, 156–161).—A wet- and dry-bulb hygrometer utilising Ni-resistance thermometers and suitable for humidity measurements in the slip-stream of flying aircraft is described. The arrangement used may be employed for the measurement of small temp. differences generally. R. CUTHILL.

**Coolometer. Ageing of materials.**—See I. **Photo-element and degree of bleaching.**—See VI.  **$(NH_4)_2SO_4$ . Cyanide.**—See VII. **Al and Al-plating.**—See X. **Microporous rubber.**—See XIV.

## PATENTS.

**Electric [induction] furnace method [for heating non-conducting materials].** E. F. NORTHRUP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,794,863, 3.3.31. Appl., 3.8.28).—Lumps of conducting material, *e.g.*, Fe, having m.p. above the temp. to which the furnace is to be heated are added to the non-conducting material to be treated. J. S. G. THOMAS.

**Production of covered weld-rods for electric arc-welding.** A. F. BURGESS. From A. O. SMITH CORP. (B.P. 353,499, 22.4.30).—A mixture of a pulverised, fibrous cellulose material and a binder is moulded or extruded on a welding rod. The binder serves to retard combustion and may be kaolin,  $SiO_2$  flour, graphite, or bauxite, and a diluent, *e.g.*, a carbohydrate, the last-named serving also as a lubricant. [Stat. ref.] C. A. KING.

**Electrodes for use in electric arc-welding or the fusion-deposition of metals.** QUASI-ARC CO., LTD., and E. F. NEWELL (B.P. 355,552, 25.9.30).—An Fe core containing 1–1.5% C, 5–7% Cr, and 2–3% Mo is coated with blue asbestos flux or slag-forming covering. J. S. G. THOMAS.

**Electrolytic cell.** C. ROTH, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,793,136, 17.2.31. Appl., 26.6.26. Ger., 1.7.25).—The supply pipe, leading from a vessel in which the level of electrolyte is maintained const., ends within a chamber filled with gas at a pressure depending on the level of electrolyte in the cell, and the outlet

for electrolyte from the chamber is arranged below the level of electrolyte in the cell. J. S. G. THOMAS.

**Electrical resistance unit.** L. T. RICHARDSON and E. M. SCHMIEL, Assrs. to CUTLER-HAMMER, INC. (U.S.P. 1,793,829, 24.2.31. Appl., 2.1.25).—MgO, mixed if desired with alundum or other inert material, surrounding a resistor in a tubular casing, is hydrated, *e.g.*, by treatment with steam in an autoclave at 165°, and finally dehydrated, preferably at a red heat.

J. S. G. THOMAS.

**Electrical precipitating apparatus [for gases].** W. HOSS, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,794,615—6, 3.3.31. Appl., 9.5.28. Ger., 28.5.27).—(A) All the discharge electrode systems are supported upon a common support, and means are provided between the systems and the support for maintaining a graded p.d. in successive units or sections of the precipitating plant. (B) The potential drop is graded by mounting the electrode systems upon semi-conducting members, *e.g.*, of concrete, or by inserting layers of semi-conducting or resistance materials of different electrical conductivity in the electric circuit of the respective units.

J. S. G. THOMAS.

**[Electrical] precipitator cleaning device.** C. W. J. HEDBERG and H. A. WINTERMUTE, Assrs. to RESEARCH CORP. OF NEW YORK (U.S.P. 1,794,074, 24.2.31. Appl., 19.6.26).—A flexible device which moves between the discharge and collecting electrodes in order to clean the surface of the latter is described. J. S. G. THOMAS.

**Apparatus for electrical precipitation of suspended material from gases. Electrode for electrical precipitation apparatus.** E. ANDERSON, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,793,664—5, 24.2.31. Appl., [A] 11.12.26, [B] 9.10.29).—(A) Semi-conducting, sectional electrodes, *e.g.*, of concrete, are strung or mounted on metallic suspension members extending within the electrodes. (B) Directly contiguous cylindrical sections of semi-conducting material are mounted one above another, and completely surround and are supported by a vertically extending metallic member.

J. S. G. THOMAS.

**Apparatus for treating air to be used for food preservation causing precipitation of atmospheric humidity, therapeutic and other purposes.** R. PAPE (B.P. 355,261, 17.5.30).—Streams of air maintained, respectively, at positive and zero or negative potentials pass through separate supply tubes and mix in the treatment chamber.

J. S. G. THOMAS.

**[L-shaped laminated] magnetic cores.** BRIT. THOMSON-HOUSTON CO., LTD., Asses. of J. V. VIENNEAU (B.P. 356,066, 22.12.30. U.S., 23.12.29).

**Chemical reactions with arc discharges. Viscosimeter.**—See I. **Gas-enriching apparatus. Gas and fertilisers. Distillation of tar.**—See II. **Pure PhNO<sub>2</sub>.**—See III. **HNO<sub>3</sub>.**—See VII. **Salt-bath furnaces. Welding. Ni-Cr coatings. Al plate.**—See X.

## XII.—FATS; OILS; WAXES.

**Twitchell reagent. VIII. Emulsifying power of three kinds of Twitchell reagents.** K. NISHIZAWA and T. INOUE (J. Soc. Chem. Ind., Japan, 1931, 34, 277—

279 B. Cf. B., 1928, 291; 1929, 946; 1931, 399).—The effect of adding acids and salts on the emulsifying power of Twitchell reagents prepared from (a) C<sub>10</sub>H<sub>8</sub>, (b) C<sub>6</sub>H<sub>6</sub>, and (c) PhOH is of the same general type as is found with other reagents (*cf. loc. cit.*). Addition of salts of the respective reagents increased the emulsifying power of (a) and (c), but decreased that of (b). The presence of glycerol and free fatty acids (optimum amount 70%) increased the emulsifying power, which is not much influenced by variations in temp. between 90 and 100°. These reagents resemble most the Pfeilring reagent.

E. LEWKOWITSCH.

**Soap boiling. II. Strengthening change.** Y. KAWAKAMI (J. Soc. Chem. Ind., Japan, 1931, 34, 263—264 B; *cf. B.*, 1931, 850).—Experiments on a coconut-tallow soap show that the concentration of alkali in the half-spent lye should be > 3% to obtain complete saponification. It is recommended to boil the grained soap with steam for 4—6 hr. at a NaOH concentration of 0.4—0.5% and then to grain out and boil for a further 4—6 hr. with conc. lye.

E. LEWKOWITSCH.

**Purification of dynamite- and saponification-grade glycerin.** A. E. BRID and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1931, 20, 762—763).—The crude saponification glycerin is diluted to *d* 1.12 and CaO equiv. to 5% of the glycerin is added. The mixture is stirred for 30 min., then at 80° for a similar time, carbonated with CO<sub>2</sub> at 70° until neutral to litmus, and filtered. The filtrate is diluted to *d* 1.12, heated to 95°, and 3% (on the glycerin) of powdered charcoal added. After stirring for 1 hr. it is filtered, giving a water-white filtrate containing 60% of glycerin. This is conc. and finally treated with 3% of Nuchar for 30 min. at 70°.

E. H. SHARPLES.

**Drying and yellowing of trilinolenic glyceride.** A. C. ELM (Ind. Eng. Chem., 1931, 23, 881—887; *cf. B.*, 1930, 827).—Changes in I-addition and -substitution vals. and peroxide nos. have been followed during the drying of artificial trilinolenin. In the first stage peroxide formation seems to account quantitatively for the saturation effected. On treatment with boiling H<sub>2</sub>O the pale dried films turns yellow. Treatment of trilinolenin with BzO<sub>2</sub>H yields a keto-derivative. Tetrameric and monomeric mol. wts. for the film were obtained by cryoscopic measurements in C<sub>6</sub>H<sub>6</sub> and camphor (Rast), respectively; the oxidised fatty acids gave dimeric vals. in camphor, but these results are attributed to association in the solvents. It is suggested that in darkness the degree of polymerisation during drying is low and coloured compounds are formed, whilst in the light more highly-polymerised colourless derivatives are produced. Morrell and Marks' hypothesis (B., 1929, 861) that yellowing is due to OH compounds could not be confirmed by determinations of the halogen-substitution val. Films exposed for 366 hr. to ultra-violet light yellowed less (? not at all) in subsequent darkness than those pre-exposed to diffuse light. E. LEWKOWITSCH.

**Composition of kapok seed.** E. P. GRIFFING and G. L. ALSBERG (Ind. Eng. Chem., 1931, 23, 908—909).—Javan kapok seed (*Ceiba pentandra*, L., carefully hulled by hand) yielded 55% of meats containing 5.7% H<sub>2</sub>O. Very little or no gossypol is present. The pale



viscous oil extracted by light petroleum had:  $d_{25}^{25}$  0.9225,  $n_D^{25}$  1.4691, sap. val. 191.6, I val. (Hanus) 94.1, unsaponifiable matter 0.94%, acid val. 9.65, saturated acids 17.15%, unsaturated acids 76.32%. The distribution of N is much as in cottonseed (cf. B., 1925, 778), but kapok seed contains more N in a form non-extractable by NaCl solution or dil. alkali. It contains principally globulin(s) and a glutelin, but no prolamine, and probably no albumin. E. LEWKOWITSCH.

**Fluoroscopic analysis and its application to Portuguese [fatty] oils.** D. A. P. FORJAZ (Rev. Chim. Pura Appl., 1930, [iii], 5, 29–34).—The fluorescence of almond oil exposed to light from the Wood lamp is an intense blue, and those of cottonseed oil and soya-bean oil are a paler blue. The fluoroscopic method may be employed to detect adulteration of olive oil, which when pure yields a yellow, orange, or red coloration; a table is given of the colours obtained with olive oils from various sources. H. F. GILLBE.

**Ageing of materials.**—See I. Detergents.—See VII.  $\text{Me}_3\text{BO}_3$  in analysis.—See XIII. Salmon oil and vitamins.—See XIX.

#### PATENTS.

**Treating liquids.**—See I. Wax compositions.—See II. Floor coverings.—See IX. Artificial cream. Bacon fat.—See XIX.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Paint thinners. I. Effect of different thinners on the durability of house paints in outdoor exposure tests.** F. L. BROWNE (Ind. Eng. Chem., 1931, 23, 868–874).—The various theories of the relative values of turpentine and other thinners are critically discussed. Comprehensive exposure tests were carried out at 5 different stations on two series of straight linseed-oil paints (based on white lead and mixed white lead-ZnO-inert material, respectively) applied to longleaf pine boards, using each of the following 10 thinners: turpentine (4 kinds), mineral spirits (from 4 types of crude petroleum), V.M. and P. naphtha, and coal-tar naphtha. It is shown that durability is virtually independent of variation of thinner inside the normal range of such products. Turpentine, however, tends to improve durability in proportion to the amount of residue it leaves permanently in the coating, and the possibilities of oxidised turpentine as a paint ingredient are indicated. S. S. WOOLF.

**Effect of resin in longleaf pine on the durability of house paints.** F. L. BROWNE and C. E. HRUBESKY (Ind. Eng. Chem., 1931, 23, 874–877).—In the tests on thinners described in the previous abstract, it was found that the behaviour of paint coatings was independent of the resin content of the longleaf pine boards (estimated by visual inspection and by ether extraction, these two methods showing reasonable concordance). High density of the wood and wide growth rings were, however, found to induce early failure of paint films by flaking. S. S. WOOLF.

**Use of methyl borate in analysis of organic paint and varnish components.** E. J. FISCHER (Farbe u. Lack, 1931, 36, 401–402).—The qual.

solubilities of 60 resins, waxes, oils, bitumens, etc. in cold and in hot  $\text{Me}_3\text{BO}_3$  are tabulated. The use of these solubility relationships in the detection of stearine in beeswax, separation of oils from waxes, resins from bitumens, etc. is indicated. S. S. WOOLF.

**Preparation of light-resistant lithopones, and of lithopones not yellowing in the dark.** V. N. SCHULTZ and A. V. TICHONOV (J. Chem. Ind. Russ., 1931, 8, 704–720, 811–824).—The following conditions should be observed in the prep. of lithopone which does not change its colour in the light or in the dark. All the Zn present in solution should be precipitated as ZnS by BaS. Cl-free  $\text{ZnSO}_4$  and BaS should be used if possible; if not, the ppt. should be washed until it contains 0.1% of its dry wt. of Cl. Should it not be possible by washing to reduce the Cl content to this value, a slight excess of BaS should be added to the ppt. The lithopone is heated during 1 hr. at 700–750°, in an inert or feebly reducing atm., in order to augment its covering power. R. TRUSZKOWSKI.

**Optical and X-ray investigations on pigments.** H. WAGNER (Z. angew. Chem., 1931, 44, 665–667).—When ultramarine, dispersed in a medium of lower refractive index, is observed with semi-reflected light it appears reddish, but if the medium has a higher  $n$  than the pigment the latter appears greenish; similar effects are obtained with other pigments which form colloidal or semi-colloidal dispersions in the medium, especially with pigments exhibiting a bronze lustre. This lustre in the case of Milori-blue can be reduced by using a medium of higher  $n$ , and intensified by using a medium of lower  $n$ . X-Ray examination of precipitated solid solutions of  $\text{PbSO}_4$  and  $\text{PbCrO}_4$  show that both the pure salts are originally precipitated in the rhombic form, but that the latter rapidly changes to the monoclinic form; mixed crystals take the form of the predominating constituent, the form of the equimol. mixture depending on the conditions of precipitation. For the manufacture of paints the rhombic form is the more suitable as it more readily disperses in the oil, requires less oil, dries more rapidly, and has a higher colouring and covering power; on the other hand, the colour is less stable, being rapidly reddened by exposure to light owing to conversion into the monoclinic form, which has a coarser grain size. No reaction occurs between chrome-yellows and -reds and linseed oil. Chrome-red has a monoclinic structure; in its prep. an ill-defined hydrated salt is first formed, which changes on keeping into the well-defined  $\text{PbCrO}_4 \cdot \text{PbO}$  with accompanying coarse grain growth, but without change of crystal structure. A. R. POWELL.

**Effect of heat and light on nitrocellulose [lacquer] films.** W. E. GLOOR (Ind. Eng. Chem., 1931, 23, 980–982).—The principal effect of ultra-violet light on nitrocellulose film is a pronounced local surface denitration (and degradation). The resulting film is brittle, the viscosity and moisture-resistance are lowered, and the development of acidity (methyl-violet test) is accelerated. When the film is kept at 70° general denitration and degradation occur; softening and a gradual lowering of viscosity and moisture-resistance become apparent. H. BURTON.

**Prevention of gelling of bronze lacquers.** C. BOGIN, V. KELLY, and W. MARONEY (Ind. Eng. Chem., 1931, 23, 982—985).—The gelation of nitrocellulose lacquers containing Cu bronze is prevented or greatly delayed by the use of 0.25—1.0% of boric, citric, malic, tartaric, or lactic acid, or some of their salts.  $H_3BO_3$  is recommended for general purposes on account of its low price and min. tarnishing or darkening effect; brass and steel surfaces are not tarnished thereby. The proportion of the inhibitor required depends on the grade and concentration of the bronze powder, the purity and age of the nitrocellulose, and on the presence of resins; the effect of the solvent is small, but large proportions of EtOH are definitely injurious. Gelation occurs more readily with dark- than with light-coloured bronzes, and Al bronzes do not cause gelation readily. No gelation was observed with Cu and Al bronzes in cellulose acetate solutions. E. S. HEDGES.

**Absorption of ultra-violet light by lacquer films.** D. C. DUNCAN, D. R. WIGGAM, and W. P. DAVEY (Ind. Eng. Chem., 1931, 23, 904—906).—The coeffs. of absorption for various wave-lengths of ultra-violet light of lacquer films were found to be unaffected by variations in the N content of the nitrocellulose and type of plasticiser used. Measurement of the absorption of individual lacquer ingredients showed that, within the range of wave-lengths found in sunlight, the effect of ultra-violet light is not due to any one wave-length, but increases rapidly as the wave-length is decreased beyond a threshold limit. Of all the ingredients of common lacquers, ester gum is the one most affected by ultra-violet light. S. S. WOOLF.

**Dry distillation of balsam of Peru.** J. DUPONT and J. J. GUERLAIN (Compt. rend., 1931, 193, 342—343; cf. B., 1931, 502).—The same results were obtained if the tolu balsam was first distilled in vac. and the residual resin then dry-distilled. Treated in this way balsam of Peru (from *Myroxylon balsamum* var.  $\beta$ -*Pereira*) yielded about 35% of resin, from which on dry distillation were obtained PhMe, styrene, and higher-boiling products insol. in aq. NaOH, together with guaiacol, 3-methoxy-*p*-cresol, and 2-methoxy-*p*-ethylphenol. C. A. SILBERRAD.

**Grading aggregates. Ageing of materials.**—See I. Value of pipe coatings.—See X. Yellowing of trilinolenic glyceride.—See XII. *Podophyllum* resin.—See XX. Preserving metal surfaces for sewage.—See XXIII.

## PATENTS.

**Manufacture of turpentine, pine oil, and rosin from woody materials rich in oleoresin.** F. C. PALAZZO (U.S.P. 1,792,392, 10.2.31. Appl., 1.8.27. It., 15.6.27).—Coniferous wood, reduced to chips of size and shape permitting subsequent use in manufacture of wood pulp, is steam-distilled until 75% of the turpentine is volatilised (and recovered in known manner), when a dil. alkaline solution, e.g., 1.5—2.5% aq. NaOH, is added to the residue and steam-distillation is continued, a further amount of turpentine and pine oil being recovered. The solution of rosin soap is acidified with dil.  $H_2SO_4$ , and the separated rosin purified by extraction with a solvent, e.g., petrol or pinene. S. S. WOOLF.

**Production of coating compositions for protecting surfaces against rust, decomposition, heat, fire, etc.** VICTORIA VEGYÉSZETI MŰVEK R.T. (B.P. 355,266, 20.5.30. Hung., 29.1.30).—Bauxite, particularly "Gantite"—a soft porous brown type of the mineral—is heated above 600°, but below its m.p., and is finely ground, sifted or washed, and mixed with a binder, e.g., linseed oil,  $Na_2SiO_3$ . S. S. WOOLF.

**Manufacture of [quick-drying] coating compositions and their application.** E. I. DU PONT DE NEMOURS & Co. (B.P. 355,271, 14.2.30. U.S., 12.6.29).—Such compositions, substantially free from cellulose derivatives, comprise a pigment combination of low oil absorption (100 pts.) and a binder which has a tendency to polymerisation, either of the polyhydric alcohol-polybasic acid type (15—60 pts.) or of the oil type (40—70 pts.), adapted to dry in 3 hr. at 71° even in thick films, e.g., 0.003 in. Several typical examples of undercoats are given. S. S. WOOLF.

**Coating materials [from rubber].** NEW YORK HAMBURGER GUMMI WAAREN COMPAGNIE (B.P. 355,547, 19.9.30. Ger., 26.6.30).—Hard substances, e.g., quartz meal or carborundum, are added to solutions of chlorinated rubber in benzene etc., to increase the resistance to shocks and blows of such coatings. H. ROYAL-DAWSON.

**Manufacture of [linoleum-type] floor coverings.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 354,324, 31.1.30).—Ground cork, wood-flour, pigments, etc. are mixed with a resinous product formed by heating a mixture of a polyhydric alcohol (glycerin, ethylene glycol), one or more oil acids (i.e., monobasic acids above  $C_{10}$ , such as those from linseed or tung oil) or glyceryl esters thereof, or a mixture of these with oil acids and a polybasic acid (e.g., phthalic acid) or its anhydride. The oil acids and/or glyceryl esters should not constitute >50% of the resinous product. The prepared mixture may be calendered on to a fibrous backing. C. A. KING.

**Treatment of pigments.** COMP. LORRAINE DE CHARBONS POUR L'ÉLECTRICITÉ (B.P. 355,503, 14.8.30. Fr., 7.9.29).—The capability of finely-grained flocculent pigments for subsequent dispersion is increased by mixing and heating them with an aq. solution of an amphoteric colloid that has been pretreated with alkali or acid according to the nature of the pigment; e.g., 1% of a gelatin brought to  $p_H$  8—9 by addition of aq.  $NH_3$  is used with C blacks. If desired, the pigment so treated is finally triturated by rolling, calendering, and/or grinding, to increase its apparent density. S. S. WOOLF.

**Treatment of rosin.** D. C. BUTTS, Assr. to HERCULES POWDER Co. (U.S.P. 1,791,658, 10.2.31. Appl., 16.10.26).—Rosin, especially wood rosin, is heated in a chemically inert atm., e.g.,  $CO_2$ , at 280—350° for about  $\frac{1}{2}$  hr., without substantial distillation and decomp., to give pale rosins of higher sp. rotation and less tendency to crystallise than the original. S. S. WOOLF.

**Synthetic resins and articles made therefrom.** INDIA-RUBBER, GUTTA-PERCHA, and TELEGRAPH WORKS Co., LTD., and F. J. CROSLY (B.P. 355,341, 23.5.30).—

Ground rubber, partly or fully vulcanised, *e.g.*, ebonite dust, is incorporated with about an equal wt. of a synthetic resin of the  $\text{PhOH}\cdot\text{CH}_2\text{O}$  type, as a filler, other known fillers, *e.g.*, wood flour, being also incorporated if desired. S. S. WOOLF.

**Preparation of transparent artificial resins by condensing phenols and formaldehyde.** G. PECHIN (B.P. 355,634, 15.1.31).—A mixture of  $\text{AcOH}$ , camphor, glycerin, and  $\text{HCl}$  is added to the resinous mass after separation of the water formed. H. ROYAL-DAWSON.

**Manufacture of porous [perforated] bodies from ebonite or other like plastic material.** SOC. ITAL. PIRELLI (B.P. 355,311, 22.4.30. Italy, 22.4.29).

**Laminated materials.**—See VIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Chemistry of [rubber] latex.** IV. Sp. gr. and crêpe-rubber yield of *Hevea* latex with reference to the influence of the non-rubber constituents. P. SCHOLZ and K. KLOTZ (Kautschuk, 1931, 7, 142—145; cf. B., 1931, 769).—Latexes of *Hevea*, *Palaquium* (gutta), *Ficus*, and *Euphorbia* have been examined as to their sp. gr. (determined gravimetrically), total solids, yield of crêpe rubber, and proportion of constituents other than those comprised in the crêped and dried coagulum. Numerous experiments with *Hevea* latex of various concentrations suggest that the sp. gr. follows the simple mixing law with 1.023 and 0.901 as the sp. gr. of the serum and rubber, respectively. The irregularity in the results, however, is such that individual values for rubber content (calc. from the sp. gr. of the latex) diverge up to 3% from the mean line. An increase in the proportion of non-rubber constituents in latex of equal crêpe-rubber yield raises the sp. gr. of the latex. D. F. TWISS.

**Röntgenographic investigation of the gutta-percha hydrocarbon.** E. A. HAUSER and G. VON SUSICH (Kautschuk, 1931, 7, 120—122, 125—127, 145—149).—Apparatus is described for the röntgenographic examination of the progressive  $\alpha$ - $\beta$  transformation and "melting" of gutta-percha and, in the same connexion, a chamber is devised for the extension of samples at const. temp. The transformation temp. between the  $\alpha$  and  $\beta$  cryst. forms of gutta is between 65° and 75°; the natural impurities in raw gutta-percha or balata do not greatly reduce the transformation temp.; the change, however, is slow at 65° but rapid at 75°. The "röntgenographic m.p." of the  $\alpha$  and  $\beta$  forms of gutta, at which they pass into an amorphous condition, are near 65° and 56°, respectively. These changes are quite distinct from the  $\alpha$ - $\beta$  transformation, which is a monotropic conversion from the  $\alpha$  form to the  $\beta$  form, these forms being merely polymorphous and not distinct chemical isomerides. The effect of stretching on gutta-percha resembles that with metals rather than that with smoked sheet rubber in that it involves orientation of existent crystallites, whereas with unfrozen smoked sheet rubber crystallisation results only on stretching. D. F. TWISS.

**Microporous rubber, its production, characteristics, and applications.** H. BECKMANN (Kaut-

schuk, 1931, 7, 149—155).—By converting rubber latex into a continuous, uniform, elastic coagulum, a wet mass of reticular structure is obtained which contracts to a solid mass on drying. If latex compounded with S is so treated and the resulting gel is vulcanised in wet steam or under  $\text{H}_2\text{O}$ , the reticular mass on drying yields a microporous soft or hard rubber. The porosity of the product may amount to 55—60% of the total vol. with ordinary latex, but using latex diluted to 10—20% rubber content the porosity may attain 80%. Microporous vulcanite is of light brown colour; it assumes the dark colour of ordinary vulcanite if consolidated by pressure, but regains its original condition if immersed in hot  $\text{H}_2\text{O}$ . It is resistant to oil, has sp. thermal conductivity  $1.9 \times 10^{-3}$ , and, if immersed in an electrolyte, offers little resistance to the electric current. Amongst possible applications is the production of diaphragms for accumulators and electrolytic purposes, filtering material, and wicks for oil lubrication. D. F. TWISS.

**Nerve of rubber.** J. BEHRE (Kautschuk, 1931, 7, 161—165).—Using solutions of 0.5 g. of rubber in 100 c.c. of  $\text{C}_6\text{H}_6$ , it is found that the method of determining the rate of ascent into a strip of filter paper (cf. B., 1926, 374) gives results in agreement with those given by the stalagmometric or drop method; both methods afford an index to the surface tension of the solutions, but the former is the more convenient. Berggren's method for determining the surface tension of solid materials gives results with rubber parallel to those of the other two methods, so that the capillary method is regarded as providing an index to the nerve of the rubber. Comparative experiments with the capillary test, using solutions, and with tensile tests on S-vulcanised products from the corresponding rubber indicate a const. relationship between the tensile strength of the vulcanised rubber and the nerve as measured by the capillary method. At equal values of nerve, light brown crêpe and sprayed rubbers exhibit higher tensile strength than "first latex crêpe" and smoked sheet. D. F. TWISS.

**Behaviour of rubber towards textile fabrics and threads.** W. ESCH (Kautschuk, 1931, 7, 155—156).—Dipping of textile fabric, *e.g.*, tyre cords, in rubber latex results in practically no penetration of rubber into the threads; in this respect rubber solutions are the more effective, but the advantage is not very great. Rubbing by mere mechanical "frictioning" with a suitably compounded rubber mixing is capable of giving satisfactory results in subsequent service (cf. B., 1931, 643). D. F. TWISS.

**Value of rubber hydrocarbon in reclaimed rubber.** C. W. SANDERSON (Ind. Eng. Chem., 1931, 23, 989—991).—An extension and confirmation (by road tests) of the work of Vogt (B., 1928, 238).

H. BURTON.  
**Grading aggregates. Ageing of materials.**—See I.

#### PATENTS.

**Manufacture of vulcanised rubber and the like.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 355,425, 27.6.30).—Compounds of the type  $\text{HO}\cdot\text{R}\cdot\text{X}\cdot\text{R}'\cdot\text{Y}$ , where R is a

bivalent aromatic hydrocarbon residue of the  $C_6H_6$  or  $C_{10}H_8$  series,  $R'$  is a similar residue of the  $C_6H_6$  series,  $X$  is a bivalent aliphatic hydrocarbon residue, and  $Y$  is  $H$  or  $OH$ , exert an antioxidant effect in rubber without disturbing the rate of vulcanisation or leading to substantial discoloration in sunlight. Typical substances are *p*-hydroxydiphenylmethane, and *pp'*-dihydroxy- $\alpha$ -diphenylethane.

D. F. TWISS.

#### Manufacture of rubber goods of high quality.

I. G. FARBENIND. A.-G. (B.P. 355,416, 17.6.30. Ger., 17.6.29. Cf. B.P. 315,916; B., 1929, 829).—Finely-divided C (soot) is incorporated in mixtures containing artificial and natural rubber, comprising the artificial and natural varieties in min. proportions of 50% and 10%, respectively.

D. F. TWISS.

**Manufacture of goods of or containing rubber or the like.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B. P. 355,287, 21.5.30).—The serum-enriched "skim" obtained in the centrifugal concentration of latex is concentrated and used for improving manufactured goods by mixing with conc. latex obtained by centrifuging, by adding to moulding powders containing rubber, or by applying it directly to or in admixture with rubber compositions in aq. dispersion or not.

D. F. TWISS.

**Coatings from rubber. Synthetic resins.**—See XIII.

### XV.—LEATHER; GLUE.

**Sampling tanning materials and extracts.** A. TURNBULL (J. Soc. Leather Trades' Chem., 1931, 15, 438—440).—Details are given for sampling liquid, solid, and pasty extracts, respectively, and also for sumac. Sampling tools are described and illustrated.

D. WOODROFFE.

**Precipitation of [vegetable] tannin by hexamethylenetetramine in aqueous solutions.** A. T. HOUGH (J. Soc. Leather Trades' Chem., 1931, 15, 406—411).—Max. precipitation was obtained at  $p_H$  5.6, but no ppt. was given in dil. solutions. The amount of ppt. was increased by the presence of metallic salts, and the following reagent is proposed: 10 c.c. each of 30% solutions of  $Zn(OAc)_2$ ,  $NH_4OAc$ , and hexamethylenetetramine, and 1 c.c. of glacial  $AcOH$ . With this reagent 1 pt. of tannin in 200,000 pts. of  $H_2O$  yields a ppt. visible to the naked eye, and 1 pt. of tannin in  $10^6$  pts. of  $H_2O$  a turbidity detectable by the Tyndall effect.

D. WOODROFFE.

**Preparation of furfuraldehyde from extracted oak bark from tanneries and other pentosan-containing substances.** D. I. MURLIS (J. Chem. Ind. Russ., 1931, 8, 697—703).—Extracted tannery oak bark waste or other pentosan-containing substance (maize cobs, sunflower-seed husks, etc.) is autoclaved for  $\frac{1}{2}$  hr. at  $158$ — $160^\circ/6$ — $7$  atm. with 5—7 pts. of 1%  $H_2SO_4$  and 4%  $NaCl$  solution, and furfuraldehyde is immediately distilled off (6—7 atm.). The distillate, containing also  $AcOH$ ,  $MeOH$ , and  $HCl$ , is neutralised with  $Ca(OH)_2$ , and the constituents are separated by fractionation. The process is completed within 3.3—5 hr., the yields of furfuraldehyde being 9.4—10.2%

on the wt. of extracted oak or sunflower husks, or 18% on the wt. of corn-cobs taken; 3% of  $AcOH$  and 1% of  $MeOH$  are also recovered. Using  $H_2O$  in place of the acid mixture in the above process, 6.5—7.0% yields of furfuraldehyde are obtained. The residue in the autoclave is, as a result of its low acidity, an excellent fuel.

R. TRUSZKOWSKI.

**Furfural[dehyde] number of tanning extracts and their mixtures with sulphite-cellulose extract.** V. NEMEC (J. Soc. Leather Trades' Chem., 1931, 15, 440—443).—Lauffmann's method (B., 1919, 379 A) of determining this value has been modified to give more concordant results. A higher figure was obtained for both sulphited and unsulphited quebracho extracts than for mangrove extract, so that the furfuraldehyde no. of quebracho extracts is lowered by admixture with mangrove. The furfuraldehyde no. was lowered proportionately to the amount of sulphite-cellulose extract added to a tanning extract.

D. WOODROFFE.

**Fixation of [vegetable] tannin in leather and modification of its characteristics.** A. T. HOUGH (J. Soc. Leather Trades' Chem., 1931, 15, 411—415).—The unfixed tannin in a vegetable-tanned leather can be fixed by treating the leather with solutions of hexamethylenetetramine. The analytical figures for such treated leathers are thereby altered: the  $H_2O$ -sol. matter is considerably reduced, the N figure increased, and the apparent degree of tannage is diminished.

D. WOODROFFE.

**Discoloration and staining of leather by copper impurities in vegetable tan liquors.** M. P. BALFE and H. PHILLIPS (J. Soc. Leather Trades' Chem., 1931, 15, 444—454).—Greenish stains on vegetable-tanned leather may be caused by the reaction of  $Cu$  in the suspender liquors with  $H_2S$  in the delimed pelts.  $Cu$  is an impurity more frequently found in chestnut extracts. The grain of the delimed pelts should be freed from  $H_2S$  by aeration of the deliming liquors or by oxidation of the  $H_2S$  with  $H_2O_2$ . Methods are suggested for the determination of  $H_2S$  in the leather, delimed pelt, deliming liquors, and suspender liquors.

D. WOODROFFE.

**Density of leather.** U. J. THUAU and A. G. DE BUDA (J. Soc. Leather Trades' Chem., 1931, 15, 415—426).—A simple densimeter is described for determining the real density of leather. The apparent density of a leather was found to exceed that of any of the four layers into which it was afterwards cut.

D. WOODROFFE.

$Na_4Fe(CN)_6$ .—See VII. Egg-yolk.—See XIX. Sewage and industrial wastes.—See XXIII.

### XVI.—AGRICULTURE.

**Fertile soil.** VI. A. J. J. VANDE VELDE, A. VERBELEN, and L. DE KOKER (Natuurwetensch. Tijds., 1931, 13, 239—246; cf. B., 1931, 556).—The influence of  $CaCO_3$ ,  $(NH_4)_2SO_4$ ,  $KNO_3$ ,  $CaCN_2$ , and superphosphates on the  $p_H$  and adsorptive capacity for different dyes of a variety of soils has been examined. The salts have no influence on the adsorption of methylene-blue, but that of methyl-violet is depressed by the  $Ca$  salts and increased by  $(NH_4)_2SO_4$  and  $KNO_3$ ; that of methylene-green is

increased considerably by  $\text{CaCO}_3$  and  $\text{CaCN}_2$ . Addition of K halides and variations of the humidity do not alter the adsorptive powers of the soils. H. F. GILLBE.

**Causes of changes in the adsorptive capacity and dispersion of soils.** M. A. VINOKUROV and V. I. KARABIZKI (Arb. sibirisch Inst. Land- u. Forstw., 1929, 13, 27—51; Bied. Zentr., 1931, 60, 293—294).—The adsorptive capacity of black earths, podsols, and saline soils is not of const. magnitude. Variations are induced by soil reaction and other factors. In columnar saline soils the  $p_H$  is influenced by the moisture content and the proportion of dispersible humate and zeolite in the adsorptive complex. In black earths and podsols the  $p_H$  varies with the concentration of the soil solution. In cultivated black earths and possibly in all cultivated soils there is an inverse relationship between the  $p_H$  of the soil solution and the activity of the nitrification process. Dispersion varies throughout the vegetative season, being least in summer and greatest in spring and autumn. The degree of dispersion of soils is closely related to the moisture content, concentration of the soil solution, reaction, and adsorptive capacity.

A. G. POLLARD.

**Neubauer's chemico-physiological method for determining assimilable phosphoric acid and potash in soils.** I. C. ANTONIANI and M. NICOLINI (Giorn. Chim. Ind. Appl., 1931, 13, 369—372).—Rye seedlings are grown according to Neubauer's method. In the ash of the plants  $\text{K}_2\text{O}$  and  $\text{P}_2\text{O}_5$  are determined by the  $\text{K}_2\text{PtCl}_6$  method and the strychnine phosphomolybdate method respectively. The average differences in the duplicates were for  $\text{P}_2\text{O}_5$ ,  $\pm 0.26$  (161 duplicate determinations); for  $\text{K}_2\text{O}$ ,  $\pm 0.65$  (151 duplicates).

O. F. LUBATTI.

**Retention of phosphoric acid by soil organic matter.** G. BALANESCO (Ann. Sci. Agron., 1930, 47, [3]; Proc. Internat. Soc. Soil Sci., 1931, 4, 59).—Purified humus does not absorb  $\text{PO}_4'''$  from phosphate solutions, whatever their  $p_H$ . The apparent fixation of  $\text{PO}_4'''$  by humus is due to the action of adsorbed cations, notably  $\text{Fe}'''$ . The fixation of  $\text{PO}_4'''$  by humus containing Ca occurs only when conditions permit the existence of a phosphate of Ca in the solid phase.

A. G. POLLARD.

**Transition of water-soluble phosphates in soils.** M. GERLACH (Superphosphat, 1931, 7, 130—133).—The interaction of  $\text{H}_2\text{O}$ -sol. phosphates and soil is discussed, together with the mechanism of the P assimilation of plants.

A. G. POLLARD.

**Distribution of assimilable phosphate in soils resulting from the action of growth factors.** L. DWORAK (Kísérlet. Közl., 1930, 33, 336; Bied. Zentr., 1931, 60, 294—295).—The distribution in soils of artificially applied phosphates varies with soil type. In a sandy soil containing  $\text{CaCO}_3$ , but deficient in P, the greatest accumulation of easily-sol. P occurred in the horizon from 10 to 30 cm. deep. The action of various growth factors does not result in a fixation of P in these soils. In a CaO-deficient soil containing adequate P, fertilisers did not penetrate deeply, although oats assimilated most P from subsurface layers.

A. G. POLLARD.

**Course of the nutrient intake [of plants] and fertiliser requirements.** T. REMY and E. DEICHMANN (Ernähr. Pflanze, 1931, 27, 301—317).—The effects of the nature, quantity, and time of application of fertilisers on the rate of nutrient intake by a number of crops in various stages of growth, and on the distribution of mineral matter in the plants, is recorded.

A. G. POLLARD.

**Regularity of plant growth, with special reference to nutritional problems.** A. RIPPEL (Ernähr. Pflanze, 1931, 27, 251—253).—The theoretical basis of Mitscherlich's growth curves is unsound, although the method of determining fertiliser requirements gives valuable practical information. Growth curves obtained with a series of increasing applications of N reach points of max. yield at periods which increase with the N dosage. Results based on these curves depend therefore on the length of the experimental period and on other soil conditions.

A. G. POLLARD.

**"Effect law" of growth factors.** E. A. MITSCHERLICH (Ernähr. Pflanze, 1931, 27, 277—286).—A discussion of the author's work and a comparison with other methods for determining the nutrient condition of soils are given.

A. G. POLLARD.

**Effect of gypsum on the assimilability of the phosphoric acid of phosphorites.** A. G. MICHALOVSKI (Düng. u. Ernte, 1930, 99—102; Bied. Zentr., 1931, 60, 318—319).—Supplementary manuring with S or gypsum accelerated the transition of the P of phosphorites into an easily-sol. form.

A. G. POLLARD.

**Effect of peat on the utilisation of the phosphoric acid of phosphorites.** S. V. LOGVINOV (Düng. u. Ernte, 1929, 211—216; Bied. Zentr., 1931, 60, 319—320).—Applications of acid sphagnum peat favoured the conversion of phosphorites into  $\text{CaHPO}_4$  or  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  in soils. Podsolised soils have a similar action. All types of peat are not equally effective.

**Nitrogen economy of forest soils where litter is removed.** A. NĚMEC (Forst. Centralbl., 1931, 53, 49—67, 147—156; Bied. Zentr., 1931, 60, 296).—Continual raking off of the litter lowers the total N and nitrate content of the humus and upper mineral layers of forest soils. The height and diam. of the trees and the N content of the needles are correspondingly reduced.

A. G. POLLARD.

**Conversion of urea in soils.** J. BORDAS and G. MATHIEU (Ann. Sci. Agron., 1930, 17, 711—727; Bied. Zentr., 1931, 60, 300—301).—The decomp. of urea in soil is a biological process and depends on the temp. and the amount of org. matter present. Addition to soil of  $(\text{NH}_4)_2\text{SO}_4$  or urea and urease accelerates the decomp. of org. matter. In soils containing nitrate and/or nitrite a slow decomp. of urea may result in losses of N. Urea in soil is not completely converted into  $(\text{NH}_4)_2\text{CO}_3$ , other org. N compounds being formed. Nitrification of urea is complete in 5—6 weeks.

A. G. POLLARD.

**Use of industrial wastes as fertilisers.** V. P. MOSSOLOV (Düng. u. Ernte, 1930, 182—186; Bied. Zentr., 1931, 60, 320).—Field trials are quoted to show the value of various classes of slaughterhouse wastes as fertilisers.

A. G. POLLARD.

**Twenty years of rotation and manuring experiments.** G. STEWART and D. W. PITTMAN (Utah Agric. Exp. Sta. Bull., 1931, No. 228, 31 pp.).—Field trials are recorded in which soil fertility was maintained at a high level by the efficient application of farmyard manure only, and by suitable systems of rotation.

A. G. POLLARD.

**Christensen and Jensen's work on the effects of manuring on soils in continuous fertiliser trials.** OPITZ (Superphosphat, 1931, 7, 136—140).—Results from continuous manurial trials in various parts of the world are examined. Prolonged use of cattle manure leads to a small but definite increase in the humus content of soils. The leaching out of N from such soils is considerable. With regular and excessive applications of superphosphate there is an accumulation of P in soil, largely in a form easily assimilable by plants. No general relationships exist between the K content of soils and the amounts of K supplied in fertilisers.

A. G. POLLARD.

**Composition of the adsorptive complex of arid soils as a basis for determining salt injury, and potash manuring in the arid tropics and subtropics.** P. VAGELER (Ernähr. Pflanze, 1930, 26, 393—394, 412—414, 440—442; Bied. Zentr., 1931, 60, 291—292).—Salt injury cannot be associated with any particular base or salt content of soils, but is closely related to the ratio of alkali present to the total adsorptive capacity. The ease of removal of salinity can be determined from the differential quotients of the capillarity equation. Sudan soils contain relatively small proportions of assimilable K and respond to heavy dressings of K fertilisers. Small applications are non-effective.

A. G. POLLARD.

**Physico-chemical changes in arable soils caused by applications of dung.** P. A. VLASSJUK (Arb. Versuchs-Stat. Uman, 1930, 10, 93; Bied. Zentr., 1931, 60, 299—300).—The H<sub>2</sub>O-supplying power of soils was increased in dry seasons by applications of stall manure. In wet seasons no effect was observed. No relationship exists between the improved H<sub>2</sub>O condition and the amount of manure applied. The nitrate and H<sub>2</sub>O-sol. P contents of soils in wet seasons were increased by manuring and the  $p_H$  tended to decrease. During the decomp. of the manure there was an increase in the proportion of aggregate soil particles of more than 7 mm. diam. at the expense of those of 25  $\mu$ —1 mm. diam., without material change in the finest particles. This aggregating effect was smaller than that produced by liming. The apparent sp. gr. of soil to a depth of 50—70 cm. increased with the amount of manure added, but the ultimate mechanical analysis of the soil was unchanged. Manuring reduced the proportion of adsorbed Na and K and increased that of Ca and Mg.

A. G. POLLARD.

**Manuring of moors and highlands.** M. POPP (Ernähr. Pflanze, 1931, 27, 262—265).—In field trials with potatoes, high yields and high starch contents were obtained only by the use of P and K fertilisers. The Cl content of the potatoes increased with the amount of Cl applied in the potash fertilisers. The PO<sub>4</sub><sup>'''</sup> content was increased by the use of potash. The general use of fertilisers did not impair the keeping quality of the tubers, nor influence their value as seed. The ripening

of the potatoes as judged by their amide content was accelerated by applications of K.

A. G. POLLARD.

**Manuring of meadows.** E. KLAPP (Ernähr. Pflanze, 1931, 15, 321—329).—The effect of manuring on the yield and composition of meadow herbage in Germany is examined and discussed in comparison with average results from other countries.

A. G. POLLARD.

**Principles of green manuring, and their application in Ceylon.** I. A. W. R. JOACHIM (Trop. Agric., 1931, 77, 4—32).—The significance of green manuring in the agriculture of Ceylon is discussed. The chemical composition of typical green crops together with their comparative rates of decomp. in soil are recorded. By efficient green manuring satisfactory N and C contents are maintained in soils and their physical condition is improved.

A. G. POLLARD.

**Is potash manuring necessary?** F. MÜNTER (Ernähr. Pflanze, 1931, 15, 334—336).—Numerous field trials are quoted showing the crop increases obtained by the use of potash fertilisers even when applied in addition to stall manure.

A. G. POLLARD.

**Influence of manuring on the quality of cereals.** EICHINGER (Ernähr. Pflanze, 1931, 15, 329—332).—Omission of K from fertilisers for rye lowered the hectolitre-wt. of the grain. No significant difference is apparent between the effects of NaNO<sub>3</sub>, Ca(NO<sub>3</sub>)<sub>2</sub>, and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> on the hectolitre-wt. K-deficient fertilisers reduce the proportion of larger grains (by sieving) in the crop.

A. G. POLLARD.

**Phosphate manuring and storage of cereals.** M. KLEIN (Superphosphat, 1931, 7, 150—151).—The effect of P manures in increasing the stiffness of straw and storage quality of cereals is discussed.

A. G. POLLARD.

**Manuring and the quality of potatoes.** MEYER-BAHLBURG (Superphosphat, 1931, 7, 151—153).—Recommendations for the prevention of scab in potatoes include top dressings with acid fertilisers at the period of active infection and the liberal use of superphosphate to induce the production of firm-skinned tubers.

A. G. POLLARD.

**Effect of nutrition on cell size in potato tubers.** G. BREDEMANN and W. SCHULTZE (Ernähr. Pflanze, 1931, 27, 293—295).—Applications of potash fertilisers whether in spring or autumn produced tubers with cells of larger diam. No relationship exists between cell size and that of starch grains. Applications of N increased the mean diam. of the cells by reducing the proportion of smaller cells, and, simultaneously, decreased the mean diam. of starch grains by increasing the proportion of small grains.

A. G. POLLARD.

**Influence of carbon dioxide and of oxygen on the sprouting and value of potato tubers.** H. BRAUN (Arb. biol. Reichsanst. Land- u. Forstwirts., 1931, 19, 17—93).—An artificially increased CO<sub>2</sub> content of the atm. in storage chambers favoured the sprouting of potato tubers, but in subsequent field trials the crop yields were either unaffected or reduced by this treatment. The effect varied with the temp. of storage and the species of potato. Reduction of the O<sub>2</sub> of the storage atm. reduced the vitality of the tubers. Under

similar conditions an increase of  $\text{CO}_2$  favoured, and a reduction of  $\text{O}_2$  retarded, the growth of fungal parasites. The bearing of this on the result of field experiments is discussed.

A. G. POLLARD.

**Quality and composition of potatoes, with special reference to the ash and potash contents of the tubers.** GEYER (Ernähr. Pflanze, 1931, 27, 286—293).—Potatoes grown on light soils have higher ash and K contents than those grown on heavy soils, although the relative magnitude of these values remains practically the same for the same varieties grown on different soils. Varieties with high ash contents usually have high K contents. There is no relationship between the condition and health of the plants and their ash and K contents.

A. G. POLLARD.

**Rate of nitrogen intake from ammonium salts and nitrates by agricultural crop plants.** K. SCHMID (Diss., L. H., Hohenheim, 1930; Bied. Zentr., 1931, 60, 309).—Maize, barley, and wheat plants were able to withdraw  $\text{NH}_4^+$  and  $\text{NO}_3^-$  more easily from very dil. solutions (0.0005N), the  $\text{NH}_4^+$  being the more rapidly absorbed. The effects, on the rate of assimilation, of the concentration and reaction of the nutrient, temp., light, and age of the plant are examined.

A. G. POLLARD.

**Water-supplying power of the soil under different species of grass and with different rates of water application.** F. A. WELTON and J. D. WILSON (Plant Physiol., 1931, 6, 485—493).—The  $\text{H}_2\text{O}$ -supplying power of soil under grass, as determined by the porous-porcelain soil point, varies with the type of grass. A narrow-leaved grass (fescue) imposed a smaller demand on available soil moisture than did the broad-leaved types examined.

A. G. POLLARD.

**H-ion concentration of soil and its relation to the importance of white clover as a honey plant.** E. OERTEL (J. Econ. Entom., 1931, 24, 627—632).—No definite relationship exists between the  $p_{\text{H}}$  of soils and the growth of white clover. Factors other than the availability of soil Ca are important in limiting the nectar secretion of white clover.

A. G. POLLARD.

**Nitrites as a source of nitrogen for the higher plants.** W. MEVIUS and I. DIKUSSAR (Jahrb. wiss. Bot., 1930, 73, 633—703; Bied. Zentr., 1931, 60, 310—311).—Maize can utilise nitrites as a source of N in neutral and alkaline solution. The optimum concentration is approx. 50 mg. of nitrite-N per litre, but at  $p_{\text{H}}$  7.0 200 mg. per litre is not appreciably injurious. The rate of N assimilation increases with the concentration of nitrite present, and with adequate C assimilation all the N constituents of the plant tissue increase proportionally. With excessive nitrite the protein synthesis is retarded and sol. N compounds accumulate. Normally there is no appreciable accumulation of nitrites in the plant. Nitrites in acid solution also may be utilised by plants, but the optimum concentration increases with rising acidity. The physiological action of the plant on nitrites and on  $\text{NH}_3$  is similar. Where the intake of the plant is excessive in relation to the C assimilation there is a considerable increase in  $\text{NH}_3$  and amide contents. "Acute" and "chronic" forms of nitrite poisoning of the maize

plant are described. The K content of nutrient solutions has a definite influence on the N intake of the plant.

A. G. POLLARD.

**Effect of reducing substances of the roots on the development of plants in nutrient solutions containing nitrates.** M. P. MASCHKOVZEV (Arb. Cuban. landw. Inst., 1929, 8, 69—82; Bied. Zentr., 1931, 60, 311).—The reducing action of plant roots is associated with the activity of *B. herbicola aureum* and *B. coli*. Bacterial activity is particularly great on the dead roots. Nitrites are toxic to the organisms. More nitrite is formed in nutrient solutions under inoculated than under sterile plants. If the stems of sterile plants are removed, neither  $\text{NH}_3$  nor nitrite is formed in the nutrient solution. With inoculated plants nitrite, but no  $\text{NH}_3$ , is produced.

A. G. POLLARD.

**Effects of high soil moisture and lack of soil aeration on the fruiting behaviour of young cotton plants.** W. B. ALBERT and G. M. ARMSTRONG (Plant Physiol., 1931, 6, 585—591).—The proportion of  $\text{O}_2$  was lower and of  $\text{CO}_2$  higher in soil which had been flooded than in that from unflooded areas. The difference in the composition of the soil atm. is associated with the more extensive shedding of fruit buds on young cotton plants in soils of high moisture content.

A. G. POLLARD.

**Influence of dry soil on [plant] root extension.** A. H. HENDRICKSON and F. J. VEIHMAYER (Plant Physiol., 1931, 6, 567—576).—Plant roots will not penetrate into soil having a moisture content less than the permanent wetting percentage.

A. G. POLLARD.

**Nitrate fertilisation and keeping quality of apples.** J. H. GOURLEY and E. F. HOPKINS (Ohio Agric. Exp. Sta. Bull., 1931, No. 479, 66 pp.).—Applications of  $\text{NaNO}_3$  to orchard soils increased the % N in the fruit and also the total N per apple, the effect being greater in grassed than in cultivated orchards. The % N in the fruit decreased, but the total N per apple increased, as the growing season advanced. The catalase activity of the fruit was increased by nitrate treatment and was closely correlated with the % N in the fruit. The  $p_{\text{H}}$  of the juice was not affected by the manuring, but its buffer action was probably reduced. No relationship exists between the N applied and the rate of respiration or the sol. pectin content of the fruit. N fertilisers reduced the colour of the fruit and caused increased scald during storage, but did not affect physiological breakdown of the stored apples.

A. G. POLLARD.

**Refined pine-tar oil for orchard and garden use.** E. R. DE ONG (J. Econ. Entom., 1931, 24, 736—743).—Pine-tar oil has a greater insecticidal efficiency than petroleum and, moreover, readily dissolves Cu resinate and nicotine. Crude distillates contain excessive amounts of org. acids. Preps. of combined insecticide and fungicide sprays of pine-tar oil, Cu resinate, and petroleum are described.

A. G. POLLARD.

**Hydrated ferric oxide as a corrective and sticker for lead arsenate and nicotine tannate.** J. M. GINSBURG and R. F. MANN (J. Econ. Entom., 1931, 24, 695—701).—Arsenical injury to apple and peach from Pb arsenate sprays is eliminated by admixture of



hydrated  $\text{Fe}_2\text{O}_3$  (4–8 lb. per 50 gals. of spray liquor). With such mixtures more As is retained on the foliage than when CaO is used as a corrective. The retention of nicotine from nicotine tannate sprays is similarly increased. A 1:1 mixture of powdered skim milk and  $\text{Fe}_2\text{O}_3$  proved a satisfactory sticker and spreader for general use in insecticides and fungicides.

A. G. POLLARD.

Relative toxicity of rotenone and nicotine to *Aphis rumicis*, L., and mosquito larvæ. H. H. SHEPARD (J. Econ. Entom., 1931, 24, 725–731).—No deterioration in toxic val. occurred during storage of  $\text{COMe}_2$  or EtOH solutions of rotenone. Slight acidity or alkalinity, such as is caused by the use of saponin or soap as spreader, increased the rate of deterioration of aq. suspensions of rotenone. Acids accelerate the settling of these suspensions. Rotenone is definitely more toxic than nicotine.

A. G. POLLARD.

Insecticidal properties of Devil's Shoestring (*Cracca virginiana*, Linn.). V. A. LITTLE (J. Econ. Entom., 1931, 24, 743–754).—Aq. suspensions of the powdered root of *C. virginiana* have a similar toxicity to *Derris*, pyrethrum, and nicotine, and are particularly efficient in the control of animal parasites.

A. G. POLLARD.

Effectiveness of dust fungicides in controlling grain smut in sorghum. B. N. UPPAL and M. K. DESAI (Agric. and Live Stock in India, 1931, 1, 396–413).—S, Cu "carbonate", and  $\text{CuSO}_4$  effectively controlled smut without injury to germination. Stimulated germination did not follow either Cu treatment, but S-dusted seed benefited in this respect. Cu "carbonate" retained its activity longer than  $\text{CuSO}_4$ .

A. G. POLLARD.

Control of the lesser peach borer with *p*-dichlorobenzene solutions. O. I. SNAPP and J. R. THOMSON (U.S. Dept. Agric., Circ., 1931, No. 172, 11 pp.).—Cottonseed oil solutions of *p*- $\text{C}_6\text{H}_4\text{Cl}_2$  (2:1) used as a tree paint gave better results than when linseed, maize, or petroleum oil or pine-tar creosote was used as solvent or when the material was used as a spray. The cottonseed oil solution may be stored without deterioration in sealed containers.

A. G. POLLARD.

Chemistry of codling-moth baits. J. R. EYER and H. RHODES (J. Econ. Entom., 1931, 24, 702–711).—The attractant value of molasses baits is associated with certain changes, e.g., decrease in the dextrose content, in the composition of the sugars which precede the formation of EtOH and AcOH. Ester formation is suggested. The production of EtOH and  $\text{CO}_2$  are important secondary factors of attraction, but the AcOH subsequently formed is only slightly attractive or even repellent. With a mean daily temp. of 28–5° addition of yeast increased the attractiveness of baits, and their active periods could be prolonged by the addition of preservatives, notably NaOBz. The attractiveness of the esters examined was in the order  $\text{C}_6\text{H}_4\text{Bu}^t\text{OAc} > \text{geranyl formate} > \text{Ph}_2\text{O} > \text{citronellal} > \text{bromostyrene} > \text{Me cinnamate}$ . Most of these were less attractive than was plain molasses, and their attractiveness was dependent on chemical composition, b.p., and odour.

A. G. POLLARD.

Waste sulphite material of paper mills as an adjuvant to certain spray materials. R. H. HURT (Virginia Agric. Exp. Sta. Bull., 1931, No. 277, 10 pp.).—Lignin pitch, obtained by digesting wood lignin with  $\text{Ca}(\text{HSO}_3)_2$ , is a satisfactory emulsifier, sticker, and wetting agent for common spray materials and may be substituted, wt. for wt., for Ca caseinate.

A. G. POLLARD.

Dry rot in sugar beet in relation to soil conditions. S. MIKLASZEWSKI (Zaklad. Gleboznawstwa, Warszawska, 1930; Proc. Internat. Soc. Soil Sci., 1931, 4, 75–77).—Dry rot in sugar beet occurs only on alkaline soils. It is immaterial whether the alkalinity is natural or produced by liming. The appearance of the disease is in part related to the relatively lower  $\text{H}_2\text{O}$  content of alkaline soils.

A. G. POLLARD.

Determination of  $\text{H}_3\text{PO}_4$ .  $(\text{NH}_4)_2\text{SO}_4$ . Cyanide. Storage of  $\text{CaCN}_2$ .—See VII.

## PATENTS.

Manufacture of [phosphatic] fertilisers. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 355,637, 23.1.31. U.S., 24.1.30).—Phosphatic rock, which may be of low grade, is treated with sufficient  $\text{HNO}_3$  to yield a product containing  $\text{H}_2\text{O}$ -sol. Ca phosphates,  $\text{Ca}(\text{NO}_3)_2$ , and free acid, the mixture is treated with sufficient  $\text{NH}_3$  to neutralise the acid and to convert at least part of the  $\text{H}_2\text{O}$ -sol. phosphate into citrate-sol. phosphate and the  $\text{Ca}(\text{NO}_3)_2$  into  $\text{NH}_4\text{NO}_3$ , and the product is treated with the gases evolved during the decomp. stage.

L. A. COLES.

Fertiliser obtainable by heating ammonium thiocyanate. O. RIPKE, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,793,133, 17.2.31. Appl., 29.5.28. Ger., 31.8.27).— $\text{NH}_4\text{CNS}$  is heated at about 300° until evolution of gases ceases.

L. A. COLES.

Production of [cellular] composite material suitable for promoting the growth of plants. A. F. BERRY (B.P. 355,278, 17.4.30).—Mixtures comprising a base material (e.g., asbestos cement and/or vegetable fibre and Portland cement), a plant food (e.g., basic slag,  $\text{NaNO}_3$ , animal manure), and  $\text{H}_2\text{O}$  are worked up to a hard product rendered cellular, e.g., by the addition of Al powder,  $\text{Na}_2\text{S}_2\text{O}_4$ , etc. to the mixture and/or by blowing with air.

L. A. COLES.

Seed disinfectant. F. J. FUNK, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,793,922 and 1,793,686, 24.2.31. Appl., [A] 18.3.26, [B] 21.5.27).—The products comprise colloidal material, e.g., potters' clay, kaolin, bentonite, gelatin, glue, agar-agar, and (A) a mercurised chloro- or nitro-phenol, or (B) an inorg. Hg compound, e.g.,  $\text{HgCl}_2$ ,  $\text{HgSO}_4$ .

L. A. COLES.

Fertilisers.—See II.

## XVII.—SUGARS; STARCHES; GUMS.

Dry rot in sugar beet.—See XVI. Honey.—See XIX.

## PATENTS.

Treating liquids.—See I. Saccharification of carbohydrates.—See V.

## XVIII.—FERMENTATION INDUSTRIES.

**Determination of the dextrinolytic activity of malt.** L. FLETCHER and J. B. WESTWOOD (J. Inst. Brew., 1931, 37, 470—478).—Starch which has been hitherto used as the substrate for the enzymic reaction has been replaced by  $\alpha$ -amylodextrin. 1 c.c. of a 4% aq. extract of ground malt is allowed to act for 1 hr. at 40° on 70 c.c. of a 3% aq. solution of  $\alpha$ -amylodextrin, buffered to  $p_H$  4.9—5.0 by mixed phosphates. The action is stopped by the addition of 3 c.c. of 2N-NaOH and the vol. made up to 100 c.c. at 15.6°. The CuO-reducing power is determined gravimetrically on 25 c.c. of the solution and the dextrinolytic activity expressed as g. of maltose produced by 1 g. of dry malt. In the early stage of the enzymic reaction the ratio of enzyme concentration to the amount of hydrolysis approaches a linear function, and the dextrinolytic activity approaches a const. up to  $R$  13—15. The zone of optimal  $p_H$  lies between 4.5 and 5.2 and is more sharply defined than with starch as the substrate. The activity as measured with  $\alpha$ -amylodextrin is not definitely related to the value as determined with starch, and, in general, it has about half that value.

C. RANKEN.

**Free acidity of some Portuguese beers.** A. V. DE LEMOS (Rev. Chim. Pura Appl., 1930, [iii], 5, 38—41).—The difference between the results obtained by the H and quinhydrone electrodes does not exceed  $p_H$  0.07. The second method is, in general, the more convenient.

H. F. GILLBE.

**Theory of ethyl alcohol rectification.** O. VON KEUSSLER (Chem.-Ztg., 1931, 55, 669—671).—A crit. survey is made of the literature dealing with the rectification of EtOH and the azeotropic mixtures involved.

T. McLACHLAN.

MeOH and EtOH.—See III.

## PATENTS.

**Preparation of beer weak in alcohol.** A. HASELBACH (B.P. 354,766, 12.5.30).—Malt wort prepared with approx. double the addition of hops usual with top-fermentation beers is fermented normally with top-fermentation yeast. If desired, wort containing half the amount of malt may be fermented as above, and the second half of the malt or a sugar solution added at the close of the fermentation.

C. RANKEN.

**Sterilisation of beer.** BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (B.P. 355,010, 7.8.30. Austr., 10.7.30).—Beer is heated to approx. 100° for 1—2 sec. by passing it at 10—15 atm. pressure between heated surfaces which are not more than 1 mm. apart and are coated with or composed of Ag, Ni, or other metal having germicidal power. The sterilised beer is cooled by passage through the preheater.

C. RANKEN.

Abs. EtOH.—See III. Water for brewing.—See XXIII.

## XIX.—FOODS.

**Testing the colour of flour.** R. RÜTER (Mühlenlab., 1931, No. 2, 9—13).—Errors in comparing the colours of flour samples arise through variations in compression, fineness, moisture content, and incidence of the light.

These factors also affect the comparison of the moistened samples, owing to variations in the thickness of the translucent dough layer. Better results are obtained by comparing doughs prepared from the samples, but the best criterion is the colour of the bread crumb, as this takes into account not only the colour of the flour, but also its baking qualities.

W. J. BOYD.

**Colloid chemistry of acid gluten solutions.** E. BERLINER (Mühlenlab., 1931, No. 2, 13—14).—The deductions and methods of Ruemele relating to gluten evaluation (B., 1931, 737, 943) are criticised.

W. J. BOYD.

**Soft curd milk.** R. L. HILL (Utah Agric. Exp. Sta. Bull., 1931, No. 227, 24 pp.).—Softness of milk curd (cf. author's test, J. Dairy Sci., 1923, 6, 509) is characteristic of the cow and, except for drastic changes, independent of the nature of the ration. The curd tends to harden in the later stages of the lactation period. Removal of the fat from milk produces a harder curd, whereas prolonged heating as in the manufacture of evaporated milk has a softening effect. Soft curd milk (< 20 g. tension by the Hill test) is digested by delicate infants without dilution. Hard curd milk is preferred for cheese-making.

A. G. POLLARD.

**Determination of keeping quality of milk.** I. Methods of comparison. II. Observations in sextuplicate. III. Observations at different temperatures and at successive intervals on the same sample. A. II. ROBERTSON (Vermont Agric. Exp. Sta. Bulls., 1930, No. 314, 46 pp.; No. 315, 39 pp.; and No. 316, 55 pp.).—I. [With J. M. FRAYER.] The keeping quality of milk as judged by the increase in bacterial numbers from the time of sampling until souring is better based on the geometric than on the arithmetic mean of the counts. When used as an index of the keeping quality the logarithmic val. of plate counts is preferred as showing less variation. The plate-count method is more accurate than the methylene-blue test for samples of low bacterial content, but inferior for highly bacterial milks.

II. [With R. I. MOODY and J. M. FRAYER.] Numerous comparisons of plate-count with methylene-blue tests are recorded.

III. [With J. M. FRAYER.] The various tests of keeping quality are fairly consistent for samples stored at 21—27°. At lower temps. the methylene-blue tests show wider variations than the plate counts.

A. G. POLLARD.

**Examination of commercial egg-yolk.** R. F. INNES (J. Soc. Leather Trades' Chem., 1931, 15, 434—438).—Fresh egg-yolk, 3 commercial samples, and a synthetic egg-yolk were analysed for loss in wt. at 105°, light petroleum extract, ash, NaCl, N, P, and emulsifiability on adding 1 drop to 5 c.c. of H<sub>2</sub>O. The results for one commercial sample and for the fresh egg-yolk were similar, but the former gave the higher ash and NaCl contents. The light petroleum extract of another commercial sample was high and the N and P contents were low; the unsaponifiable matter in the oil was also low and a poor emulsion was obtained in H<sub>2</sub>O. Greasy leather resulted from its use, and it was concluded that some animal oil had been mixed in with the product.

to emulsion in  $H_2O$  was obtained with the synthetic egg-yolk. The figures for the third commercial sample were normal. A spuc scraped from leather treated with it was found to be a mixture of palmitic and stearic acids, the development of which could not be ascribed directly to the sample. D. WOODROFFE.

**Chemical changes in the fat of frozen and chilled meat. III. Bacon.** C. H. LEA (J.S.C.I., 1931, 50, 343—349 r).—The free acidity values observed in the fat of tank-cured bacon during storage at  $-10^\circ$  for periods up to 152 days, followed by hanging at  $15^\circ$  for 18 days, gave no indication of attack by micro-organisms on the fat, nor were any signs of tainted flavour observed which might have been attributed to this cause. The superficial fat of freshly cured bacon was found to contain small quantities of active O of the order of 2—7 c.c. (as 0.002N- $Na_2S_2O_3$  solution per g.) which rendered the fat very susceptible to further oxidation during storage. When stored at  $-10^\circ$  the exposed surface of the fat oxidised comparatively rapidly, reaching active O contents of 30—60 c.c. in 152 days, without, however, developing any appreciable yellow colour. At  $15^\circ$ , on the other hand, the bacon became yellow on the surface, and (after cold storage) sometimes in the interior of the fat. On cooking, the oxidised portions of the fat had an unpleasant rancid flavour. The effect of smoking on bacon after storage at  $-10^\circ$  was to arrest the progress of superficial oxidation for a period, and there is some evidence that oxidation in the interior of the fat may also be somewhat retarded. So far as could be ascertained, rapid cooling of the carcasses of pork at  $5^\circ$  prior to curing had no appreciable effect on the fat. The addition of cod-liver oil in small quantities to the diet of the pigs up to the time of slaughter was without effect on the I val. of the fat of the bacon, but appeared to have a somewhat adverse effect on its keeping properties.

**Honey investigations.** E. F. PHILLIPS (J. Econ. Entom., 1931, 24, 581—589).—A review of the problems involved in the preparation of honey for marketing. The need of wider examination of the physical properties of honey is emphasised. A. G. POLLARD.

**Crystallisation of honey.** E. J. DYCE (J. Econ. Entom., 1931, 24, 597—602).—The quality of cryst. honey is highest when the crystals are small. Crystal growth and the factors and method of controlling fine crystallisation are discussed. A. G. POLLARD.

**Effect of temperature on honey in storage.** H. F. WILSON and G. E. MARVIN (J. Econ. Entom., 1931, 24, 589—597).—Honey may be stored below  $11^\circ$  without change of colour or flavour. Storage at  $11$ — $18^\circ$  involves greater risk of fermentation than at higher or lower temps. At  $26.6^\circ$  colour change and risk of fermentation are small. From  $26.6^\circ$  to  $38^\circ$  fermentation is unlikely, but the colour is affected. The possibility of the partial bleaching of honey by sunlight is noted. A. G. POLLARD.

**Moisture determination in honey by means of the refractometer and the vacuum drying oven.** G. E. MARVIN and H. F. WILSON (J. Econ. Entom., 1931, 24, 603—604).—The sand-bath method is of doubtful accuracy and gives results consistently 2—2½%

lower than those by the refractometer method. There is a fairly uniform gradation between the results of the above and the glass-plate, blotting-paper, and asbestos methods. A. G. POLLARD.

**Treatment of soya beans for human consumption.** M. KRAJČINOVIČ (Arh. Hemijn, 1931, 5, 239—242).—The amount of substance extracted by  $H_2O$  from the raw beans increases with the acidity of the  $H_2O$  from 2.5% for ordinary  $H_2O$  to 8.9% for 0.5% HCl. Alkaline extraction is unsuitable. The digestion coeff. is increased by the above treatment. R. TRUSZKOWSKI.

**Effects of acetylene on the ripening processes of bananas.** R. HARTSHORN (Plant Physiol., 1931, 6, 467—484).—Treatment with  $C_2H_2$  accelerated the ripening process, as shown by the rates of softening, respiration, and of starch hydrolysis, and by changes in colour and flavour. A. G. POLLARD.

**Vitamins in canned foods. XI. A canned food diet.** E. F. KOHMAN, W. H. EDDY, and C. Z. GURIN (Ind. Eng. Chem., 1931, 23, 1064—1066; cf. B., 1931, 944).—Rats and guinea-pigs fed on a diet consisting wholly of canned foods (sterilised by heat) show normal reproduction in the fifth and third generations, respectively. The rate of growth (and wt. at maturity) is higher than that reported as normal. H. BURTON.

**Salmon oil and canned salmon as sources of vitamin-A and -D.** C. D. TOLLE and E. M. NELSON (Ind. Eng. Chem., 1931, 23, 1066—1069).—The oil recovered from canned salmon contains as much vitamin-D as does cod-liver oil; little vitamin-A is present. Oils produced from the offal contain much vitamin-D, but the vitamin-A content varies appreciably. Commercial oils show considerable variations in their vitamin contents; the amount of -D is usually of the same order as in cod-liver oil. H. BURTON.

**Industrial wastes.**—See XVI and XXIII.

#### PATENTS.

**Production of a milk preparation.** W. R. B. ST. J. GATES, J. TAVROGES, and COW & GATE, LTD. (B.P. 354,917, 11.6.30).—Milk powder is mixed with sugar, milk is added, and the whole is evaporated with continuous stirring under vac. The product, after being moulded and cooled, has 10—15% of moisture. E. B. HUGHES.

**Treatment of natural or artificial cream.** B. BORTHEN (B.P. 355,611, 29.11.30. Nor., 29.11.29).—Cream is treated in homogenisers at different temps., first above the m.p. of the emulsified fat, then below this point, whereby the consistency and flavour of the cream are improved. H. ROYAL-DAWSON.

**Treatment of tea and coffee.** H. MUCH (B.P. 354,771, 5.5.30).—It is claimed that in tea and coffee the properties considered harmful are rendered ineffective by first treating the dry substance with "lipid tannic acids" (prep. described). E. B. HUGHES.

**Improving the taste of, and removing poisonous matter from, infusions of tea and coffee.** W. STELKENS (B.P. 354,942, 1.7.30. Ger., 2.7.29).—Undesirable flavours are removed from tea and coffee infusions

by mixing the dry substance with activated C or  $\text{H}_2\text{SiO}_3$  before preparing the drink, with or without  $\text{H}_2\text{O}$ -sol. substances such as glycerin or sugar. E. B. HUGHES.

**Jelly preparation.** CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 355,130, 11.11.30. U.S., 12.11.29).—Pectin is mixed with a material which will liberate a free edible acid on boiling, such as acid anhydrides, acid chlorides, esters, compounds of the type of glycerotriphosphoric acids, K chloroacetate, etc. The disadvantages of prejellation and lumpiness of the jelly are thus avoided, a correct  $p_{\text{H}}$  is ensured, and risk of deterioration of the pectin on keeping removed.

E. B. HUGHES.

**Roasting oven. Processing apparatus [for milk].**—See I. Air for food preservation.—See XI.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Testing of pharmaceutical hydrogen peroxide.** R. A. DINIS (Rev. Chim. Pura Appl., 1930, 5, [iii], 106—121).—Methods are described in detail.

H. F. GILLBE.

**Extraction as applied to N.F. [National Formulary] preparations.** E. GUTH and H. A. LANGENHAN (J. Amer. Pharm. Assoc., 1931, 20, 746—754).—The total extractive, the % of total extractive extracted under varying conditions, and attempts to measure the amount of swelling occurring when various vegetable drugs are treated with alcoholic menstrua of different EtOH concentrations are described. Drugs containing measurable constituents (jalap and nux vomica) have been similarly examined, and the composition and uses of the alcoholic menstrua of the U.S.P. and N.F. are discussed.

E. H. SHARPLES.

**Decomposition and preservation of fluid extracts of ergot, D.A.B. VI.** H. OETTEL (Arch. exp. Path. Pharm., 1931, 161, 359—367).—The extracts may lose over half their activity within 1 year, but a drier preparation made by the Krause method from an extract, D.A.B. VI, retained its activity intact on keeping.

W. O. KERMACK.

**Cantharidin from *Mylabris pustulata*, Fb., India.** B. H. IYER and P. C. GUHA (J. Indian Inst. Sci., 1931, 14A, 31—39).—The adult beetles are collected by hand, killed with  $\text{CHCl}_3$ , sun-dried, and preserved whole or powdered. For free cantharidin the powder is extracted with  $\text{CHCl}_3$ , the fats being removed from the  $\text{CHCl}_3$  extract by  $\text{CS}_2$ . Cantharidin is crystallised from EtOH (m.p.  $218^\circ$ ; yield 1.35%). For total cantharidin 100 g. of powder are treated with 150 g. of EtOAc and 2 g. of  $\text{H}_2\text{SO}_4$ , after 48 hr. 4 g. of  $\text{BaCO}_3$  are added, and the mixture is extracted for 48 hr. with EtOAc. After removal of the solvent, the residue is macerated several times with cold light petroleum and the crude cantharidin treated with a small quantity of hot EtOH (yield 2.3%). Cantharidin is slowly volatilised. *Cantharidinhydrazide*,  $\text{C}_{10}\text{H}_{14}\text{O}_3\text{N}_2$ , m.p.  $118^\circ$ , prepared by treating an alcoholic solution of cantharidin with an excess of hydrazine hydrate at  $0^\circ$  and allowing the temp. to rise to that of the room, on treatment with  $\text{PhCHO}$  in aq. solution at  $100^\circ$  for  $\frac{1}{2}$  hr. forms a *benzylidene* derivative,  $\text{C}_{17}\text{H}_{18}\text{O}_3\text{N}_2$ , m.p.  $158^\circ$ .

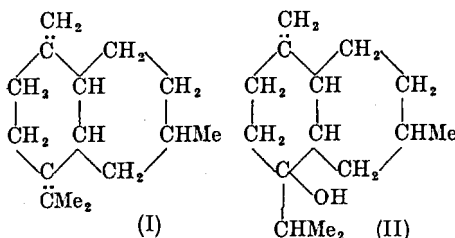
C. C. N. VASS.

**Assay of tablets of resin of *Podophyllum*.** L. E. WARREN (J. Assoc. Off. Agric. Chem., 1931, 14, 380—386; cf. B., 1930, 393).—The powdered tablets are mixed with sand (13 times wt. of resin) and exhausted by percolation with EtOH, followed by hot continuous extraction with a fresh supply of the same solvent. An aliquot portion of the tincture is treated with 0.6% HCl and repeatedly extracted with equal vols. of  $\text{CHCl}_3$ . The  $\text{CHCl}_3$  is washed with 0.6% HCl, which is again extracted with  $\text{CHCl}_3$ . The combined  $\text{CHCl}_3$  extracts are evaporated to dryness, 1 c.c. of EtOH is added, and the whole again taken to dryness. T. McLACHLAN.

**Determination of mercury in preparations and mixtures.** G. WEISSMANN (Pharm. Zentr., 1931, 72, 561—565).—The prep., after destruction of org. matter if necessary, is heated with  $\text{H}_2\text{O}$ , HCl, and  $\text{NaH}_2\text{PO}_2$ , and the ppt. is filtered off, washed free from Cl, dissolved in  $\text{HNO}_3$ ,  $\text{KMnO}_4$  added, decolorised with  $\text{FeSO}_4$ , and titrated with 0.1N- $\text{NH}_4\text{CNS}$ . Many org. substances need no preliminary destruction of org. matter, but give metallic Hg on treatment with  $\text{NaH}_2\text{PO}_2$ . Modifications of the method for the determination of Hg in medicinal preps. are given.

E. H. SHARPLES.

**Sesquiterpenes. I. Sesquiterpene and sesquiterpene alcohol from Japanese camphor oil.** S. KOMATSU, H. FUJIMOTO, and S. TANAKA (Mem. Coll. Sci. Kyoto, 1931, A, 14, 149—171).—The sesquiterpene, which probably exists as (I) in the oil, is isolated from the high-boiling fraction as a mixture, b.p.  $119-127/8$  mm.,  $[\alpha]_D +16.2^\circ$  to  $+17.3^\circ$ ; isomerisation (migration of double linking into ring) occurs during distillation over Na. The sesquiterpene is unaffected by



Na and amyl alcohol, is dehydrogenated by S at  $180-200^\circ$  to cadalene, is oxidised by  $\text{KMnO}_4$  in aq.  $\text{COMe}_2$  to isophthalic acid, by  $\text{MnO}_2$  and  $\text{H}_2\text{SO}_4$  to prehnitic acid, and by  $\text{O}_3$  in  $\text{CHCl}_3$  to  $\text{CH}_2\text{O}$ , formic acid, an aldehyde, and a diketone  $\text{C}_{11}\text{H}_{14}\text{O}_2$ , b.p.  $100-110/4$  mm. (*semicarbazone*, m.p.  $219^\circ$ ). The alcohol (II), b.p.  $157-160/12$  mm.,  $[\alpha]_D +35.5^\circ$ , isolated through the Na salt from the oil (fraction b.p.  $140-180/12$  mm.), is dehydrogenated by S at  $180-230^\circ$  to cadalene, and reduced catalytically (Pd) in AcOH at  $40-50^\circ$  to the corresponding saturated alcohol (III), b.p.  $145-150/12$  mm.,  $[\alpha]_D +7.5^\circ$ . Reduction of (II) with  $\text{H}_2$  and Ni at  $150-160/80$  atm. also gives (III) (with b.p.  $160-168/19$  mm.,  $[\alpha]_D +16.6^\circ$  to  $+20.7^\circ$ ), which is dehydrated by Japanese acid clay to a hydrocarbon (IV),  $\text{C}_{15}\text{H}_{26}$ , b.p.  $138-140/20$  mm. (IV) and  $\text{O}_3$  in  $\text{CCl}_4$  give  $\text{COMe}_2$  and a product, reduced by Na and EtOH to an alcohol  $\text{C}_{12}\text{H}_{22}\text{O}$ , b.p.  $120-130/4$  mm.,  $[\alpha]_D -7.2^\circ$  (dehydrated to a hydrocarbon  $\text{C}_{12}\text{H}_{20}$ , b.p.  $145-155/25$  mm.). Oxidation of (II) with alkaline  $\text{KMnO}_4$  gives oxalic and  $\beta$ -methyladipic acids, whilst with  $\text{O}_3$  in  $\text{CCl}_4$ ,  $\text{CH}_2\text{O}$ , formic acid, and a keto-alcohol,  $\text{C}_{14}\text{H}_{24}\text{O}_2$ , are produced. Reduction of this

keto-alcohol with Na and EtOH affords an alcohol  $C_{14}H_{26}O$ , b.p. 137–140°/5 mm., dehydrated to a hydrocarbon  $C_{14}H_{24}$ , b.p. 143–145°/30 mm. (ozonolysis products,  $COMe_2$  and a compound  $C_{14}H_{24}O_3$ ). Dehydration of (II) with  $Ac_2O$  at 150–160° gives a hydrocarbon  $C_{15}H_{24}$ , b.p. 136–142°/16 mm.,  $[\alpha]_D +51.4^\circ$  [additive compound with  $Hg(OAc)_2$ ; ozonolysis products,  $CH_2O$ , formic acid, and a compound  $C_{14}H_{22}O_4$  (semicarbazone, m.p. 63–65°)], whilst with Japanese acid clay a mixture of hydrocarbons  $C_{15}H_{24}$ , b.p. 131–134°/14 mm.,  $[\alpha]_D +22.1^\circ$  [ozonolysis products,  $COMe_2$  and a compound  $C_{12}H_{18}O_4$  (semicarbazone, m.p. 55–57°)], and b.p. 137–140°/14 mm.,  $[\alpha]_D -23.7^\circ$  (ozonolysis products,  $COMe_2$  and a compound  $C_{11}H_{18}O_3$ ), is produced. (II) is dehydrated by phenylcarbimide to a hydrocarbon  $C_{15}H_{24}$ , b.p. 134–139°/18 mm.,  $[\alpha]_D +46.6^\circ$  [dihydrochloride; additive compound with  $Hg(OAc)_2$ ], oxidised by  $O_3$  to a substance (semicarbazone, m.p. 90–93°). Alternative formulæ are suggested for these hydrocarbons.

H. BURTON.

Fatty acids from cantharis oil.—See XII. Pyrethrum extracts.—See XXIII.

## PATENTS.

Remedies for combating diseases. C. F. CHARLTON (B.P. 355,435, 2.7.30).—The omentum or mesentery of a pig or other mammal is ground and extracted with aq. EtOH. The extract is standardised by dilution with NaCl solution followed by a determination of its action in dissolving red blood cells. For hypodermic injection the EtOH is removed and replaced by  $H_2O$ .

E. H. SHARPLES.

Dental cement.—See IX. Air for therapeutic purposes.—See XI.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

The photographic emulsion: after-ripening. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1931, 7, 219–259).—The changes in sensitivity which occur after washing the emulsions ("after-ripening") and the effects produced by eight variables are discussed. The amount of after-ripening depends on the extent to which sensitivity nuclei have been formed during ripening. The influences of temp., and of bromide, chloride, and  $[H]$ , are in agreement with predictions of the chemical reactions forming sensitivity nuclei. Experiments with different kinds of gelatin have been conducted; the gelatin/Ag halide effect can be explained by its influence on the rate of development. An increasing % of AgI leads to an increased practicable after-ripening. After-ripening during storage is discussed. Chemical analysis shows that there is an increase in non-halide Ag; the non-halide Ag is photographically inert.

W. R. ANGUS.

Development of the solarised latent image. A. P. H. TRIVELLI and E. C. JENSEN (J. Franklin Inst., 1931, 212, 351–367).—The development of such images for pure AgBr emulsions with *p*-aminophenol, pyrogallol, and quinol developer without KBr, and with quinol developers containing 0.002–0.269*N*-KBr, has been investigated. For normal exposures the density is proportional to the logarithm of the KBr

concentration, deviations occurring with extreme exposure and development. The proportionality holds only in a limited way for the solarisation region. At the threshold of the first reversal of the solarisation there is no correlation. The tie-point of the solarisation curves moves towards the origin of the distance/log *E* axes with increasing content of KBr in the developer.

H. J. EMELÉUS.

## PATENTS.

[Multi-colour] photographic screens. B. GÁSPÁR (B.P. 354,167, 31.1.30. Ger., 2.2.29).—Thermoplastic threads with coloured cores are wound on a spool in many layers. The spool is cut open, flattened out, stretched under heat, and cut into sections, which are superimposed; the treatment is repeated till a large block is formed, from which thin sections are cut and pressed on carriers to form colour screens.

J. LEWKOWITSCH.

Sensitising photographic emulsions. KODAK, LTD., Assecs. of L. G. S. BROOKER (B.P. 354,264, 3.2.30. U.S., 2.2.29).—A naphthothiocarbocyanine dye is used to sensitise emulsions beyond 640 mμ. Analogues containing O or Se in place of S may be used.

J. LEWKOWITSCH.

[Producing markings for synchronisation on] sound and picture films. I. G. FARBERIND. A.-G. (B.P. 354,455, 5.6.30. Ger., 5.6.29).

Fireproofing materials.—See VI.

## XXII.—EXPLOSIVES; MATCHES.

Distribution of solvent throughout [nitrocellulose] powder grains. J. DESMAROUX (Mém. Poudres, 1930—1, 24, 101–105).—The powder consisted of a mixture of  $CP_1$  and  $CP_2$  (N 12.88%), containing 40% of sol. nitrocellulose, incorporated with  $Et_2O$ -EtOH. Samples were prepared by drilling holes of various diameters in portions cut from the cords and removing the outer surface in a lathe so as to give const. thickness. Tests were made (a) on the dried powder and (b) on the powder after steeping at 80° for 32 hr. When the diameters of the samples were 9–11, 6–8, and 3–5 mm., respectively, the % of EtOH and  $Et_2O$  were 2.60, 5.73; 2.62, 6.89; 2.56, 6.04 in the dried powder, and 1.13, 4.38; 1.45, 5.46; 1.50, 4.90 in the steeped powder. An undrilled sample (1.5 mm. diam.) contained EtOH 1.87% and  $Et_2O$  5.13%. In the steeped samples the amount of EtOH at the surface was 0.60, and of the  $Et_2O$  0.85 of that at the centre. Distribution of solvent does not in any way follow a sinusoidal curve. The diffusion in a thin superficial layer is very slow.

W. J. WRIGHT.

Determination of moisture in SD powder and SD powder paste. G. A. FLEURY and LAMBERT (Mém. Poudres, 1930—1, 24, 137–145).—Errors in Dupré's method are due to the difficulty in determining the vol. of gas owing to the influence of temp. and pressure, occlusion of gas by the carbide, the solubility of  $C_2H_2$  in salt solution, the correction for tension of aq. vapour instead of the vapour of salt solution, and impurities in the  $C_2H_2$ . A modification adopted by the Commission des Poudres with a blank test gave good results with powders, but was unsuitable for paste. In a method used at Sévran, the powder is heated with

$C_6H_6$ , and the  $H_2O$  distilled over with the solvent is read off. For SD dough containing 0.46, 5.2, 8.8, 15.8, and 21.2%  $H_2O$ , respectively, the corresponding values found were 0, 4.9, 8.6, 15.8, and 21.2%. This method gives more accurate results than drying in vac. over  $H_2SO_4$ . It is unsuitable for powders containing volatile solvent, but may be used for black powder, ballistite, nitrocellulose powders, and nitroglycerin. W. J. WRIGHT.

**Analysis of the decomposition products of nitrocellulose powder heated to 135° in a current of inert gas.** J. GOUJON (Mém. Poudres, 1930—1, 24, 73—85).—Samples of  $CP_1$ , free from carbonate and dried at 100°, were heated for 8 hr. in a current of pure  $N_2$ . Analysis of the products of decomp. gave:  $NO_2$  27.18,  $NO$  32.72,  $N_2$  1.97,  $N_2O$  1.57,  $H_2O$  20.83,  $CO_2$  12.17, and  $CO$  3.44%, 60% of the total N being evolved as NO and 30% as  $NO_2$ . Contrary to Koehler and Marquoyrol's (B., 1922, 348 A) contention that the decomp. products of CP powder when heated in vacuum at 40°, 75°, and 100° contain only  $N_2$ , NO, and  $NO_2$ , it is shown that a correct interpretation of their results proves them to be analogous to those obtained at 135°, the actual figures being 50% of the N as NO and 35% as  $NO_2$ . In both series of experiments the amounts of  $H_2O$ ,  $CO_2$ , and CO were equal, the amount of  $H_2O$  being nearly twice as much as that found by Will. W. J. WRIGHT.

**Determination of the capacity of nitrocellulose for gelatinisation of nitroglycerin.** J. DESMAROUX (Mém. Poudres, 1930—1, 24, 86—100).—The method consisted in maintaining a small quantity of dried nitrocellulose in contact with a solution of nitroglycerin in  $CHCl_3$  until equilibrium was established. Assuming the nitroglycerin to have the same free energy in the two phases, a method of calculating the free energy in  $CHCl_3$  is given. The values for the activity of the nitroglycerin in  $CHCl_3$  and in nitrocellulose of various N contents are tabulated and plotted. W. J. WRIGHT.

**Equilibrium of nitrocellulose-nitroglycerin-water mixtures.** P. DEMOUGIN (Mém. Poudres, 1930—1, 24, 106—118).—The feasibility of rendering SD powder more homogeneous by treating the nitrocellulose-nitroglycerin mixture with  $H_2O$  was examined. Samples of CP were impregnated with various amounts of nitroglycerin in presence of a fixed amount of  $H_2O$ , the nitrocellulose-nitroglycerin mixture being then separated from the aq. nitroglycerin solution and the respective nitroglycerin contents being found from N determinations. The amount of nitroglycerin dissolved from a 66/25 nitrocellulose-nitroglycerin mixture was about 0.9 g./litre. Treatment of such paste with about 10 times its wt. of  $H_2O$  would involve a loss of about 1% of nitroglycerin. As this represents only two thirds of the amount required for saturation, there is no risk of separation of nitroglycerin by repeated use of the same  $H_2O$ , especially since the solubility varies little with the differences of temp. experienced in practice. W. J. WRIGHT.

**Dynamite-grade glycerin.**—See XII.

#### PATENTS.

**Primer composition.** C. H. PRITHAM (U.S.P. 1,794,732, 3.3.31. Appl., 1.12.26).—A non-corrosive

primer composition for percussion caps consists of Hg fulminate 37,  $Ba(NO_3)_2$  32, Sb sulphide 28, ground glass 3, and an explosive, e.g., trinitrotoluene or pentaerythritol tetranitrate 4—8 pts. W. J. WRIGHT.

**Nitrocellulose propellant explosive.** C. M. A. STINE and C. E. BURKE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,792,516, 17.2.31. Appl., 27.5.25. Renewed 25.7.27).—The explosive comprises nitrocellulose mixed with a nitrated ester of lactic acid and a polyhydric alcohol having 2—3 OH groups, e.g., nitroglyceryl or nitroglyceryl nitrolactate (cf. U.S.P. 1,792,515; B., 1931, 965). W. J. WRIGHT.

**Nitrated esters of polyhydric alcohols.**—See III. **Pyroxylin.**—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Treatment of sewage containing industrial wastes.** R. S. LANPHEAR (Sewage Works J., 1931, 3, 276—282).—The sewage of Worcester, Mass., contains waste from foundries, dyeworks, slaughter-houses, wire mills, and tanneries. The spent acid and CaO from the last two are the most important and cause a considerable increase in sludge production, but little trouble otherwise as long as the rates of admission to the sewer are regular. The cost of plant operation, exclusive of any fixed charges, is \$0.26 per head. In an experimental activated-sludge plant 5 cu. ft. of free air were required per gal. of sewage treated, with an aeration period of 6.3 hr. and a sludge re-aeration period of 5.1 hr. C. JEPSON.

**Effects of industrial wastes on the operation of municipal sewage treatment works.** L. F. WARRICK (Sewage Works J., 1931, 3, 266—275).—The overloading of small sewage plants with large vols. of waste from creameries and vegetable canning plants should be prevented by pre-treatment at the factory. All plants should try to equalise their rate of flow; in creameries this might be difficult owing to acid fermentation. Corn and pea-canning factories should pass their waste through a fine-mesh screen before coagulating with  $FeSO_4$  and  $Ca(OH)_2$  and treating on a 6—7-ft. percolating filter. Mineral oils should be excluded from plants operating the activated-sludge process. C. JEPSON.

**Operation results at the Los Angeles sewage reclamation plant.** R. F. GOUDY (Sewage Works J., 1931, 3, 214—222; cf. B., 1931, 567).—As a result of 7 months' operation it is claimed that the effluents obtained from this experimental plant are suitable for drinking water. Details of the sewage purification installation are given; the activated-sludge plant is fitted with Imhoff paddles and diffusers and shows a reduction of 98% in the biochemical O demand. The excess sludge is digested at 29° and produces sufficient gas to operate the entire plant, the cooling water from the gas-electric generator being sufficient to maintain the temp. of the digester. C. JEPSON.

**Separation, digestion, and disposal of sewage solids.** J. R. DOWNES (Sewage Works J., 1931, 3, 237—242).—The successful operation of digestion tanks depends largely on the regularity and uniformity of the supply of fresh solids. A digested sludge containing 4% of solids is recommended as most suitable for

disposal on drainage beds, as it dries in half the time required for a 6% sludge and in one third that needed for an 8% sludge.

C. JEPSON.

#### Handling and disposal of [sewage] screenings.

F. L. FLOOD (Sewage Works J., 1931, 3, 223—231).—The methods adopted at a number of American sewage-disposal plants are described and discussed. Pressing to reduce the moisture content and incineration are recommended, though digestion offers prospects when the method has progressed beyond the experimental stage.

C. JEPSON.

**Determination of dissolved carbon in the analysis of aqueous effluents [from sewage works].** H. BACH [with K. GLASER] (Z. anal. Chem., 1931, 85, 161—170). The  $H_2O$  is boiled with dil.  $H_2SO_4$  to expel  $CO_2$ , cooled, neutralised with NaOH (free from  $Na_2CO_3$ ), and boiled with 15 c.c. of 10% NaOH, 1 g. of  $KMnO_4$ , and 4 drops of 1%  $H_2PtCl_6$  solution under reflux for 1 hr. The liquid is then acidified with  $H_2SO_4$  and the  $CO_2$  formed by oxidation of the dissolved C is expelled by boiling and collected in NaOH solution. The latter is then treated with  $BaCl_2$  at  $100^\circ$  until no further ppt. is formed, then exactly neutralised (phenolphthalein) with HCl, and the undissolved  $BaCO_3$  is determined volumetrically by dissolution in excess of 0.1N-HCl and back-titration with 0.1N-NaOH.

A. R. POWELL.

#### Determination of organic carbon in sewage.

E. V. MILLS (J.S.C.I., 1931, 50, 375—377 T).—A wet-combustion method by chromic acid is described. Inorg. C is first removed by aspiration. The conditions necessary for complete oxidation are indicated; the  $CO_2$  produced is directly determined as  $BaCO_3$ . A correction is applied for the sulphite content of the ppt., but no precise directions can be given for washing the ppt. free from adsorbed alkali.

**Preservation of metal surfaces above and below sewage surfaces.** R. S. RANKIN (Sewage Works J., 1931, 3, 243—245).—Al paint above the sewage level and an asphalt-base paint below are recommended as the final coats over red lead in linseed oil for the preservation of metallic surfaces at sewage works.

C. JEPSON.

**Petroleum extracts of pyrethrum.** H. H. RICHARDSON (J. Econ. Entom., 1931, 24, 763—764).—A petroleum fraction ( $d$  1.5, b.p.  $155$ — $202^\circ$ ) was slower in paralytic action on flies and less toxic finally than a heavier fraction ( $d$  1.39, b.p.  $192$ — $270^\circ$ ). Three intermediate fractions (b.p.  $205$ — $260^\circ$ ) showed little essential difference. The efficiency of extraction of pyrethrum with petroleum varied with the fineness of the powder. Samples passing 15-, 20-, 30-, and 45-mesh gave 75—80% extraction, whereas 200-mesh material showed 90%. No loss of toxicity of the various extracts occurred within 1 year.

A. G. POLLARD.

#### Dephenolisation of coal-tar distillery water.

L. I. KAZATSKHOV (J. Chem. Ind. Russ., 1931, 8, 720—723).—PhOH cannot be quantitatively removed from  $H_2O$  by extraction with paraffin oil, kerosene, benzine,  $C_6H_6$ , sunflower oil, castor oil, fish oil, or oleic acid. The best solvent is castor oil, which extracts 55% of PhOH from a 0.1% and 87% from a 1% solution (1 vol. of solvent : 2 vols. of solution). From 96 to 99% of the

PhOH is extracted by shaking 1 pt. of a mixture of 70% of xylene and 30% of heavy pyridine fraction (containing quinoline etc.) with 5 pts. of a 0.4% solution. The solvent and the PhOH can be regenerated by the action of NaOH. Total removal of PhOH is effected by using activated charcoal. PhOH cannot be removed by aeration at  $80$ — $90^\circ$ .

R. TRUSZKOWSKI.

**Black sulphur-dye waste.**—See IV. **Cyanide.**—See VII. **Action of  $H_2O$  on cement.**—See IX. **Life of water-pipes.**—See X. **Purification of  $H_2O$ .**—See XI.

#### PATENTS.

**Sewage disposal apparatus.** G. R. RODDY, Assr. to CHAIN BELT CO. (U.S.P. 1,795,143, 3.3.31. Appl., 16.8.29).—The continuous removal of sludge from flat-floored settlement tanks is accomplished by wooden scrapers on an endless belt traversing the floor from end to end. A convenient width to be covered by one set of scrapers is 16 feet, and in order to use wide tanks without divisional walls rows of columns are suggested as a means of supporting the scraper mechanism on its return journey. In order to pass these columns and at the same time cover the whole tank floor, the column base is made diamond-shape, scrapers in adjacent rows are staggered, overlapping at the ends, and the ends themselves are hinged, being pressed back when in contact with the column base and straightened again by means of a spring when the pressure is released.

C. JEPSON.

**Treatment for brewing purposes of water containing carbonates.** J. H. COLLETT (B.P. 355,069, 18.9.30).—The water after treatment with  $Ca(OH)_2$  and  $CaCl_2$  is heated to  $71$ — $79^\circ$  and then sufficient  $NaHSO_4$ ,  $KHSO_4$ , or  $H_2SO_4$  is added to reduce the  $p_H$  to  $7.0$ — $7.2$ . After settlement the supernatant water can be withdrawn and used as required.

C. JEPSON.

**Sterilisation of liquids [e.g., water].** G. A. KRAUSE (B.P. 355,086, 30.9.30. Ger., 12.10.29).—The sterilising action of  $Cl_2$  or  $O_3$  is combined with that of the oligodynamic metals (cf. B.P. 279,085; B., 1929, 540) to their mutual advantage. The amount of gas required is reduced, thus preventing the presence of any excess, and the action of the metals is stimulated, with a saving in the time of contact necessary. [Stat. ref.] C. JEPSON.

**Preparation of water-purifying material.** G. BORROWMAN (U.S.P. 1,793,670, 24.2.31. Appl., 14.9.27).—Lignites or brown coal may be substituted for zeolites in softening water by base exchange. The lignite is crushed to a suitable size, washed, treated with brine, and rewashed ready for use.

C. JEPSON.

**Apparatus for impregnating a poisonous gas with warning gas.** Soc. FRANÇ. DU "GAZ SANOS" (B.P. 354,945, 4.7.30. Fr., 24.9.29).—The HCN or other poisonous gas is passed through a tube containing hydrophilous cotton impregnated with, e.g., chloropierin or benzyl bromide, which thus becomes mixed with the gas and conveys warning of its presence. Suitable apparatus is described.

C. JEPSON.

**Clarifying plant. Filtration system. Conditioning air. Colorimeter.**—See I. **Zeolites.**—See VII. **Treating air.**—See XI.



# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 6 and 13, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Determination of solids in steam by conductivity.** J. K. RUMMEL (Ind. Eng. Chem. [Anal.], 1931, 3, 317—320).—The electrical conductivity method of determining solids in steam condensates is discussed and improvements are suggested. A gas separator for removing objectionable gases, mainly CO<sub>2</sub>, from steam samples has been developed. E. S. HEDGES.

**Recent developments in fractional distillation.** C. H. S. EDMONDS (J. Inst. Petroleum Tech., 1931, 17, 425—443).—These have been largely in the direction of the single-flash principle. The modern tube still is fitted with 2 banks of tubes, in the first of which the liquid, e.g., oil, is heated by the conduction and convection from the hot gases, and in the second by radiation from the hottest parts of the furnace walls. Furnace efficiency is obtained by correctly proportioning the radiant and convection heating and maintaining turbulent flow in the tubes. The pressure in the tubes is such as to allow considerable vaporisation to take place so that it is unnecessary to heat the oil beyond the temperature required to produce vaporisation in the flash chamber. Tube sizes vary from 2 in. to 6 in., and with high capacity these may be arranged for parallel flow to reduce the pressure. Modern fractionating equipment consists of large bubble-tray towers from which a number of side streams are taken, these being brought to specification in reboiling or stripping sections with the aid of superheated steam. Automatic temp. control is largely used. Heat economy is obtained by means of modern heat exchangers, which are described. Tube-still units are fitted with both atm. and vac. fractionating equipment. An additional flash tower is also often fitted, in which a residue can be separately vaporised without the vac. loss due to the resistance of the main tower. The performance data of large units are given. T. A. SMITH.

**Analysis of molecular-physical qualities of lubricating oils and their importance in semi-fluid friction.** W. BÜCHE (Petroleum, 1931, 27, 587—601).—The "slippery" quality of various lubricating oils is in direct proportion to their adsorptive power, as measured by the heat of wetting of finely-divided Fe (cf. B., 1925, 435). The dividing line between semi-fluid and fluid friction is ascertained. E. DOCTOR.

**Tunnel kilns.**—See VIII. **Cryoscopy.**—See XIX. **Filtering materials.**—See XXIII.

See also A., Oct., 1143, Precision thermostat (—25° to 500°). Thermocouples. Calorimeter for liquefied gases. 1145, Powder analysis by X-rays.

### PATENTS.

**Melting furnaces.** K. WIRGES (B.P. 356,422, 8.8.30. Ger., 8.8.29).—The furnace comprises a vessel of oval section supported in a cradle of circular section. During melting the vessel or bath is rocked through nearly 90° each way, thus producing a continual expansion and contraction of the surface of the bath; to pour, it is rocked through more than 90°. B. M. VENABLES.

**Obtaining high temperatures in rotary furnaces for fusing minerals or other substances.** H. GARREAU (B.P. 356,184, 1.3.30. Fr., 18.12.29).—The furnace is arranged to be fired with either fat or lean pulverised coal according to the temp., and the air may be preheated either by heat from the furnace or by separate means. B. M. VENABLES.

**Recuperative counterflow furnace.** HEVI DUTY ELECTRIC Co. (B.P. 355,839, 4.6.30. U.S., 5.6.29).—The furnace is of the type in which the goods are preheated in one passage and cooled in an adjacent passage while travelling the reverse way, the actual application of live heat being made in both passages at the return end. In the apparatus described one passage is vertically over the other, transfer of heat being effected by convection currents and of the goods by a lift. B. M. VENABLES.

**Furnace fronts.** J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 356,390, 19.7.30).—An air heater for attachment to a furnace front is described; the temp. of the ingoing air is regulated by varying the proportions heated and unheated. B. M. VENABLES.

**Drying apparatus.** A. G. MURDOCH (B.P. 355,685, 26.5.30).—The apparatus comprises an externally heated drum provided on the interior with a continuous helical blade inclined so as to advance the material. Between the turns of the blade metal plates are fixed, forming scoops which also have an inclination, usually to advance the material. Air, which may be subjected to the heat of the exterior furnace, is drawn through the drum by a fan, and the drum is extended beyond the heated zone in the form of an outlet screen. B. M. VENABLES.

**Machines for drying milk etc.** MILKAL, LTD., and J. M. E. SIERRA (B.P. 355,960, 5.9.30).—In a spray-drying apparatus the hot air is twice passed through perforated diaphragms before being allowed to come in contact with the spray, and when several sprays are used in a common outer chamber each is surrounded by an inner chamber containing the diaphragms. B. M. VENABLES.

**Drying apparatus, suitable for tea etc.** M. J. BALDWIN (B.P. 356,385, 16.7.30).—The apparatus comprises a number of superposed annular sets of

\* The remainder of this set of Abstracts will appear in next week's issue.

trays which are slowly rotated, and when a tray reaches one point of the circle it is tilted and drops its contents on to the next storey. The sets of trays are bounded and supported on the outside by a lagged cylindrical wall and on the inside by a downwardly extending hot-air supply pipe from the mouth of which the air returns upwards through the trays. B. M. VENABLES.

**Heaters suitable for gaseous fluids.** A. FOLLIET and N. SAINDERICHIN (B.P. 356,071, 30.12.30. Fr., 30.12.29).—The gases to be heated are passed through a number of tubes arranged in vertical parallel planes, and the heating gas is supplied through slit-like jets between these planes so that the sheets of flame do not impinge on the tubes; the latter are connected preferably in series-parallel, the tubes of one set (in series) being coplanar and alternately straight and sinuous. B. M. VENABLES.

**Cooling of liquids.** F. B. DENN. From PEVELY DAIRY Co. (B.P. 356,476, 8.9.30).—Devices are described for efficiently spreading the cooling liquid over an apparatus comprising vertical tubes connected by horizontal headers. B. M. VENABLES.

**Machines for grinding and refining paint, cellulose, chocolate, chemicals, etc.** J. MCINTYRE (B.P. 355,648, 25.2.30).—The grinding is effected between a fixed, corrugated, outer casing and mullers which are supported on spring arms. The arms are pivoted on an inner series of pins arranged on a circle and are pressed outwards by an outer ring of pins; both rings rotate together, but relative adjustment between them is produced (through linkwork) by a screw and nut on the end of a shaft within the driving shaft. B. M. VENABLES.

**Conversion of material from pulverulent into noduliform state by addition of humidity.** N. NIELSEN (B.P. 355,825, 30.5.30).— $H_2O$  is added to the material while in a worm conveyor-mixer and the mixing is completed in a revolving drum, preferably arranged as a screen to remove free particles. B. M. VENABLES.

**Mixing apparatus.** P. LENART (B.P. 356,608, 24.12.30. Ger., 29.10.30).—The apparatus comprises a centrifugal impeller running below a disc-shaped guide with entry aperture through the centre, the whole being capable of operation at adjustable levels and of being lifted out of the vessel containing the mixture. B. M. VENABLES.

**Screening and mixing machines or appliances.** F. PARKER, LTD., and F. W. PARKER (B.P. 355,850, 6.6.30).—A method of mounting a mixer and a rotating screen upon the same wheeled carriage is described. B. M. VENABLES.

**Separation of solids from liquids by settling.** ASH Co. (LONDON), LTD., and P. B. SILK (B.P. 356,098, 21.2. and 18.11.30).—Material such as water-borne ashes is collected in an elevated tank from which the water is removed by decantation while the solids are accumulating and afterwards by drainage from several levels. The drained solids are dropped into vehicles, aided if necessary by jets of water or steam. Provision may be made for re-using the water. B. M. VENABLES.

**Treatment of liquids [e.g., resolution of ammonia liquor].** KOPPERS Co., Assees. of M. SHOELD (B.P.

356,116, 28.5.30. U.S., 17.8.29).—A particular constituent is removed from a liquid by repeated stripping with a carrier fluid which, in between each stripping contact, is purified by scrubbing with an absorbent fluid. In the case of removal of  $PhOH$  from  $NH_3$  liquor by means of steam and  $NaOH$  solution, the stripping-scrubbing tower is inserted between the free- and fixed- $NH_3$  stills, the former being operated at a temp. sufficient to remove  $NH_3$ ,  $CO_2$ , and  $H_2S$ , but insufficient to cause appreciable loss of  $PhOH$ . In the tower the flow of steam arising from the  $CaO$  still is upwards through all compartments in series, the  $NH_3$  liquor before liming flows downwards through alternate compartments (flow to adjacent compartments being prevented), and the  $NaOH$  solution is pumped upwards through the other alternate compartments, finishing at the top one, though the flow in each compartment is countercurrent to the gas. The scrubbing liquid would also flow downwards if it were necessary for the carrier fluid to leave in a pure state, but in the case of  $NH_3$  liquor a single flow of steam removes both the fixed  $NH_3$  and  $PhOH$ , the former alone being conveyed away by it. B. M. VENABLES.

**Testing of viscous liquids.** H. A. GILL. From GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 356,011, 20.10.30).—An apparatus for determining the cohesion of liquids comprises an upper vessel from which the liquid is caused to flow vertically downwards through a nozzle under a definite pressure head, through the air, into a lower vessel containing a mobile and transparent liquid in which the point at which the thread of liquid becomes broken may be easily judged. The two vessels are mounted on a mast with scale of distance. B. M. VENABLES.

**Separation of carbonic acid and other vapours of low b.p. from gases.** M. FRÄNKEL (B.P. 355,923, 7.8.30. Ger., 19.8.29).—The gas is chilled by means of cold accumulators and an expansion engine and the frost produced is, on reversal, sublimed into another gas. In the case of removal of  $CO_2$  from blast-furnace gas so that it may be re-used, treatment with C is inefficient because the vol. of the  $CO_2$  reduced is doubled. In this invention the blast-furnace gases are compressed to about 1.5 atm., passed through a cold accumulator, in which  $CO_2$  and  $H_2O$  will be deposited, then expanded, but little because the pressure will be useful when re-used as blast, and finally passed through another cold accumulator in which it is heated and from which it removes moisture derived only from the previous flow of air (cf. *infra*). Meanwhile air is being passed the reverse way through a similar pair of accumulators; it is compressed to 2 atm. and then expanded to 1.1 atm., depositing  $H_2O$  on the entry accumulator and removing  $CO_2$  and  $H_2O$  from the previous flow of gas from the outlet accumulator. The greater range of expansion of the air permits complete sublimation of the frost and makes up the cold losses of the plant. B. M. VENABLES.

**Vaporising apparatus for use with oxygen and other liquefied gases, in particular for the supply of welding and other outfits.** PETITS FILS DE F. DE WENDEL & CIE. (B.P. 356,579, 25.11.30. Fr., 31.1.30).—The liquefied gas is siphoned out of the storage bottle

into a jacket surrounding it and is there heated as required by adjusting the ventilation of a surrounding box.

B. M. VENABLES.

**Removal of strata of adsorption media from adsorption apparatus.** METALLGES. A.-G. (B.P. 356,534, 15.10.30. Ger., 18.10.29).—The lowest, used-up stratum of material is evenly removed through an axial aperture by means of jets of fluid directed along the conical floor of the compartment from the circumference inwards. Distributing devices comprising  $\Lambda$ -shaped or flat rings may also be provided in the lower part of the material.

B. M. VENABLES.

**Evaporators for cooling air in refrigerating chambers or cabinets.** R. SEARLE (B.P. 356,406, 28.7.30).

**[Timing] means for controlling cyclic-operated plant.** TULLY, SONS & Co., LTD., and C. B. TULLY (B.P. 356,825, 17.3.30).

**Rotary retorts.**—See I. Compositions of high thermal resistivity.—See VIII. Tubes for heating liquids etc.—See X. Propagating endothermic reactions.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Bulk density of [crushed] coking coal.** K. LEVEN (Glückauf, 1931, 770; Fuel, 1931, 10, 369—371).—This val. decreases, passes through a min., and then increases with increasing moisture content. Thus for one coal the bulk densities corresponding with 0%, 10%, and 30% of moisture were 858, 721, and 1137 kg./cu. m., respectively. The relative amounts of coal,  $H_2O$ , and air corresponding with any given  $H_2O$  content have been calc.

A. B. MANNING.

**Flotation of coal slurries.** H. A. J. PIETERS (Brennstoff-Chem., 1931, 12, 325—327).—Flotation of fine coal slurries of 20—25% concentration has been successfully effected by means of the following agents: (a) phenols,  $\alpha$ -naphthylamine, pyridine, thiocarbanilide, (b) tar oils, (c) turpentine, (d) petroleum, benzol, (e) various alcohols, (f) various mineral oils, and (g) easily emulsified oils, e.g., turpentine oil. With alcohols the flotation power increases with mol. wt. Water-glass or glycerin, addition of which increases the surface tension, has no action; colloidal solutions, e.g., of saponin or starch, have also no flotation power. Addition of electrolytes, with the exception of alkalis, has little influence on the flotation power of the above agents; addition of alkali greatly diminishes the froth flotation in  $PhOH$  solutions. The efficiency of the process diminishes with increasing concentration of the slurry.

A. B. MANNING.

**Action of heat on a South Wales steam coal.** E. LEWIS (Fuel, 1931, 10, 372—376).—Analyses have been made of the residues obtained when the coal is heated at temps. from 380° to 990°, under a pressure of 20—25 mm. Hg. The results, on the ash-, S-, and N-free basis, when plotted on a C : H diagram, lie on a straight line, inside the coal band (cf. Drakeley, B., 1922, 165 A; Hickling, Trans. Inst. Min. Eng., 1927, 72, 261). It is suggested that coals of the anthracite series have been produced by the action of heat on bituminous coals. The apparent break in the coal band, as plotted by Hick-

ling, at a composition of 86% C may be explained on this hypothesis.

A. B. MANNING.

**Behaviour of solid fuels during oxidation.** VI. Influence on the ignition and combustion properties of the fuel of exposure to oxygen, nitrogen, carbon dioxide, and water. B. MOORE (Fuel, 1931, 10, 344—349; cf. B., 1931, 659).—Exposure of bituminous coal for several hr. at atm. temp. and 650 mm. Hg pressure to air rich in  $O_2$  or  $N_2$  caused no marked change in the ignition and combustion characteristics of the coal. Similar exposure to air rich in  $CO_2$  led to increased reactivity of the coal to  $O_2$ . The normal moisture of air-dried coal is not objectionable in connexion with the ignition and combustion properties of coal, but moisture considerably in excess of the normal content raises the relative ignition temp., reduces the combustible capacity, and decreases temporarily the tendency towards spontaneous ignition.

A. B. MANNING.

**Reactivity of coke.** III. Influence of iron compounds. J. H. JONES, J. G. KING, and F. S. SINNATT (Dept. Sci. Ind. Res., Fuel Res., Tech. Paper, 1930, No. 25, 42 pp.; cf. B., 1929, 630).—Cokes which contain Fe in the form of metal or oxide as a constituent of the ash, or which have been impregnated with  $Fe_2O_3$ , give high values for the initial reactivity ( $R_I$ ) followed by a more or less sharp decrease with continued passage of  $CO_2$  to the final approx. const. reactivity ( $R_{III}$ ), when examined by the Fuel Research method. This catalytic activation by Fe of the reaction between the coke and  $CO_2$  is exhibited only under conditions which can bring about reduction of the Fe compound to the metal, e.g., heating the coke in  $H_2$ , or in  $N_2$  at a sufficiently high temp. With the continued passage of  $CO_2$  over the coke at 950° the Fe is oxidised to  $FeO$ , the catalytic action of which is slight, and the reactivity falls to the  $R_{III}$  val., which is a close approximation to the reactivity of the ash-free coke. A metallurgical coke containing no reducible Fe compound gives a flat curve when reactivity is plotted against vol. of  $CO_2$  passed. The catalytic influence of the Fe can be eliminated by (a) extracting the sol. Fe with mineral acids, (b) adding  $SiO_2$ ,  $Al_2O_3$ , or  $TiO_2$  to the coke, followed by heating in  $N_2$  at 1000°, or (c) treating the coke with  $H_2S$  at 950°. Cokes may contain Fe in a form sol. in acid, but not directly reducible to metal; by treatment of the coke with  $O_2$  at 450° a proportion of the Fe may be converted into the reducible form, thereby bringing about an increase in  $R_I$ . The possible effect of other inorg. constituents of the ash has been studied, but the results show that with a normal coke the preponderating catalytic effect must be ascribed to Fe.

A. B. MANNING.

**Reactivity of coke.** J. H. JONES (Gas World, 1931, 95, Coking Sect., 100—108; cf. preceding abstract).—The greater part of this paper describes work already published. The reactivities of cokes obtained by carbonising blends of a caking and a non-caking coal, both low in ash, are related approx. linearly to the proportion of each coal in the blend.

A. B. MANNING.

**Ermelo torbanite as a source of oils in the Union of S. Africa.** W. BLELOCH (J. Inst. Petroleum Tech.,

1931, 17, 521—540).—The torbanite can be advantageously distilled in a Pumpherston retort with min. decomp. of the volatile constituents and with recovery of the N content as  $\text{NH}_3$ . The crude oil can be refined for use in internal-combustion engines.

C. W. GIBBY.

**Corrosive effect of gasolines and motor benzols on copper.** F. H. GARNER and E. B. EVANS (J. Inst. Petroleum Tech., 1931, 17, 451—463).—Cu sulphides result from the attack of S and compounds containing loosely attached S present in motor fuels. Previous work and the methods of determining the effect of the presence of free S and of mercaptans are reviewed, and a method in which the spirit is heated with 0.5 g. of Cu-bronze powder is described. After  $1\frac{1}{2}$  hours' heating, the Cu-bronze is oxidised with Br and the sulphate precipitated with  $\text{BaCl}_2$ . The quantity of corrosive S was found not to be related to the total quantity of S present. Fuels containing up to 0.5 mg. S per 100 c.c. may be considered non-corrosive. Those containing  $> 1$  mg. are liable to cause serious corrosion. T. A. SMITH.

**Montan wax and montan [wax] size.** J. MARCUSON and P. LEDERER (Chem. Umschau, 1931, 38, 253—255).—Old and recent samples of crude montan wax having similar characteristics (m.p.  $78-82^\circ$ ,  $76-82^\circ$ ; acid. val. 27.8, 31; sap. val. 88.8, 92, respectively) differed in composition—resins 20, 29%; alcohols 17, 6.7%; normal wax acids 49.5, 52.3% (mean mol. wt. 460); oxy-acids 3, 3%; S-containing acids (characteristic of crude montan wax) 6.5, 8%; asphaltic material and humic acids none. A distilled wax contained 56% of wax acids, 28.6% of montanone, and 13.4% of hydrocarbons; it is characterised by a zero acetyl val. of the unsaponifiable matter. Refined montan wax (with  $\text{CrO}_3$  and  $\text{AcOH}$ ) contained Ca soaps, no resin nor S compounds, and only traces of oxy-acids; it yielded 76.2% of fatty acids (mean mol. wt. 347). The unsaponifiable matter had acetyl val. 110 and consisted of higher alcohols (6.7% of the refined wax, distinguishing it from other waxes which contain 50—55% of alcohols). Sized paper may be extracted by  $\text{C}_6\text{H}_6$ -EtOH (8:2); resin is detected in the extract by the Morawski test, and fatty and (oxidised) resin acids are separated by saponification and esterification, acids from montan sizes having mean mol. wt. 399—405. The paper is further extracted with water in order to determine animal size.

E. LEWKOWITSCH.

**Economies in steam consumption [in coke works].** G. J. GREENFIELD (Gas World, 1931, 95, Coking Sect., 113—114).

**Fractional distillation. Friction and lubricating oils.**—See I. Pressure hydrogenation. Solid  $\text{CH}_4$ .—See III. HCN from gases.—See VII. Gas generator in metallurgy.—See X. Source of power alcohol.—See XVIII.

See also A., Oct., 1134, Production of  $\text{H}_2$  by the water-gas reaction.

#### PATENTS.

**Treatment of coal slurry.** B. NORTON (B.P. 354,986, 23.7.30).—Slurry water from the main settling tank of a coal-washing plant is subjected to the action

of vibrating sieves or other device for the recovery of the fine coal slurry contained therein, and is passed thence to another settling tank wherein it is allowed to remain stationary for sufficient time to allow the fine particles to settle. The water is withdrawn from this tank through a discharge opening, e.g., a flexible or hinged pipe, which sinks as the water level falls, and is passed to the sump of a pump by which it is returned to the main settling tank. The deposited material is removed from the secondary settling tank by means of a scraper conveyor.

A. B. MANNING.

**Treatment of coal and cannel for obtaining products therefrom.** A. McCULLOCH and A. ECCLES (B.P. 355,019, 12.8.30).—The coal is treated with  $\text{Cl}_2$  at room temp., placed under a vac. or washed with  $\text{H}_2\text{O}$  to remove the HCl formed, and extracted with an org. solvent, preferably  $\text{CHCl}_3$ . The extracts, amounting to about 30% of an average chlorinated bituminous coal, are resinous materials, whilst the residues yield on carbonisation highly absorbent carbonaceous materials.

A. B. MANNING.

**Rotary retorts [for low-temperature carbonisation].** C. B. WISNER (B.P. 356,136, 27.2.30. U.S., 17.4.29).—In a cylindrical or cylindro-conical, externally-heated retort the heat-transmitting surface is increased at the plastic zone by subdividing the circle into quadrants or sextants having flue spaces in between the radial walls, and the layer of material is caused to be thinner than in the balling zone by the provision of an annular baffle at the outlet and a helical blade which urges forward the material just after leaving the plastic zone. The material present in the plastic zone should be about 5 lb. per sq. ft. of surface, and in the balling zone 10—15 lb. per sq. ft.; the rate of flow of heating gas should be not less than 50 ft. per sec. through a flue space  $2-2\frac{1}{2}$  in. wide. Other suitable dimensions are given.

B. M. VENABLES.

**Removal of carbon deposits from cylinders.** I. MIDDLEY, JUN., and C. A. HOCHWALT, Assrs. to GEN. MOTORS RES. CORP. (U.S.P. 1,786,860, 30.12.30. Appl., 2.2.26).—The cylinders of internal-combustion engines are decarbonised by applying to the metal, heated to above  $65^\circ$ , a high-boiling solvent, e.g.,  $\text{NH}_2\text{Ph}$ ,  $\text{NPhMe}_2$ , pyridine, with or without a low-boiling solvent, e.g., EtOH,  $\text{C}_6\text{H}_6$ . The addition of  $\text{C}_{10}\text{H}_8$  assists the solvent action.

D. K. MOORE.

**Manufacture of carburetted water-gas.** HUMPHREYS & GLASGOW, LTD., Assces. of P. W. JANEWAY, JUN. (B.P. 355,099, 20.10.30. U.S., 21.11.29).—The blow gases produced during the first part of the air-blasting of the fuel bed are burned with secondary air in the carburettor and superheater as in usual practice; during the latter part of the air-blasting, however, high-coke-forming oil is introduced into the top of the fuel bed and the resulting carburetted gases are passed unburnt through the carburettor and superheater to storage. A further quantity of the oil may be supplied to the fuel bed during the subsequent up-run with steam, if desired. It is preferable to follow the up-run with a down-run with steam which has been passed through the carburettor and superheater.

A. B. MANNING.



**Production of gaseous and liquid fuels.** RUHR-CHEMIE A.-G. (B.P. 354,215, 27.1.30. Ger., 26.1.30).—Solid carbonaceous fuels are converted into water-gas which after purification is subjected to a known process for the synthesis of  $C_6H_6$  with simultaneous production of gases containing  $CH_4$ . These are converted into illuminating gas by being heated at least once for a short period at  $>1000^\circ$ , according to B.P. 316,126 (B., 1930, 500), after previous concentration if necessary, with the simultaneous recovery of the aromatic hydrocarbons. H. S. GARLICK.

**Separation and treatment of bituminous sands.** K. A. CLARK, ASSR. to GOVERNORS of UNIVERSITY of ALBERTA (U.S.P. 1,791,797, 10.2.31. Appl., 5.5.28).—The bituminous material is mixed with a reagent giving an alkaline reaction on hydrolysis, *e.g.*,  $Na_2SiO_3$ , and the mixture introduced in small amounts at a time with agitation into a large supply of hot water containing a  $H_2O$ -sol. inorg. compound that precipitates the reagent, *e.g.*,  $CaCl_2$ , and the silt or clay. The separated bitumen is recovered from the surface of the wash water. H. S. GARLICK.

**Apparatus for low-temperature distillation or other heat treatment of bituminous materials.** J. Y. JOHNSON. From E. G. FARBERIND. A.-G. (B.P. 355,020, 12.8.30).—The materials are heated in vertical chambers in the upper parts of which mechanical stirring devices (scrapers, chains, etc.) are provided in order to prevent caking of the charge. They extend at most to a depth where the temp. is not high enough to bring about appreciable corrosion of the metal.

A. B. MANNING.

**[Bituminous] protective coverings for pipes, tubes, etc.** STANDARD OIL CO. OF CALIFORNIA (B.P. 354,803, 17.3.30. U.S., 7.12.29).—The covering comprises a tape bearing a bituminous mastic, which consists essentially of a mineral aggregate and bitumen, the latter amounting to  $< 25\%$  (preferably 10–18%) of the material.

A. B. MANNING.

**Distillation of tar and rectification of the distillate therefrom.** BARRETT CO., ASSEES. of W. McK. BYWATER (B.P. 355,031, 20.8.30. U.S., 31.8.29).—The tar is brought into direct and intimate contact with hot gases, *e.g.*, coal-distillation gases, and the gases and vapours produced are passed through a rectifying column and then through a condenser wherein they are partly cooled by indirect contact with tar or water. The higher-boiling oils which separate are returned to the rectifier and refluxed therein. The gases and uncondensed vapours are passed into another condenser in which the lower-boiling oils are separated. The distillation may be so controlled as to leave a pitch with m.p. at least  $300^\circ$ .

A. B. MANNING.

**Apparatus for cracking oil.** G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,791,617, 10.2.31. Appl., 25.4.21. Renewed 28.9.26).—Oil is passed under superatm. pressure through a cracking coil disposed in a furnace to an enlarged horizontal vapour chamber into which it is introduced from the opposite extremities through pipes of relatively smaller diam. than the main transfer line.

The unvaporised residue is drawn off from the bottom of the chamber, and the vapours are led to a dephlegmator and condenser.

H. S. GARLICK.

**Tank for cracking mineral oil.** A. E. PEW, JUN., and H. THOMAS, ASSRS. to SUN OIL CO. (U.S.P. 1,794,200, 24.2.31. Appl., 26.6.29).—An oil-heating unit comprises a tank with end heads, enclosing the major part of a pipe coil that consists of pipe sections connected by return bends, the pipe sections being welded at one end to an end head and at the other to a wall of a slidable box situated in the tank between the end heads. The return bends are positioned outside the end head at one end and at the other end are contained within the box body, which has a projecting extension secured to the end head and an opening through which access may be had to the interior of the box secured by a removable cover. H. S. GARLICK.

**Cracking of [fuel] oil.** F. C. VAN DE WATER, ASSR. to PETROLEUM LABORATORIES, INC. (U.S.P. 1,788,933, 13.1.31. Appl., 21.11.23. Renewed 17.2.30).—Crude oil is heated in a topping still until all the light fractions have been removed. The heavier vapours are passed at atm. pressure first through narrow superheating coils mounted under the still and maintained at  $535$ – $700^\circ$  and then through larger tubes maintained at the same temp., whereby they become stabilised without appreciable deposition of C.

T. A. SMITH.

**Apparatus for conversion of heavy hydrocarbons into lighter ones.** W. LANDES (U.S.P. 1,792,912, 17.2.31. Appl., 10.7.26. Renewed 15.8.30).—The apparatus consists of an upright chamber leading upwardly from a furnace and through which the products of combustion flow; an upright, elongated expansion chamber, situated centrally within it, has an arm projecting to one side. The expansion chamber has an upper vapour outlet and lower discharge means, and hydrocarbon and steam nozzles extending through the entire length of the arm to direct preheated hydrocarbon and superheated steam against the opposite wall of the expansion chamber.

H. S. GARLICK.

**Separation of volatile impurities from solid or liquid hydrocarbons.** A. ESAU (B.P. 351,576, 5.3.30. Ger., 13.3.29).—The hydrocarbons are subjected to the high-frequency field effect of short electric waves of 100 m. or less (frequencies of  $3 \times 10^6$  Hertz or more), whereby air and  $H_2O$  are removed. The vessel in which the treatment is carried out is supported between two electrodes.

T. A. SMITH.

**Production of low-boiling oils from solid carbonaceous materials.** STANDARD OIL DEVELOPMENT CO., ASSEES. of R. T. HASLAM and P. L. YOUNG (B.P. 352,672, 4.6.30. U.S., 25.6.29).—Coal paste or heavy mineral oil is hydrogenated to produce chiefly middle oil, which is separated, the residues being returned for further hydrogenation. The middle oil is cracked to produce petrol, the pitch and heavy oil produced being again hydrogenated. The original hydrogenation is carried out at  $375$ – $425^\circ/20$  atm., whereby products boiling below  $205^\circ$  are produced only in small quantities. The middle-oil fraction is treated with aq. alkali to recover phenols before cracking.

T. A. SMITH.

**Reactivation of catalyst used in the destructive hydrogenation of hydrocarbon oils.** STANDARD OIL DEVELOPMENT Co., Assecs. of E. B. PECK (B.P. 352,850, 20.10.30. U.S., 13.11.29).—The used catalyst is separated by filtration and then submitted to a prolonged treatment (2—12 hr.) with  $H_2$  at below  $375^\circ$  and at pressures preferably above 50 atm. Pure  $H_2$  need not be used, the gases from the hydrogenation process after scrubbing to remove hydrocarbons being satisfactory. T. A. SMITH.

**Treatment of hydrocarbon oils with liquid sulphur dioxide.** EDELEANU GES.M.B.H. (B.P. 354,582, 2.9.30. Ger., 12.2.30).—The feeding of the ingredients as well as the actual mixing is effected by and in one and the same pump or group of pumps.

H. S. GARLICK.

**Treatment of hydrocarbon oils.** W. M. STRATFORD, ASSR. to TEXAS Co. (U.S.P. 1,792,877, 17.2.31. Appl., 7.6.28).—Cracked naphtha is treated to effect partial separation of unsaturated compounds of substantially the same b.p. by continuously contacting the oil in vapour form with a bed of solid, absorbent, catalyst, maintained under superatm. pressure, and washing the catalyst during contact of the vapours with a solvent for the polymeride formed, *e.g.*, a hydrocarbon boiling within the range of kerosene; the pressure on the catalyst, as well as its temp., is raised as its activity decreases.

H. S. GARLICK.

**Conversion of hydrocarbon oil.** G. EGLOFF and J. C. MORRELL, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,791,618, 10.2.31. Appl., 18.2.22. Renewed 16.10.28).—Oil is raised to cracking temp. in a heating zone, passed to an evaporation zone, and the vapours are led to a dephlegmator. The dephlegmated vapours are passed through a chamber containing catalytic material, *e.g.*, Ni-pumice, and the treated vapours passed to a condenser. The condensate is collected and the uncondensed gases are passed through a desulphuriser and returned to the stream of dephlegmated vapours prior to their treatment with catalyst. H. S. GARLICK.

**Conversion of liquid carbonaceous materials into products of low b.p.** H. C. WADE (B.P. 352,448, 1.3.30. U.S., 22.5.29).—Heated hydrocarbon vapours are rapidly passed through a metallic catalyst (*e.g.*, treated nichrome wire) in the presence of not less than 9 vol.-% of steam. The liquid is vaporised in a spiral mounted in a furnace and then passed through a spiral containing the catalyst. The product is free from S even though the charging stock contained it. [Stat. ref.]

T. A. SMITH.

**Production of lubricating oils from crude oils and the like by treatment with hydrogen at high temperatures and pressures.** STANDARD OIL DEVELOPMENT Co., Assecs. of J. M. JENNINGS (B.P. 352,775, 13.8.30. U.S., 14.8.29).—Heavy hydrocarbon oil and  $H_2$  are passed in opposite directions over catalytic material at  $375$ — $440^\circ/100$  atm., the production of light oil being limited to 15—20%. The temp. of the catalyst is controlled by circulating the oil and gas, these being heated in external coils. The  $H_2$  is purified before its return to the process. The catalyst consists of a mixture of Cr and Mo oxides.

T. A. SMITH.

**Reclamation of asphalt from residuum [from cracking of petroleum products].** R. P. CROTHERS (U.S.P. 1,788,792, 13.1.31. Appl., 27.3.28).—The asphaltic residue containing free C is mixed with fuel oil and allowed to settle while maintained at  $280^\circ$ . The asphaltic oil is then withdrawn and the operation repeated several times. After distilling off the fuel oil a satisfactory asphalt is obtained.

T. A. SMITH.

**Treatment of crude oil (A) having tar and asphalt content, (B) of the Pennsylvania type.** W. S. BAYLIS, ASSR. to FILTROL Co. OF CALIFORNIA (U.S.P. 1,788,653—4, 13.1.31. Appl., 12.11.27).—Mixtures of oil and decolorising clay are passed through a heating coil to a dephlegmator column. The residue containing clay is removed from the bottom of the column and filter-pressed. Cuts free from clay are taken from the upper portion of the column. Heat exchangers are fitted to recover heat from the various fractions. Asphaltic oils are first distilled to remove asphalt before treating the distillate with clay.

T. A. SMITH.

**Making white petrolatum.** H. F. GALLAGHER, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,791,926, 10.2.31. Appl., 27.10.25).—Crude petroleum is blended with heavy asphalt-containing residues and an approx. equal vol. of a heavy-oil distillate, and agitated by air-blowing with oleum, thereby forming a rapidly settling sludge from the asphaltic material, which carries down the less easily settling sludge from the crude petrolatum. The sludges are allowed to settle, withdrawn, and the reaction mixture is successively neutralised, washed with a solvent for sulphonic compounds, *e.g.*, EtOH, steamed, and filtered through a finely-divided decolorising material.

H. S. GARLICK.

**Fuel for internal-combustion engines.** W. HELMORE (B.P. 354,398, 13.2.30).—A non-volatile fuel oil has added to it at least two primers, one of which has a lower spontaneous ignition temp. than the fuel oil, *e.g.*,  $MeNO_3$ , and at least one of which explodes when rapidly heated, *e.g.*,  $EtNO_3$ , polynitro-derivatives such as picric acid, and may have a higher spontaneous ignition temp. than that of the fuel oil. The exploding primer is dissolved in the igniting primer, and the addition made as a solution in fuel oil.

H. S. GARLICK.

**Oil for manufacture of textiles.** L. MELLERSH-JACKSON. From TWITCHELL PROCESS Co. (B.P. 354,297, 3.2.30).—An oil-sol. sulphonated mineral oil (2.5—25%), from which the entrained unsulphonated oil has been substantially removed, is combined with a relatively white mineral lubricating oil (97.5—75%) of low viscosity, to give a product that is readily sol. in  $H_2O$ .

H. S. GARLICK.

**Burners.** R. ROSCH A.-G. (B.P. 356,639, 5.2.31. Ger., 5.2.30).

**[Reservoir casings for oil] filters.** TÉCALEMIT, LTD. (B.P. 356,392, 19.7.30. Fr., 19.7.29).

**Resolution of  $NH_3$  liquor.  $CO_2$  from gases.**—See I. **Oxidation catalyst for hydrocarbons.**—See VII. **Moulding materials etc. from coal.**—See XIII. **C black for rubber. Vulcanisation products.**—See XIV. **Chewing gum.**—See XIX.

## III.—ORGANIC INTERMEDIATES.

**Pressure hydrogenation with iodine as catalyst.** J. VARGA and L. ALMÁSI (Brennstoff-Chem., 1931, 12, 327—329).—PhOH, *p*-cresol, crude oils, tars, and brown coal have been hydrogenated at 450° and under 100—110 atm. initial  $H_2$  pressure, with and without the addition of I (0.5%), in a rotating autoclave. With the exception of the brown coal the hydrogenation of these materials was markedly promoted by the I, or by the  $FeI_2$  formed therefrom. *p*-Cresol was more readily reduced than PhOH and gave  $C_6H_6$  and PhMe, some hydroaromatic and probably also some aliphatic hydrocarbons. The fractions of the crude oils and tars boiling above 300° were converted mainly into benzene. Addition of I was particularly effective in the hydrogenation of bituminous coal tar. In all cases the addition of I brought about a decrease in the PhOH content of the product. The results were difficult to repeat quantitatively because of the variation in the catalytic activity of the walls of the autoclave, due to deposition or removal of a layer of asphalt. A. B. MANNING.

**Solid methane.** J. BRONN (Chem.-Ztg., 1931, 55, 702).—Pure  $CH_4$  can be frozen by the use of liquid  $N_2$  at  $-186^\circ$ , but in the presence of much  $O_2$  or olefines a considerably lower temp. is necessary. The solid cannot be vaporised without melting, owing to its low v.p. A. R. POWELL.

**Determination of water content of glacial acetic acid by thermometric titration.** T. SOMIYA (J. Soc. Chem. Ind., Japan, 1931, 34, 281—282 b).—The AcOH is heated with standard  $Ac_2O$  solution and excess of the latter is determined as described previously (B., 1929, 274). E. H. SHARPLES.

**Determination of aniline content of aniline salts by thermometric titration.** T. SOMIYA (J. Soc. Chem. Ind., Japan, 1931, 34, 279—280 b).— $NH_2Ph$  is determined in its salts by titration with a standard  $Ac_2O$ -AcOH-pyridine solution, using a thermometric measurement for the end-point. E. H. SHARPLES.

**Detection of benzyldenesorbitol.**—See XVIII.

See also A., Oct., 1144. **Condensation.** 1152. **Prep. of *m*-aminocresol.** 1169. **Reaction of diazosulphonates derived from  $\beta$ -naphthol-1-sulphonic acid.**

## PATENTS.

**Production of acetaldehyde from acetylene.** G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,016, 19.3.30).— $B_2O_3$  in combination with a non-volatile acid, e.g.,  $H_3PO_4$ , is used as catalyst at 360° for hydration of  $C_2H_2$  by steam; preferably 100% excess of steam or  $C_2H_2$  is employed. C. HOLLINS.

**Preparation of vinyl esters [from acetylene].** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,318, 27.8.30. U.S., 28.8.29).— $C_2H_2$  is led into AcOH or other lower aliphatic acid below 50° (e.g., 5—15°) in presence of a Hg catalyst and of sulphoacetic acid and  $Ac_2O$ . The product after addition of NaOAc is distilled over NaOAc or  $Ca(OAc)_2$  in vac. The yield with AcOH is 60% of vinyl acetate and 13% of ethylidene diacetate. C. HOLLINS.

**Manufacture of acrylic esters from  $\beta$ -chloropropionic acid esters.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,518, 24.3.30).—A  $\beta$ -chloropropionic ester is heated with conc.  $H_2SO_4$ ,  $H_3PO_4$ ,  $ZnCl_2$ , toluenesulphonic acid, or other acid-reacting compounds containing inorg. acid radicals at 95—240°, whereby HCl is split off. The yields are 75—90%.

C. HOLLINS.

**Production of primary alcohols [by hydrogenation of esters].** H. T. BÖHME A.-G. (B.P. 351,359, 8.10.30. Ger., 25.11.29. Addn. to B.P. 346,237; B., 1931, 834).—The process of the prior patent is applied to the hydrogenation of esters of di- or polyhydric alcohols, especially fatty acid glycerides, e.g., coconut oil.

C. HOLLINS.

**Wetting, penetrating, foaming, and dispersing agents.** H. T. BÖHME A.-G. (B.P. 351,456, 20.3.30. Ger., 3.4.29).—Aliphatic acids above  $C_8$  (e.g., ricinoleic or hydroxystearic acid) or aromatic acids (e.g.,  $\beta$ -naphthoic acid) are sulphonated and esterified with an alkyl ether of a polyhydric alcohol, e.g., glycol mono-methyl or -ethyl ether.

C. HOLLINS.

**Preparation of aliphatic sulphuric acid compounds [wetting and emulsifying agents].** H. T. BÖHME A.-G. (B.P. 351,013, 18.3.30. Ger., 21.3.29).—Sulphonated ricinoleic or other aliphatic acid above  $C_8$  is neutralised and treated with a  $H_2O$ -insol. solvent ( $C_2H_5Cl$ ,  $CCl_4$ ,  $Et_2O$ , liquid hydrocarbons) in presence of enough  $H_2O$  to give 2 layers, in order to extract non-sulphonated products.

C. HOLLINS.

**Cleansing agents.** H. T. BÖHME A.-G. (B.P. 351,403, 18.3.30. Ger., 5.4.29. Addn. to B.P. 318,610; B., 1931, 290).—Sulphonated lauryl alcohol is a more active wetting agent than sulphonated higher or lower alcohols.

C. HOLLINS.

**Preparation of sulphonation products [wetting agents].** H. T. BÖHME A.-G. (B.P. 351,452, 18.3.30. Ger., 20.3.29).—Alcohols derived from the mixed fatty acids of coconut or palm-kernel oil, especially the 50—60% first collected on distillation, are sulphonated under mild conditions to give esters or more vigorously to give sulphonic acids; the latter are stable to hot-drying, calendering, etc.

C. HOLLINS.

**Manufacture of derivatives of quinoline [8-nitro- and -amino-5:6-dialkoxyquinolines].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 351,068, 22.3.30).—The halogen in 5-halogeno-8-nitro-6-alkoxyquinolines is exchanged for an alkoxy group and the product reduced. 5-Chloro-, 5-bromo-, and 5-iodo-8-nitro-6-methoxyquinolines, m.p. 202—203°, 204—205°, and 210—212°, respectively, give with NaOMe in MeOH at 130—140°/4 atm. 8-nitro-5:6-dimethoxyquinoline, m.p. 126—128°, which is reduced to an amine, m.p. 148°. 8-Nitro-6-methoxy-5-isopropoxyquinoline, m.p. 77—78° (amine, m.p. 125—127°), and 8-nitro-5-methoxy-8-ethoxyquinoline, m.p. 86° (amine, m.p. 119°), are also described.

C. HOLLINS.

**Manufacture of phthalic anhydride.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,185, 12.5.30. U.S., 11.5.29).—In the vapour-phase oxidation of  $C_{10}H_8$ , the parts of the reaction vessel in contact with the reactants are made of a metal or alloy which does not form a



stable phthalate or which is not oxidised under the reaction conditions, *e.g.*, Ni, Cr, Ag, Al, calorised Fe, Ni-Cr-Fe, Fe-Si, Cr-W-Co, or stainless steel.

C. HOLLINS.

**Manufacture of styrene and the like.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,310, 19.8.30. U.S., 20.8.29).—PhEt, or other aromatic hydrocarbon with at least one side-chain,  $\text{CH}_2\text{-CH}_2\text{R}$ , is passed as vapour over a dehydrogenating catalyst ( $\text{CeO}_2$  and/or  $\text{ZnO}$ , with promoters if desired) at about  $650^\circ$ . The yield is above 90%.

C. HOLLINS.

**Manufacture of polymerised vinylnaphthalenes and of moulding compositions, varnishes, lacquers, and the like therefrom.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 355,032, 20.8.30. U.S., 21.8.29).— $\alpha$ - or  $\beta$ -Vinylnaphthalene is polymerised either by heating alone to about  $300^\circ$ , or by heating in a solvent, *e.g.*, PhEt, with or without a catalyst, *e.g.*,  $\text{Bz}_2\text{O}_2$ ,  $\text{SnCl}_4$ , to give pale resins suitable for use in lacquers etc.

S. S. WOOLF.

**Antiseptics.**—See XXIII.

#### IV.—DYESTUFFS.

See A., Oct., 1163, Thionaphthen dyes. 1170, Thiohydantoin dyes.

##### PATENTS.

**Manufacture of vat dye preparations for cotton printing.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 350,936, 17.3.30).—Aminoanthraquinones are used in place of the hydroxyanthraquinones of B.P. 349,955 (B., 1931, 969). [Stat. ref.]

C. HOLLINS.

**Manufacture of dyes and dyeing of cellulose esters or ethers.** J. Y. JOHNSON. From I. G. FARBENIND, A.-G. (B.P. 350,928, 17.2.30).—Aminoanthraquinonecarboxylic amides containing a second auxochromic group and, if desired, a further  $\text{NH}_2$  group are synthesised; they give deep shades on acetate silk etc. Examples are: 1-aminoanthraquinone-2-carboxylamide, m.p.  $288\text{--}289^\circ$ , condensed with  $\text{CH}_2\text{O}$  in conc.  $\text{H}_2\text{SO}_4$ , nitrated, and the  $\text{NO}_2$ -compound, m.p.  $285\text{--}289^\circ$ , reduced to 1:4-diaminoanthraquinone-2-carboxylamide (blue), also obtainable from 4-nitro-1-aminoanthraquinone-2-carboxyl chloride, m.p.  $238\text{--}239^\circ$ , or from 1-amino-4-*p*-toluenesulphonamidoanthraquinone-2-carboxyl chloride; 1-methylaminoanthraquinone-2-carboxylamide (greenish-blue) by hydrolysis of the nitrile; 4-nitro-1-hydroxyanthraquinone-2-carboxylamide (from the acid chloride, m.p.  $227\text{--}228^\circ$ ) reduced to the 4-amino-amide (violet), m.p.  $287^\circ$ ; 1:4-diaminoanthraquinone-2-carboxyl-methylamide (reddish-blue) and -anilide (violet) from 1-amino-4-*p*-toluenesulphonamidoanthraquinonecarboxyl chloride.

C. HOLLINS.

**Manufacture of vat dyes of the 1:2:2':1'-anthraquinoneazine [indanthrone] series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,032, 21.2.30).—Indanthrones are oxidised to the corresponding azines and condensed with  $\text{CH}_2\text{O}$ , and, if desired, halogenated, greener shades being produced. C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,557, 28.3.30).—Bisanthraquinone- $\beta$ -

carboxyl derivatives of *p*-arylenediamines or 4:4'-diaminodiarlyls are converted by way of the imide chlorides into bisthiazoles by the action of sulphurising agents (S in  $\text{C}_{10}\text{H}_8$ ). Examples are vat dyes from the di-imide dichlorides of dianthraquinone- $\beta$ -carboxylated benzidine (dichloride, m.p.  $322^\circ$ ; yellow vat dye) and *p*-phenylenediamine.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** I. G. FARBENIND, A.-G. (B.P. 351,774, 22.7.30. Ger., 30.7.29).—Bisthioxanthenes of the type  $\text{Aq} < \text{CO} > \text{Ar} < \text{CO} > \text{Aq}$  are synthesised from bis-(2-carboxy-1-anthraquinonylthiol)-benzenes or -naphthalene by the action of  $\text{PCl}_5$ ,  $\text{Ac}_2\text{O}$ ,  $\text{H}_2\text{SO}_4$ , etc. Examples are the bisthioxanthenes from the bis-2-carboxy-1-anthraquinonyl derivatives of 1:6-, 2:6-, 2:7-dithiolnaphthalenes, and *m*-dithiolbenzene; the products are orange vat dyes.

C. HOLLINS.

**Manufacture of an azo dye [pigment and ice colour].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 351,585, 2.4.30).—4:6-Dichloro-*m*-toluidine is diazotised and coupled in substance or on the fibre with 2:3-hydroxynaphthoic 3-chloro-2:4-dimethoxyanilide to give Turkey-red shades of good fastness. C. HOLLINS.

**Manufacture of [dis]azo dyes.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 351,322, 29.8.30. U.S., 30.8.29).—An aminodisazo compound, having at least 2  $\text{SO}_3\text{H}$  and/or  $\text{CO}_2\text{H}$  groups, but no OH or additional  $\text{NH}_2$  group, is condensed with a nitroaroyl halide, reduced, condensed again with nitroaroyl halide, and finally reduced. Examples are: 2:6:8-naphthylaminedisulphonic acid  $\rightarrow$  *m*-toluidine  $\rightarrow$  *m*-toluidine, and metanilic acid  $\rightarrow$  Cleve acid  $\rightarrow$  *m*-toluidine, *p*-nitrobenzoyl chloride being used in both cases. The products give yellow and orange-brown shades, respectively, on cotton, wool, silk, or viscose silk, becoming redder on development with  $\beta$ -naphthol, yellower with phenylmethylpyrazolone. C. HOLLINS.

**Manufacture of a vat dye [of the perylene series].** F. BENSA (B.P. 351,220, 6.6.30. Austr., 16.7.29).—3:9-Dibenzoylperylene is electrolytically oxidised, *e.g.*, in conc.  $\text{H}_2\text{SO}_4$ , to give a violet vat dye. C. HOLLINS.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Estimation of moisture content in fabrics by cobalt chloride test paper.** K. YAMADA, II. OTSUKA, T. NAKAMURA, and F. TATEBE (J. Soc. Chem. Ind., Japan, 1931, 34, 135—136 B).—The duration of colour change in Co chloride paper is used as a measure of moisture content of fabrics, an accuracy of 3% being claimed.

T. T. POTTS.

**Wood chemistry. VII. Digestion experiments on woods of "Doronoki" and "Ezoyanagi."** Y. UYEDA and G. MIYASHITA (J. Cellulose Inst., Tokyo, 1931, 7, 131—132).—The results of experimental digestions of the two new pulp woods already described (B., 1931, 436) are given. The sulphite process yields a better pulp than does the soda process.

T. T. POTTS.

**Production of cellulose from pine wood by the sulphite process.** O. ROUTALA and J. SEVÓN (Suomen Kem., 1931, 4, 45—50).—Sulphite pulp is prepared

from wood of high resin content (*e.g.*, northern pine wood and hemlock) by treating with ordinary bisulphite liquors containing added org. acids and Na<sup>+</sup> effected by adding 25% of a solution prepared by precipitation of Ca from sulphite waste-liquor by means of Na<sub>2</sub>SO<sub>4</sub>. Wood from very old pine trees (resin 12%, H<sub>2</sub>O 53.5%) yields pulp of good quality but slightly yellowish colour.

T. T. POTTS.

**Determination of lignin in wood pulp.** A. NOLL (Zellstoff u. Papier, 1931, 11, 409—411).—Methods based on the hydrolysis of cellulose by means of 72% H<sub>2</sub>SO<sub>4</sub> have been found most suitable, and a rapid modification of the method is worked out. 3 g. of bleached pulp or 1 g. of unbleached, in the form of finely rasped powder, are treated with 5 c.c. of dimethylaniline followed by 25 c.c. of 78% H<sub>2</sub>SO<sub>4</sub>. Hydrolysis commences immediately, and the progress of the decomp. may be determined by testing a control solution for dextrin. When dextrin is completely hydrolysed the reaction mixture is poured into 200 c.c. of hot H<sub>2</sub>O, boiled for 5 min., and the floccular lignin ppt. collected on a filter. The resulting lignin is found to retain the skeletal form of the original fibre and gives no cellulose reaction. Examination in ultra-violet light in conjunction with chemical analysis shows that the lignin is free from Cl, N, and S. Resin is shown to have no effect on the efficiency of the method.

T. T. POTTS.

**Chemical investigations of bamboo. III. Pentosans of bamboo.** S. OGURI (J. Soc. Chem. Ind., Japan, 1931, 34, 233—235 B; cf. B., 1931, 479).—Xylan is the principal pentosan of Japanese bamboo, araban being present in traces only.

T. T. POTTS.

**Hygroscopic moisture of cellulose. III.** S. OGURI and S. TERUI (J. Soc. Chem. Ind., Japan, 1931, 34, 229—233 B; cf. B., 1931, 836).—The moisture content of various forms of cellulose in equilibrium with atms. of different R.H. has been determined and isothermals are plotted. Isohygrometric curves are given, with formulæ for the calculation of moisture content for several materials from the R.H. based on Langmuir's equation.

T. T. POTTS.

**Theory of rosin sizing [of paper].** K. KANAMARU (J. Soc. Chem. Ind., Japan, 1931, 34, 78—87 B).—Measurements of the electrokinetic ( $\zeta$ ) potentials at the interfaces of cellulose, rosin, and Al(OH)<sub>3</sub> have been made by the streaming-potential method for varying conditions of sizing. The addition of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> to pulp and rosin soap solution causes precipitation of Al(OH)<sub>3</sub> with an increase in [H<sup>+</sup>], rosin particles being flocculated and the complex, Al(OH)<sub>3</sub> + rosin, being formed. Increase in the concentration of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and of H<sup>+</sup> decreases the negative charge on the rosin particles and increases the positive charge on the Al(OH)<sub>3</sub>, the above complex becoming more positive. Decrease in the  $\zeta$ -potential follows increased concentration of Al<sup>+++</sup>. Ideal sizing conditions are determined by adjustment of the relative  $\zeta$ -potentials consequent on the addition of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> in such amount as will neutralise the negative charge on the cellulose and the positive charge on the Al(OH)<sub>3</sub> + rosin complex. The results of a number of measurements of potential and conductivity are given.

T. T. POTTS.

**Sizing process using electropositive fibres.** K. KANAMARU (J. Soc. Chem. Ind., Japan, 1931, 34, 123—129 B).—The process arises out of the author's electrical theory of sizing (*cf.* previous abstract). Electropositive fibres are prepared by the deposition of Al(OH)<sub>3</sub>,  $\zeta$ -potentials are measured before and after the addition of rosin soap, and optimum sizing conditions attained as already described (*loc. cit.*)

T. T. POTTS.

**Montan wax size.**—See II. NaOH from cellulose liquors.—See VII. Latent images on paper.—See XXI.

See also A., Oct., 1124, Structure-viscosity of cellulose derivatives. 1149, Action of SO<sub>2</sub> on lignin. 1198, Hemicelluloses of box-wood.

#### PATENTS.

**Production of spinnable fibre from flax or other straws.** J. WINDRUM (B.P. 355,915, 31.7.30).—The straws pass through a series of pairs of fluted rollers which separate the wood from the fibre, thence, if desired, through liquids such as NaOH, NaOCl, H<sub>2</sub>O, etc., and afterwards between a pair of toothed or spiked cylinders for separating adhering fibres from one another. The fibres are then delivered in spinnable length as an uncarded sliver, the seeds and bolls being collected from the fluted rollers and the fibre, pectic matter, and woody dust from the toothed cylinders.

F. R. ENNOS.

**[Accelerators for acid] felting of wool.** H. T. BÖHME A.-G. (B.P. 354,851, 20.5.30. Addn. to B.P. 318,610; B., 1931, 290).—A sulphonation product of the higher aliphatic alcohols (C<sub>9</sub> or above), *e.g.*, of lauryl or stearyl alcohol, is added to the acid liquor.

A. J. HALL.

**Production of materials having the appearance of textile fabrics.** BRIT. CELANESE, LTD. (B.P. 355,860, 16.6.30. U.S., 15.6.29).—Two or more layers of fabric composed of org. derivatives of cellulose are superimposed and subjected to heat and pressure under such conditions that they coalesce to give a compound fabric retaining on one or both sides the woven texture of the original material.

D. J. NORMAN.

**Manufacture of cellulose derivatives.** U.S. INDUSTRIAL ALCOHOL Co. (B.P. 355,382, 29.5.30. U.S., 12.6.29).—The reaction temp. is controlled by conducting the reaction in a closed vessel and incorporating with the reaction mixture a volatile liquid, particularly liquid SO<sub>2</sub>, the regulated volatilisation of which is used to absorb heat. Thus by using a closed reaction vessel fitted with a reflux condenser cooled from a refrigerating plant, and also a heating coil, it is possible by using a thermostat to maintain a const. reaction temp. or to vary the temp. in any predetermined manner.

D. J. NORMAN.

**Manufacture of cellulose esters.** BRIT. CELANESE, LTD. (B.P. 355,690, 26.5.30. U.S., 25.5.29).—Org. esters of cellulose prepared with H<sub>2</sub>SO<sub>4</sub> or other S-containing catalyst are freed from combined S by dissolving them in, *e.g.*, AcOH and adding to the solution a metal salt, *e.g.*, acetate of Ba, Sr, or Pb, which will react with the S compounds to give an insol. compound. Cellulose

acetate thus purified has a heat test about 70° higher than the untreated acetate. D. J. NORMAN.

**Manufacture of cellulose esters.** KODAK, LTD., Assecs. of C. J. MALM (B.P. 356,012, 20.10.30. U.S., 22.10.29).—The back-hydrolysis of cellulose triacetate to a product sol. in  $\text{COMe}_2$  is effected by adding to the final reaction mixture, which preferably contains  $\text{C}_2\text{H}_4\text{Cl}_2$  as diluent (cf. B.P. 351,118; B., 1931, 877), enough EtOH (or MeOH) to convert the whole of the  $\text{Ac}_2\text{O}$  and AcOH into EtOAc (the latter reaction providing the  $\text{H}_2\text{O}$  necessary for the hydrolysis of the cellulose triacetate) and leave a sufficient excess of uncombined EtOH to form with the  $\text{C}_2\text{H}_4\text{Cl}_2$  a solvent mixture in which the cellulose acetate remains dissolved. If  $\text{C}_2\text{H}_4\text{Cl}_2$  is not used in the acetylation bath a suitable quantity must be added subsequently with the EtOH.

D. J. NORMAN.

**Altering the solubility of cellulose acetates.** C. F. BOHRINGER & SOHME, G.M.B.H. (B.P. 355,419, 21.6.30. Austr., 21.6.29).—The  $\text{CHCl}_3$ -sol. triacetate is converted into the  $\text{COMe}_2$ -sol. acetate by treatment with comparatively conc. inorg. acids (about 60% anhyd. acid) in the presence of salts or esters which are sol. in the acid, but have no solvent action on cellulose acetate. These salts, the function of which is to retard the solvent and degrading action of the acid, are used in quantities approaching saturation point. Suitable solutions include a saturated solution of  $\text{KNO}_3$  in 80%  $\text{HNO}_3$ , and a mixture of 10 kg. of 96–97%  $\text{H}_3\text{PO}_4$  with 9 kg. of cyclohexyl acetate.

D. J. NORMAN.

**Manufacture of fatty acid esters of cellulose or of derivatives thereof.** I. G. FARBENIND. A.-G. (B.P. 356,073, 31.12.30. Ger., 31.12.29).—In the two-stage esterification process of B.P. 281,927 (B., 1928, 258) difficulty is experienced in controlling the temp. in the second stage. This is obviated by using in this stage normal or acid salts or esters of  $\text{H}_2\text{SO}_4$ , with or without addition of free  $\text{H}_2\text{SO}_4$ , e.g.,  $\text{Na}_2\text{SO}_4$ ,  $\text{NaHSO}_4$ , pyridine sulphate,  $\text{Me}_2\text{SO}_4$ , etc. The salt may be formed *in situ* by adding an AcOH solution of NaOAc to the reaction mixture and then sufficient  $\text{H}_2\text{SO}_4$  dissolved in AcOH to give, e.g., 6% of  $\text{NaHSO}_4$  and 1.5% of  $\text{H}_2\text{SO}_4$  on the wt. of original cellulose.

D. J. NORMAN.

**Manufacture of cellulose ethers.** A. LIEBSTER (B.P. 355,563, 6.10.30. Ger., 10.10.29).—Raw cellulosic material, which has been predigested with dil. alkali, washed with  $\text{H}_2\text{O}$  until neutral, and dried, is treated with a slight excess of conc. alkali and the resulting unripened alkali cellulose is etherified with rather more than the theoretical amount of an inorg. acid ester ( $\text{EtCl}$ ).

F. R. ENNOS.

**Manufacture of nitrocellulose.** H. C. HEIDE. From E. TSCHUDIN (B.P. 355,492, 11.8.30).—Wood pulp, after reducing to a fineness not exceeding 80 Schopper, is treated with small quantities of NaOH and  $\text{CS}_2$ , precipitated in acidified  $\text{H}_2\text{O}$ , dried, and nitrated with approx. equal pts. of conc.  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ .

F. R. ENNOS.

**Treatment [saccharification] of cellulosic materials.** M. JUNIEN (B.P. 354,472, 16.6.30. Fr., 14.6.29).

—The max. yield of fermentable products and volatile by-products is obtained if free  $\text{O}_2$  is removed from the material (sawdust) by heating with  $\text{H}_2\text{O}$  under reduced pressure before introducing the dil.  $\text{H}_2\text{SO}_4$ , and the hydrolysis is then conducted in the presence of excess of  $\text{H}_2\text{O}$  at a progressively rising temp. (using indirect heat) in an autoclave designed to ensure rapid circulation of the liquor. The volatile by-products obtained during hydrolysis together with those subsequently obtained by distillation of the residual lignin with dil.  $\text{H}_2\text{SO}_4$  are neutralised and condensed in multiple-effect heat exchangers to recover the heat. D. J. NORMAN.

**Coated flexible materials.** BAKELITE CORP., Assecs. of H. L. BENDER (B.P. 355,318, 12.5.30. U.S., 17.5.29).—Cellophane or other smooth-surfaced base is coated with a solution of a flexible resinoid, e.g., an EtOH or  $\text{COMe}_2$  solution of a phenol-tung oil-methylene resin. Other resinoids, e.g., of the polybasic acid-polyhydric alcohol type or PhOH-urea type, may also be used in conjunction with plasticising agents. The resulting coating adheres firmly, has a glossy surface, is resistant to moisture and heat, and gives on a viscose base a product of high dielectric strength (2000–4000 volts per mil).

D. J. NORMAN.

**Manufacture of [rubbered] paper.** R. & W. WATSON, LTD., J. H. WATSON, and H. E. ANDERSON (B.P. 355,470, 25.7.30).—A sheet of rubber is interposed between two webs of paper, immediately after the latter have been “made” on the wire and before couching; the whole is then pressed and dried between heated rollers.

F. R. ENNOS.

**Cleaning and polishing material [in sheet form].** E. E. STRAWN (B.P. 355,323, 20.5.30).—Soft, porous, unsized paper (“water leaf”) is impregnated with a soap emulsion of paraffin,  $\text{H}_2\text{O}$ , and abrasive (e.g.,  $\text{CaCO}_3$ ).

F. R. ENNOS.

**Production of paper and rubber and similar combinations.** L. MELLERSH-JACKSON. From GEN. RUBBER Co. (B.P. 356,310, 11.6.30).—See U.S.P. 1,773,201; B., 1931, 407.

**Washing machine for spun cakes of artificial silk.** BRIT. BENBERG, LTD. (B.P. 356,415, 31.7.30. Ger., 5.8.29).

**Grinding cellulose. Testing viscous liquids.**—See I. NaOH from wood-pulp liquors etc.—See VII. Films for glass surfaces. Abrasive implements.—See VIII. Insulating materials.—See XI. Cellulose ester enamels. Impregnated products from coal.—See XIII. Recovery of waste materials [cotton]. Reclaiming rubber-fibre products.—See XIV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Kiering of vegetable [cotton] fibres.** G. ULLMANN (Textilber., 1931, 12, 577–581).—A superior white and a more complete removal of the impurities in cotton materials are obtained by kiering with the hydroxides of metals such as Al, Ca, Sr, and Ba, than with NaOH; even better results are obtained by kiering with both of these classes of alkali present, this being possible if a suitable dispersing agent (e.g., a sulphonate of an

aliphatic alcohol) is added to the kier liquor. Excellent results are secured by kieren, with or without pressure, with NaOH and Sirial (Böhme A.-G.; a combination of  $\text{Al}_2\text{O}_3$  and an aliphatic alcohol sulphonate capable of dispersing or rendering  $\text{H}_2\text{O}$ -sol. the soaps of various metals) since under these conditions the natural cotton fats and waxes are rapidly saponified. Under comparable conditions NaOH,  $\text{Ca}(\text{OH})_2$ , and  $\text{NaOH} + \text{Ca}(\text{OH})_2$  and Sirial saponified 13.5, 21.9, and 33.9%, respectively, of olive oil. The saponifying action of NaOH on olive oil was increased in the ratio 1.45 : 2.06 : 2.65 by adding equiv. amounts of  $\text{Na}_3\text{AlO}_3$  and Sirial, respectively. The superior whiteness of cotton kieren with Sirial is due to more efficient removal of pectin impurities by  $\text{Al}(\text{OH})_3$  than by NaOH.

A. J. HALL.

**Partial alkaline hydrolysis of [cellulose] acetate silk preparatory to dyeing.** W. COLTOF, H. I. WATERMAN, and I. G. WOLF (Z. angew. Chem., 1931, 44, 163—168).—Cellulose acetate silk hydrolysed by immersion in dil. NaOH containing NaOAc as a levelling agent acquires an affinity for Benzopurpurin 4B which is a max. when the resulting loss of wt. is about 1.7%. Cross-sections of partly hydrolysed fibres dyed with Benzopurpurin 4B and Cellit Fast Red (dyes having affinities for cellulose and cellulose acetate, respectively) show that the degree of hydrolysis increases from the surface to the centre of each fibre. From an examination of the  $\text{COMe}_2$ -sol. portion of partly hydrolysed fibres, lower acetates than cellulose triacetate were definitely shown to be present. It is concluded that from the surface to the centre of each fibre the OAc content of the cellulose acetate present steadily increases, except in highly saponified fibres where an outer layer of pure cellulose exists. The degree of hydrolysis resulting from treatment in NaOH solutions increases with the concentration of the alkali and the temp. of the solution, but the lower is the concentration, the less the proportion of alkali which reacts.

A. J. HALL.

**Two-colour effects on mixtures containing viscose silk.** S. R. TROTMAN and J. E. BATEMAN (J. Soc. Dyers and Col., 1931, 47, 231—232).—Viscose silk has a marked affinity for basic dyes which is not affected by the presence of org. acids, whereas wool has little affinity for such dyes in a bath having  $p_{\text{H}} > 5.5$ ; it is thus possible to dye wool and viscose silk fibres different colours simultaneously in a bath containing acid and basic dyes and having  $p_{\text{H}} > 5.5$ . Suitable acid dyes include most of the Lissamine, Kiton, and Xylene colours, and Supranol Brilliant Red B, Fast Acid Violet 2RN, Brilliant Geranine B, Alizarine Direct Grey, and Azo Rubinole; Rhodamine 3B, Brilliant Phosphine G, Malachite Green, Chrysoidine R, Methylene Blue, and Methyl Violet are satisfactory basic dyes. Dyeing is effected at 80° in a solution of tartaric acid (1 g./litre) containing Glauber salts (10%) and the acid dye; after the wool is dyed the bath is cooled to 60°, a basic dye added, and the cotton dyed. A similar but less satisfactory result can be obtained with unbleached cotton and viscose mixtures, the natural proteins in the cotton giving this fibre dyeing properties similar to those of wool.

A. J. HALL.

## Measurement of the colour of dyed textiles.

**VIII. Numerical expression and calculation of fading.** P. W. CUNLIFFE and P. N. LAMBERT (J. Soc. Dyers and Col., 1931, 47, 225—231; cf. B., 1931, 583).—It is not satisfactory to measure the fading of dyed materials in terms of the surface concentration of dye, since the % loss of dye after a given exposure is approx. the same for different original concentrations of the same dye, whereas, to the eye, the lighter shades fade more than the darker shades. Two methods based on pure colorimetry (the Guild trichromatic colorimeter) are described for calculating the amount of fading, and are illustrated by reference to the fading of several dyes as expressed by numerous colour charts. In the first method only the colorimetric saturation (saturation being a physical quantity proportional to the ratio of the amount of homogeneous light of a certain wavelength to that of the total light in a mixture of the homogeneous and white light which matches the colour measured) as measured by the distance of a point on the colour chart from white or undyed fabric is used, whilst in the second this quantity is combined with the brightness factor (luminosity). The results obtained by both methods on the fading of different colours on mercerised cotton agree well with visual determinations.

A. J. HALL.

**Chemistry of desizing [textiles].** M. M. TSCHILIKIN (Textilber., 1931, 12, 29—34).—Quant. experimental data are given concerning the hydrolysis of starch by treatment with cold and hot acids ( $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ ) and with starch-liquefying enzymes (e.g., Diastafar and Biolase), and the removal of starch sizes from sized cotton by acid, alkaline, and enzyme treatments. The removal of starch from sized fibres by treatment with 1—3%  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$  solutions is only partly due to solubilisation of the starch by hydrolysis; the greater part is removed mechanically due to loss of adhesion. Starch removal is most efficiently effected by means of enzymes.

A. J. HALL.

**Montan wax size.**—See II. **NaOH recovery from cellulose liquors.**—See VII. **Dry-cleaning soaps.**—See XII. **Mn in water [for bleaching].**—See XXIII.

See also A., Oct., 1173, **Reactions of silk fibroin with  $\text{SnCl}_4$  solution.**

## PATENTS.

**Dyeing of wool.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,512, 25.4.30).—Wool goods are dyed evenly with badly levelling dyes at 50—70°, or other temp. not more than 20° below the b.p. of the bath, provided that up to 30% of substances having a strong levelling action (e.g., non-dyeing sulphonation products of org. compounds containing at least 8 C atoms in the mol., i.e., sulphonic acids or sulphuric esters are added in addition to the usual easily  $\text{H}_2\text{O}$ -sol. strongly acid agents.

A. J. HALL.

**Dyeing [animal fibres] with vat dyes.** J. MORTON, J. E. G. HARRIS, and MORTON SUNDOWN FABRICS, LTD. (B.P. 354,777, 10.3.30).—Vat dye liquors are made suitable for dyeing animal fibres, without adversely affecting their properties, by the addition of acidic substances such as resorcinol,  $\text{PhOH}$ , and quinol (but not  $\text{H}_3\text{BO}_3$ ;

cf. B.P. 340,267; B., 1931, 345), which have a dissociation const. of  $10^{-8}$  to  $10^{-13}$ , and a buffering action similar to that of  $H_3BO_3$ , so that the alkalinity of the dye liquor does not exceed  $pH$  8. A. J. HALL.

**Dyeing of animal fibres.** H. T. BUCHERER (H. BUCHERER) (B.P. 355,727, 24.4.30. Ger., 24.4.29).—Indigoid, vat, sulphur, and mordant dyes, and other acid dyes of the azine, oxazine, thiazine, and triphenylmethane series are employed from a bath containing  $NH_3$  [or  $(NH_4)_2CO_3$ ] and  $Na_2S_2O_4$ , but without addition of soaps (cf. B.P. 274,550; B., 1927, 840). Many mordant (*e.g.*, alizarin and galloxyaniline) and acid dyes have an increased affinity for animal fibres when in the form of their leuco-compounds. A. J. HALL.

**Dyeing of [cellulose] acetate silk [black].** I. G. FARBENIND. A.-G. (B.P. 353,592, 23.5.30. Ger., 23.5.29).—Black shades which give clear white discharges with Zn formaldehydesulphoxylate are obtained by treating with a dye of the type (4)  $H_2N \cdot R \cdot N \cdot N' \cdot R' \cdot NX_2$  (4'), where R and R' are phenylene residues which may contain halogen or alkyl substituents, and  $X_2$  is an aliphatic residue, *e.g.*, MeEt,  $(CH_2 \cdot CH_2 \cdot OH)_2$ , then diazotising on the fibre, and coupling with  $\beta$ -naphthol-3-carboxylic acid. Yellowish, but not white, discharges are obtained if both components are applied simultaneously to the silk and the black shade is then developed by treatment with  $HNO_2$ . A. J. HALL.

**Manufacture of fast dyeings and prints with vat dyes.** DURAND & HUGUENIN SOC. ANON. (B.P. 356,577, 25.11.30. Ger., 25.11.29. Addn. to B.P. 220,964; B., 1925, 879).—In the development of dyeings and printings obtained with ester salts of leuco-vat dyes,  $Al(ClO_3)_3$  alone is used instead of a mixture of an oxidising agent, a catalyst, and an acid-yielding substance as previously required. A. J. HALL.

**[Production of] crêpe fabrics.** BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 356,233, 3.5.30).—Crêpe fabrics, particularly those containing cellulose ester or ether yarns, are produced by weaving, in warp and/or weft, tightly twisted yarns which have been previously impregnated with an emulsion of a swelling or gelatinising agent (*e.g.*, BzOH for cellulose acetate yarns) and dried, and then exposing the fabric to conditions (*e.g.*, scouring or steaming) which promote the swelling action (cf. B.P. 351,999 and 352,000; B., 1931, 880). Brocade effects are obtained by printing fabrics with swelling agents and facilitating their action similarly. A. J. HALL.

**[Wetting out agents for] mercerising liquors.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 354,946, 5.7.30).—To each litre of the liquor 1–20 g. of the sulphuric esters of low-mol. aliphatic alcohols ( $C_4$  to  $C_8$ ) or their  $H_2O$ -sol. salts, especially alkali-metal salts, are added, their dissolution being assisted by the further addition of org. solvents, *e.g.*, mono-ethyl or -butyl ethers of ethylene glycol, phenols, and Turkey-red oil. A. J. HALL.

**Treatment of textile materials containing organic derivatives of cellulose.** BRIT. CELANESE, LTD. (B.P. 355,466, 18.7.30. U.S., 30.7.29).—Cellulose acetate etc. materials are delustred by impregnation or

printing with aq. triethanolamine (or di- or mono-ethanolamine) followed by steaming or other treatment with a hot aq. medium. A. J. HALL.

**Treatment [weighting] of textile materials.** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 355,331, 21.5.30. Addn. to B.P. 329,659; B., 1930, 815).—The prior process is modified by supplying the metallic radical of the insol. weighting substance from a neutral or acid bath instead of by mechanical impregnation. A. J. HALL.

**Sizing of textiles.** J. M. LYONNET (B.P. 354,819, 15.5.30. Fr., 15.5.29).—The yarn is sized in a solution of 0.5–2% of rubber dissolved in  $C_6H_6$  or  $CS_2$ , with or without the addition of EtOH or petroleum; the size can be completely removed by scouring with "shop" liquor made slightly alkaline or ammoniacal. H. ROYAL-DAWSON.

**Dyeing machines.** TURNER, ATHERTON & CO., LTD., and G. ATHERTON (B.P. 356,860, 10.7.30).

**Open-width dyeing machines.** H. W. METTLER (B.P. 356,470, 3.9.30. Switz., 21.11.29).

**Washing, drying, and cleaning apparatus [for clothes].** K. W. SKÖLD (B.P. 356,560, 3.11.30).

**Textile oil.**—See II. Wetting agents.—See III. Dyeing of cellulose esters etc. Cotton printing.—See IV. Rubber cement.—See XIV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Vanadium catalyst processes.** B. WAESER (Brennstoff-Chem., 1931, 12, 335–337).—Recent developments in the application of V catalysts, especially in the  $H_2SO_4$  industry, are briefly discussed. A. B. MANNING.

**Production of hydrogen cyanide from technical gases.** H. KÜSTER (Brennstoff-Chem., 1931, 12, 329–334).—Previous work on the production of HCN by the action of  $NH_3$  on C and by the interaction of  $NH_3$  and gaseous hydrocarbons is discussed (cf. B., 1931, 536; A., 1931, 176). HCN has also been produced by subjecting a mixture of  $CH_4$  and  $N_2$  to an electric discharge under reduced pressure. With a load of 1.6 kw., a pressure of 10–15 mm., and a gas mixture containing 15–25% of  $CH_4$ , the reaction gas contained about 10% of HCN; part of the  $CH_4$  was simultaneously converted into  $C_2H_2$ . A. B. MANNING.

**Recovery of caustic soda in the cellulose industry.** E. R. CHRYSTALL (Ind. Chem., 1931, 7, 372–373).—The waste soda liquor from cotton mercerising and viscose manufacture is of 16–17% concentration and contains hemicellulose. The further use of the soda requires the removal of the hemicellulose. Oxidation processes produce  $CO_2$ , which involves recausticisation, and these have never been worked in a large scale. In present practice dialysis is used, which gives an 8% solution free from org. matter. Both the Cerini plant, which uses parchmētised flat cloth bags fixed in tanks, and the Heibig dialyser (a filter press with parchment paper in place of filter cloth) are satisfactory in practice. C. IRWIN.

**Determination of traces of sodium in potassium salts.** M. FÖRSTER (Ann. Chim. anal., 1931, [ii], 13, 257—261).—Difficulties in the use of the Mg U acetate method are discussed. In one method described the salts are converted into sulphates and the mixture is extracted with aq. EtOH ( $d$  0.939), in which  $K_2SO_4$  is much less sol. than is  $Na_2SO_4$ . In a second method preliminary separation is effected by taking advantage of the slight solubility of  $NaClO_4$ , compared with that of  $KClO_4$ , in a mixture of 1 vol. of EtOH and 2 vols. of EtOAc; the method is rapid and accurate. Phosphate, which is frequently present in commercial K salts, must be removed before using the Mg U acetate method for Na.

H. F. GILLBE.

**Determination of small amounts of bromide in chloride.** B. S. EVANS (Analyst, 1931, 56, 590—593).—The sample is dissolved in  $H_2O$  and  $\frac{1}{3}$  vol. of  $H_2SO_4$  run in, and 5 g. of solid  $CrO_3$  are added. A steady stream of air is drawn through for 1 hr. and passed through an absorption flask containing standard  $As_2O_3$  made alkaline with  $Na_2CO_3$ . The  $As_2O_3$  oxidised is titrated by standard I. When only traces of Br are present, it is first absorbed in NaOH and, after reoxidation by acid  $CrO_3$ , volatilised into 0.01N- $As_2O_3$ .

T. McLACHLAN.

**Decomposition of raw phosphates with sulphuric acid.** F. HALLA (Z. angew. Chem., 1931, 44, 659).—The reaction between  $Ca_3P_2O_8$  and  $H_2SO_4$  takes place in two stages with the formation of the intermediate product  $CaHPO_4 \cdot CaSO_4 \cdot 4H_2O$ , which has been detected by X-ray examination and for which the name "Ardealite" is suggested (cf. Stollenwerk, B., 1927, 554).

A. R. POWELL.

**Change of water-soluble phosphoric acid content of superphosphate during storage in bulk.** IV. Relationship of the manufacturing and storage conditions of superphosphate to the decline of its water-soluble phosphoric acid content (Suppl.). T. SHŌJI and E. NANAI (J. Soc. Chem. Ind., Japan, 1931, 34, 268—270 B).—Superphosphate made from phosphates of other than Christmas Island origin shows little loss of sol.  $P_2O_5$  during storage under summer conditions. Pressure up to 1.0 atm. has no effect in producing this deterioration, and double superphosphate made by the use of  $H_3PO_4$  does not show any loss even at 60°. Ordinary superphosphate made from Christmas Island or Rasa phosphates showed deterioration at 100°, the higher temp. accelerating the action, but not altering the final equilibrium.

C. IRWIN.

**Rapid determination of small quantities of lead in calcium phosphate.** J. R. NICHOLLS (Analyst, 1931, 56, 594—595).— $Ca_2HPO_4$  is dissolved in 2N-HCl and a conc. solution of  $NH_4$  citrate added to prevent precipitation. Slightly ammoniacal KCN is added, followed by 2 drops of  $Na_2S$  (10% solution prepared from colourless crystals), and the colour compared with a standard prepared under the same conditions.

T. McLACHLAN.

**Aluminium acetate solution.** J. G. OBERHARD (Pharm. Ztg., 1931, 76, 1048—1050).—The properties and preparation by various methods of colloidal  $Al(OH)_3$  are described.  $Al(OAc)_3$ , D.A.B. VI, can be imitated by peptisation of freshly precipitated  $Al(OH)_3$  either

with HCl or with  $AlCl_3$ . With the addition of sufficient AcOH to give the necessary odour the solution fulfils all the requirements of the official solution.

E. H. SHARPLES.

**Chemistry of chloroamines.** J. F. T. BERLINER (J. Amer. Water Works' Assoc., 1931, 23, 1320—1333).—The prep., properties, and reactions of  $NH_2Cl$ ,  $NHCl_2$ , and  $NCl_3$  are described. The first two are of importance in water treatment owing to the increased use of the  $NH_3$ - $Cl_2$  process for sterilisation purposes.

C. JEPSON.

**Effect of temp. on Fe oxides.**—See II. Al stearate for paints.—See XIII.

See also A., Oct., 1119,  $H_2SO_4$ ,  $H_2O$ . 1123, Ag sols.  $WO_3$  sols. 1134, Catalysts for production of  $H_2$  by the water-gas reactions. 1135, Synthesis of  $NH_3$ . 1139, Formation of N oxides from  $N_2$ - $O_2$  mixtures. Formation of  $O_3$  from  $O_2$  by electronic discharge. 1172, Auro-sulphites and -thiosulphates of org. bases. Org. Au compounds.

## PATENTS.

**Burner for pyrites, sulphur, and spent oxide.** F. BERTISON and R. J. ELY (B.P. 354,473, 17.6.30).—The apparatus comprises a tower having a number of rotating hearths, supported on an axial shaft, and fixed rabbles.

B. M. VENABLES.

**Production of hydrocyanic acid [from ammonium thiocyanate].** GES. F. KOHLENTCHNIK M.B.H. (B.P. 356,190, 22.5.30. Ger., 6.6.29).—Dil.  $HNO_3$  and  $NH_4CNS$  solutions are run alternately into  $NH_4HSO_4$  solution at 95—97° under conditions such that the liquid always contains at least 1% of free  $HNO_3$ , i.e., the  $HNO_3$  used slightly exceeds 2 mols. per mol. of  $NH_4CNS$ ; the reaction may be so effected that excess liquid is drawn off continuously and HCN recovered from it by distillation.

L. A. COLES.

**Recovery apparatus [for soda from wood-pulp liquors].** F. FALLA, ASSR. to EDGE MOOR IRON CO. (U.S.P. 1,794,006, 24.2.31. Appl., 24.9.29).—In order to prevent the deposition of Na salts on the tubes of waste-heat boilers working in conjunction with soda-recovery furnaces, a part of the cooled gases leaving the boiler is fed back into the furnace and mixed with the hot furnace gases before they enter the boiler to reduce their temp. to, e.g., 760°, so that entrained Na salts solidify and settle out as dust.

D. J. NORMAN.

**Improvement of caustic soda solutions for manufacture of viscose.** HOESCH & Co. (B.P. 355,379, 27.5.30. Ger., 28.5.29).—When using chemically pure NaOH solutions prepared by, e.g., the electrolytic Hg-cathode process in the manufacture of viscose silk, the filters and spinning nozzles tend to become choked with minute aggregates, apparently indistinguishable from normal viscose. This is obviated by adding to the NaOH solution small quantities of Na salts, or salts or hydroxides of K, Fe, or Mn, e.g.,  $NaCl$ ,  $K_4Fe(CN)_6$ , K ferrate, etc.; 1% or more on the wt. of NaOH may be used in the case of Na or K salts, or with Fe or Mn compounds about 0.1%. [Stat. ref.] D. J. NORMAN.

**Preparation of calcium sulphate.** P. SPENCE & SONS, LTD., and S. F. W. CRUNDALL (B.P. 355,694,

25.2.30).—The process described in B.P. 319,228 (B., 1929, 1014) for obtaining anhyd.  $\text{CaSO}_4$  in a finely-divided form is applicable to "sol. anhydrite," prepared by heating  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  at  $200^\circ$ , since the disintegrating action of the  $\text{H}_2\text{SO}_4$  is not dependent on its dehydrating action. The concentration of the acid must, however, be sufficient to effect conversion without formation of acid Ca salts. Heat may be applied to accelerate disintegration.

W. J. WRIGHT.

**Leaching of phosphatic rock.** KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G. (B.P. 356,627, 19.1.31. Swed., 13.2.30).—The rock, together with some of the suspension from the last vat of the series and some of the  $\text{H}_3\text{PO}_4$  obtained in the process, is added to the first of a series of, e.g., 5 leaching vats and  $\text{H}_2\text{SO}_4$  and the liquor obtained in washing the  $\text{CaSO}_4$ , together with an alkali sulphate, if desired, are added to the last vat but one of the series to ppt. the Ca. The part of the suspension from the last vat not returned to the first is filtered to remove the cryst.  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ .

L. A. COLES.

**Manufacture of phosphoric acid and calcium cyanamide.** H. WADE. FROM INTERNAT. AGRICULTURAL CORP. (B.P. 355,955, 1.9.30).—Phosphate rock is reduced with a carbonaceous reducing agent in an electric furnace, the CO and P produced being carried to a water-cooled condenser, where the P is condensed and the CO removed. The liquid P is conducted to an oxidising furnace, and the mixture of  $\text{P}_2\text{O}_5$  and  $\text{N}_2$  passed to a precipitator, whence the  $\text{N}_2$  is returned to the reducing furnace to combine with the  $\text{CaC}_2$  produced from the Ca slag. [Stat. ref.]

W. J. WRIGHT.

**Production of phosphorus and phosphorus-oxygen compounds from previously sintered raw phosphates.** METALGES. A.-G. (B.P. 356,038, 12.11.30. Ger., 29.11.29. Addn. to B.P. 347,937; B., 1931, 632).—A suitably sintered product from raw phosphate, sand, and coke dust is obtainable only if the phosphate contains at least 2% of  $\text{Al}_2\text{O}_3$ , and substances containing  $\text{Al}_2\text{O}_3$  must therefore be added to such materials as Morocco phosphate. It is advantageous to irrigate the hot, sintered material with a mixture of coke dust and  $\text{H}_2\text{O}$ .

W. J. WRIGHT.

**Production of phosphorus-halogen and phosphorus-halogen-oxygen compounds.** N.V. ELEKTROCHEM. IND. (B.P. 356,238, 30.5.30. Holl., 30.5.29).—Mixtures of phosphates, e.g.,  $\text{Ca}_3(\text{PO}_4)_2$ , with C are treated with  $\text{Cl}_2$  at, e.g.,  $500\text{--}600^\circ$  in the presence of  $\text{ZnCl}_2$ ,  $\text{FeCl}_3$ , or  $\text{Cu}_2\text{Cl}_2$ .

L. A. COLES.

**Recovery of phosphotungstates.** J. F. DARLING, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,790,905, 3.2.31. Appl., 25.7.29).—The solution is acidified with HCl and the phosphotungstic acid precipitated by addition of an aromatic amine, e.g., dimethylaniline, ethylaniline, or *o*-toluidine. The ppt. is treated with NaOH and the solution steam-distilled to recover the amine.

A. R. POWELL.

**Treatment of vanadium ores, particularly vanadates of lead and other metals.** A. GILDEMEISTER and E. CAMPAGNE (B.P. 354,816, 13.5.30. Fr., 24.6.29).—The ore is fused with NaCl, KCl, and/or  $\text{CaCl}_2$  together

with a reducing agent, e.g., wood charcoal or NaCN. Pb, Cu, etc. are thus reduced to a metallic regulus and a slag is obtained from which insol.  $\text{V}_2\text{O}_5$  is recovered by leaching out the chlorides with hot  $\text{H}_2\text{O}$ .

A. R. POWELL.

**Separation of beryllium compounds from minerals or substances containing beryllium by chlorination.** W. KANGRO and A. LINDNER (B.P. 356,380, 14.7.30. Ger., 30.7.29).—Ores containing Be oxide, silicate, aluminate, etc., or mixtures of oxides obtained from Be alloys, are treated with  $\text{Cl}_2$  at temps. such that  $\text{BeCl}_2$  is obtained as vapour, e.g., about  $1000^\circ$ ; the gaseous products are passed into  $\text{H}_2\text{O}$  to dissolve the  $\text{BeCl}_2$ , or are treated in an annexed chamber with a countercurrent of  $\text{O}_2$  to convert the  $\text{BeCl}_2$  into BeO. Material containing Fe, Mg, etc. is first treated with  $\text{Cl}_2$  at a lower temp. so that  $\text{FeCl}_3$ ,  $\text{MgCl}_2$ , etc. are formed and removed before the  $\text{BeCl}_2$  volatilises.

L. A. COLES.

**Recovering [potassium, magnesium, and calcium] salts from [sulphate] minerals.** E. P. SCHOCH (U.S.P. 1,794,551—3, 3.3.31. Appl., [A] 20.8.28, [B] 29.12.28, [C] 7.11.29).—(A) Polyhalite is heated at  $500^\circ$  until dehydrated and the  $\text{MgSO}_4$  and  $\text{K}_2\text{SO}_4$  are then extracted with boiling  $\text{H}_2\text{O}$ , leaving a residue of  $\text{CaSO}_4$ . (B) The liquor obtained in (A) is evaporated, treated with  $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 6\text{H}_2\text{O}$  from a later stage, and a crop of  $\text{K}_2\text{SO}_4$  obtained by cooling. A second addition of mixed crystals is made and the solution further evaporated by spraying in the air, whereby a crop of  $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 6\text{H}_2\text{O}$  is obtained which is returned to the first stage of the crystallisation. The mother-liquor is again evaporated by heating to obtain crystals containing more  $\text{MgSO}_4$  than  $\text{K}_2\text{SO}_4$ , and these form the second addition mentioned above. Further evaporation yields  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , and a K-Mg mother-liquor which is passed back one stage. (C) The roasted mineral is extracted with boiling  $\text{H}_2\text{O}$  and the residue of anhyd.  $\text{CaSO}_4$  removed at  $100^\circ$ . The mother-liquor is worked up as described in (B) except that the air-spray evaporation is replaced by heating the solution under reduced pressure so that the mixed crystals separate as  $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 4\text{H}_2\text{O}$ .

A. R. POWELL.

**Manufacture of (A) solutions of complex compounds, (B) compounds, containing fluorine and aluminium.** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 354,824—5, 15.5.30).—(A) Insol. or sparingly sol. fluorides or fluosilicates are boiled with a solution containing  $\text{AlCl}_3$  sufficient to form complex  $\text{AlCl}_3 \cdot \text{AlF}_3$ , which is readily sol. Si, if present, separates as gelatinous  $\text{SiO}_2 \cdot \text{aq}$ . (B)  $\text{CaF}_2$  is boiled with  $\text{AlCl}_3$  and sufficient  $\text{H}_2\text{SO}_4$  to convert the Ca into  $\text{CaSO}_4$ , which is removed and the solution concentrated to expel HCl; on addition of NaF a hydrated double salt containing  $\text{AlCl}_3 \cdot \text{AlF}_3$  is precipitated. If  $\text{Ca}(\text{OH})_2$  be added instead of NaF a ppt. of  $\text{AlF}_3 \cdot \text{Al}(\text{OH})_3$  readily sol. in aq. NaOH is obtained.

A. R. POWELL.

**Production of catalytic materials [for oxidation of hydrocarbons].** N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of M. DE SIMÓ (B.P. 355,008, 6.8.30. U.S., 7.1.30).—A paste of sawdust and  $\text{Al}_2\text{O}_3$  is impregnated with  $\text{Ni}(\text{NO}_3)_2$  solution and, after drying, the



mass is carbonised at 800° in H<sub>2</sub>. The coke is then heated in air at 800° until all C is burnt off and the resulting porous material forms a highly active oxidising catalyst. A. R. POWELL.

**Hypochlorite compositions for bleaching, sterilising, and deodorising.** L. T. HOWELLS, and ELECTRIC SMELTING & ALUMINUM Co. (B.P. 356,767, 4.6.30).—See U.S.P. 1,779,274; B., 1930, 538.

**Demineralisation of bones.** GÉLATINES HASSELT & VILVORDE SOC. ANON., and M. C. G. A. BERGER (B.P. 356,344, 20.6.30).—See F.P. 634,361; B., 1930, 294.

**Separating CO<sub>2</sub> from gases.**—See I. Fe from Fe(CO)<sub>5</sub>.—See X.

### VIII.—GLASS; CERAMICS.

**Application of the down-draught principle to tunnel kilns.** W. D. RICHARDSON (J. Amer. Ceram. Soc., 1931, 14, 572—577).—The cars of the Drayton kiln are provided with perforated floors leading into flues which communicate at the front end with a separate car carrying a baffle wall and vertical flue which, in turn, leads to the top of the next car. A drop damper from the roof of the kiln registers with the top of the baffle wall. The Richardson kiln has a flat, suspended arch and the cars carry their own baffle wall and vertical flue. Drop arches register automatically with a tilting damper on the top of each baffle wall at the end of every movement of the car. This type of kiln gives uniform heating, easy control of atm., and general flexibility of the intermittent down-draught kiln. J. A. SUGDEN.

**Graphical representation of the molecular formulas of ceramic glazes.** H. H. HOLSCHER and A. S. WATTS (J. Amer. Ceram. Soc., 1931, 14, 583—587).—A graph is given correlating the composition and maturing temp. of Pb glazes (cones 015—6), fritted glazes, Pb and leadless glazes (cones 08—6), Bristol glazes (cones 6—10), and porcelain glazes (above cone 10). The chart shows the relationship between the various types of glazes in a simple way and indicates at what temp. a certain constituent becomes important. The prediction of maturing temp. from the composition (and *vice versa*) is only approx. J. A. SUGDEN.

**Systematic study of sheet-iron cover enamels.** A. I. ANDREWS and D. G. BENNETT (J. Amer. Ceram. Soc., 1931, 14, 590—602).—Triaxial diagrams for general workability and acid-resistance of a felspar, Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, SiO<sub>2</sub>, Na<sub>2</sub>CO<sub>3</sub>, NaNO<sub>3</sub>, CaF<sub>2</sub>, cryolite, Sb<sub>2</sub>O<sub>3</sub> enamel are fully worked out. The value of their systematic study is emphasised. Good enamels were obtained within wide limits of variation of their cryolite, CaF<sub>2</sub>, and Sb<sub>2</sub>O<sub>3</sub> contents. Combination of high Sb<sub>2</sub>O<sub>3</sub> and low fluoride contents increases the acid-resistance, and CaF<sub>2</sub> is less detrimental in this respect than cryolite. High-CaF<sub>2</sub> enamels were dull. Firing temps. (if held within correct range) did not affect acid-resistance. Enamels low in cryolite or high in SiO<sub>2</sub> and felspar were difficult to smelt. Enamels which had lost their gloss in the acid test were found to regain their original gloss on refiring, together with a much enhanced acid-resistance. J. A. SUGDEN.

**Laboratory equipment for stiff mud [clay] tests, including exhaustion of the air.** E. LOVEJOY and

H. E. COVAN (J. Amer. Ceram. Soc., 1931, 14, 533—540).—A simple auger machine with provision for evacuation was used to test the effect of de-airing and other factors on the flow of various clays and shales. De-airing greatly improves the manner in which the material passes through the die and increases the shrinkage, strength, and density of the dry ware. The gain in strength and bonding power of the aged clay on de-aeration is less than that of the freshly pugged clay. De-airing freshly pugged clay is more effective in increasing the bonding strength than ageing without subsequent de-airing. De-aerated aged clay has the max. strength. Wet-pan preparation of clays seems to have advantages over pug-mill preparation. J. A. SUGDEN.

### PATENTS.

**Non-splintering or safety glass sheets.** LAMINATED GLASS, LTD., M. V. BARRY, and C. H. H. LAMBERTY (B.P. 356,394, 22.7.30).—Gelatin sheets are interposed between glass sheets, using a mixture of glycerin and AcOH as adhesive, and the edges are sealed. The intermediate layer may comprise, *e.g.*, two gelatin sheets with interposed designs in paper, Al foil, etc. for the production of display glass. L. A. COLES.

**Production of films for avoiding the formation of steaming and adherence of droplets of rain on glass, mirrors, etc.** COMP. INTERNAT. DES INDUSTRIES CHIM. "INTERCHIMIC" (B.P. 356,479, 8.9.30. Belg., 10.9.29).—The films comprise cellulose derivatives, 12—15% of glycerin, and 3—8% of nicotine diluted with 2 vols. of EtOH. L. A. COLES.

**Manufacture of changeant glass.** GLASHÜTTENWERKE VORM. J. SCHREIBER & NEFFEN (B.P. 355,887 and 355,899, [A] 3.7.30, [B] 15.7.30. Czechoslov., [A] 18.7.29, [B] 8.8.29).—(A) A light-transmitting glass is covered with another of different colour, the colours being such that the outer layer is wholly or partly transparent to the inner, whilst the inner layer is almost or completely non-transparent to the outer. The layers may differ in thickness in different parts or lie in oppositely directed wedges; depressions, ribs, etc. may be provided on the surface. (B) One at least of the layers in (A) contains a fluorescent colour, and one may be colourless. L. A. COLES.

**Extracting the impurities from quartz, silicates, and particularly from clay.** W. W. TRIGGS. FROM SOC. DES PROD. CHIM. "ALTERRA" SOC. ANON. (B.P. 356,145, 2.5.30).—The material, after pretreatment if necessary with SO<sub>2</sub> solution to remove Ca, Mg, etc., is treated with dil. H<sub>2</sub>SO<sub>4</sub> containing at least 4% of cryst. FeSO<sub>4</sub> to dissolve Fe compounds, and is then filtered and washed. L. A. COLES.

**Manufacture of compositions and articles of high thermal resistivity and low sp. gr.** C. G. F. CAVADINO (B.P. 356,191, 23.5.30).—Asbestos and/or mica is treated with HF to yield a self-binding composition, which, *e.g.*, is mixed with the product obtained by the action of NaOH on starch to cause it to adhere to steam-pipes etc., or with granulated cork for the production of fireproof insulating material. L. A. COLES.

**Abrasive implements.** H. WADE. FROM BAKELITE CORP. (B.P. 356,188, 1.5.30).—Flexible resins prepared

by condensing a fatty (tung) oil with PhOH and subsequently hardening by heating with a methylene derivative are used for bonding abrasive grains, *e.g.*, on to a paper or cloth base. L. A. COLES.

**Obtaining graduated tinted glass [lenses for spectacles].** A. W. and E. H. HAWES and R. H. BRADSHAW (B.P. 356,832, 18.6.30).

**W-glass joints.**—Sec X.

## IX.—BUILDING MATERIALS.

**Importance of chemical analysis in determining the rational composition of Portland cement.** O. F. HONUS (Cement, 1931, 4, 992—1000).—The method of calculating the rational composition of cement from the ultimate analysis is described, the principal constituents being proportioned to the following compounds:  $3\text{SiO}_2, \text{Na}_2\text{O}(\text{K}_2\text{O}), \text{CaO}$ ;  $2\text{CaO}, \text{Fe}_2\text{O}_3$ ;  $3\text{CaO}, \text{Al}_2\text{O}_3$ ;  $2\text{MgO}, \text{SiO}_2$ ;  $2\text{CaO}, \text{SiO}_2$ ;  $3\text{CaO}, \text{SiO}_2$ ;  $\text{CaSO}_4$ ; free  $\text{CaO}$ ; free  $\text{SiO}_2$ . In judging quality from rational analyses it seems probable that the strength increases with the content of  $3\text{CaO}, \text{SiO}_2$  and decreases as the screen residue ( $>180$  sieve) increases. C. A. KING.

**Hydration of Portland cement.** VIII. Hydration of the calcium aluminate. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1931, 34, 294—298 B; cf. B., 1930, 906).—If finely-powdered cement clinker is shaken with  $\text{H}_2\text{O}$  the  $\text{Al}_2\text{O}_3$  dissolves at a rate which is inversely proportional to the free  $\text{CaO}$  in the clinker. A special clinker was prepared (low in  $\text{CaO}$ ) which, after extraction with  $\text{H}_2\text{O}$  and addition of saturated  $\text{CaO}$  solution to the filtered extract, yielded a ppt. on keeping. This was collected, washed, and dried; it had the composition  $3\text{CaO}, \text{Al}_2\text{O}_3, 10.5\text{H}_2\text{O}$ , and consisted of thin hexagonal plate crystals (optical characteristics given). C. IRWIN.

**Effect of temperature on the setting time of Portland cement.** F. WHITWORTH (Cement, 1931, 4, 1001—1002).—The time of setting of a normal cement (initial 110 min., final 190 min.) was not altered by heating the cement to  $100^\circ$  and cooling. When heated to  $150^\circ$  the initial set occurred in 10 min., due to the added gypsum being converted into  $\alpha$ -anhydrite. At  $200^\circ$  a true rapid set (initial 6 min., final 10 min.) develops, coinciding with the transition of gypsum into  $\beta$ -anhydrite, the dissolution of which is too slow to affect the natural quick-setting properties. A return to normal set was reached at  $500^\circ$ , at which temp. gypsum is converted into a sol. anhyd.  $\text{CaSO}_4$ . C. A. KING.

## PATENTS.

**Manufacture of cement.** "MIAO" MÜHLENBAU U. IND. A.-G. (B.P. 356,611, 31.12.30. Ger., 31.12.29).—It is claimed that a porous and easily ground cement clinker is produced by heating the raw materials until sintering commences, then cooling below the sintering temp., and finally calcining the material completely in the usual manner. The intermediate cooling can be effected by lengthening the furnace or by air-cooling between two kilns, of either the rotary or the shaft type. C. A. KING.

**Production of magnesian Portland cements.** E. BOYNEBURG (B.P. 355,678, 19.5.30. Austr., 3.4.30).—The raw material contains sufficient Fe or Mn to bind the  $\text{MgO}$  as sinter-magnesite; for 5, 25, and 40%  $\text{MgO}$ ,

3.5, 6, and 8%  $\text{Fe}_2\text{O}_3$ , respectively, are added, *e.g.*, by the addition of suitable proportions of clay.

L. A. COLES.

**Hydraulic cements and the like.** K. BIEHL (B.P. 356,144, 2.5.30. Ger., 21.3.30).—As a hydraulic addition agent for  $\text{CaO}$  and cement, clay, marl of low  $\text{CaO}$  content, argillite, and volcanic material, excluding natural hydraulites, are heated at  $500$ — $700^\circ$ , the precise temp. being determined empirically, to develop the max. hydraulic properties. C. A. KING.

**Manufacture of concrete [resistant to attack by water and to shrinkage].** K. WINKLER (B.P. 355,902, 16.7.30).—Clinker of grain larger than usual, *e.g.*,  $>3\%$  of 0.25—2 mm. diam., in addition, if desired, to sand or sandy gravel, is added to the raw material. The clinker may be of material different from the main batch; *e.g.*, it may consist of weakly-hydrating cement, and may be pretreated with one or more solutions containing alkali, alkaline-earth, or F compounds, or oxalic acid, or with vapours of acids containing F, and then dried. Materials for retarding or accelerating the setting may also be added. L. A. COLES.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Gas generator as a direct producer of metallurgical products.** N. E. RAMBUSH and F. F. RIXON (Inst. Chem. Eng., Oct., 1931. Advance proof, 15 pp.).—The development of the slagging producer is reviewed, in particular the type in use at St. Etienne. An average charge consisted of shale (65% of ash) 42.1%, inferior coke 25.9%, limestone 24.5%, and scrap Fe 7.5%, and the air-blast was preheated to  $600^\circ$ . The total fixed C of the charge was about 25%, and 1700 kg. of slag suitable for cement and 950 kg. of silicious pig Fe (6—8% Si) were produced per 1000 kg. of fixed C. Trials carried out on an experimental producer showed that practically any kind of ore or metallurgical refuse can be treated. Zn, Cd, Pb, and Sn are volatilised in all cases and may be recovered by cleaning the gas. In some cases Mn and Ag are volatilised, but Cu and precious metals are recovered in a fusible matte mixed with FeS and possibly Fe. Possible use for hot slagging producers is to be found in the treatment of ferruginous shales, carbonaceous (*e.g.*, black-band) ironstone, residues from Zn retorts and Pb furnaces, tailings from Sn ores, and refuse dumps from disused Zn and Pb mines. C. A. KING.

**Solidification and crystallisation of steel ingots: influence of the casting temperature and the undercooling capacity of the steel.** B. MATUSCHKA (Iron and Steel Inst., Sept., 1931. Advance copy, 16 pp.).—Solidification and crystallisation of steel ingots are absolutely regular phenomena initiated and governed by the cooling of the mould. The internal solidification conditions are influenced chiefly by the casting temp. and the undercooling capacity of the steel. A low casting temp. and the absence of adsorbed gases and non-metallic inclusions tend to produce a finer primary crystallisation and less segregation, *i.e.*, a more uniform ingot and therefore one of higher quality.

A. R. POWELL.

**Attack on mild steel in hot-galvanising.** E. J. DANIELS (Inst. Metals, Sept., 1931. Advance copy, 17 pp.).—The rate of attack of molten Zn below 500° is proportional to the sq. root of the time of immersion and is governed probably by the diffusion of Zn through the layer of FeZn<sub>7</sub> formed immediately on contact of Zn and Fe. Above 500° the Zn apparently has unimpeded access to the Fe surface and the Zn-Fe layer is non-coherent and porous. Very little increase in attack resulted from small additions of Al, Sn, or Sb, but Cd caused a granular and non-protective layer of FeZn<sub>7</sub> to form, also brought about by Ni and Cr in the steel. From the practical point of view the life of the galvanising kettles depends on the temp. not exceeding 480°, as below this temp. the Zn-Fe compound is stable and the attack restricted to the diffusion of Zn through the solid layer. C. A. KING.

**Mottled tinplates.** J. C. JONES (Iron and Steel Inst., Sept., 1931. Advance copy, 11 pp.).—Different types of steel tinned under the same conditions vary considerably in the degree to which they are prone to exhibit mottle markings after tinning. The markings show up best when the tinplate is treated in a 25:5:70 HCl-HNO<sub>3</sub>-H<sub>2</sub>O mixture until the Sn is dissolved, then washed, and rubbed gently with cotton wool. Substitution of a flux containing NH<sub>4</sub>Cl for the usual ZnCl<sub>2</sub> flux on the tinning bath results in almost complete elimination of mottle; the same result is obtained with AlCl<sub>3</sub>. It is suggested therefore that mottle may be avoided by the use of a flux of low viscosity which is kept in a state of const. gentle ebullition by the presence of gas bubbles, usually HCl. A. R. POWELL.

**Influence of silicon on nickel steel.** R. HARRISON (Iron and Steel Inst., Sept., 1931. Advance copy, 19 pp.).—Addition of Si up to 0.87% to steel containing 0.4% C, 4% Ni, and 0.3–0.8% Mn raises the elastic limit, yield point, and tensile strength, but lowers the elongation, reduction of area, and notched-bar impact figures. For steel which is to be converted into plates neither the Si nor the Mn should exceed 0.5% to prevent irregularities in the sheet due to partial air-hardening. In large sections when the Mn is insufficient to ensure the full hardening effect, a further slight addition of Mn has a greater effect than a much greater addition of Si; with a high Mn content increase of Si will maintain a hardened condition in larger cross-sections. Si raises slightly both the A<sub>cl</sub> and A<sub>r1</sub> points of a 4% Ni steel in a similar manner to its effect on a plain C steel. A. R. POWELL.

**Effect of molybdenum on medium-carbon steels containing 1–2.5% Mn.** G. BURNS (Iron and Steel Inst., Sept., 1931. Advance copy, 16 pp.).—Addition of up to 0.3% Mo to 1.3% Mn steels produces little improvement in the mechanical properties in the normalised condition, but reduces the tendency to temper-brittleness and produces a more uniform hardening throughout a large cross-section. In steels containing 2.3% Mn addition of 0.5% Mo almost completely removes the susceptibility to temper-brittleness and imparts to the steel mechanical properties similar to those of oil-hardened and tempered Ni-Cr-Mo steels. A. R. POWELL.

**Surface hardening by nitrogen of special aluminium-chromium-molybdenum steels on a production basis.** W. H. CUNNINGHAM and J. S. ASHBURY (Iron and Steel Inst., Sept., 1931. Advance copy, 14 pp.).—The articles of Al-Cr-Mo steel (Nitalloy) are packed in a gastight nichrome container through which NH<sub>3</sub> is passed while the whole is heated at 495–505° for 40–100 hr. Parts which are to be kept soft are first coated with a thin layer of 80:20 Pb-Sn solder; the excess of which is brushed off while hot to avoid it running on to other parts during nitriding. Practically no growth takes place on parts nitrided at 485° for 20 hr., but the growth of parts nitrided for 100 hr. at 505° may amount to 0.001–0.006 in. according to the cross-section. The outermost layer, 0.001–0.0015 in. thick, of nitrided steel is relatively soft, but the diamond hardness of the next layer should be between 1020 and 1170. Ingress of air during nitriding and during cooling must be prevented, as otherwise the case is very brittle and a larger growth occurs; the steel surface under these conditions is characterised by the presence of a slate-blue coloration. A. R. POWELL.

**Alloys of iron research. X. Chromium-iron constitutional diagram.** F. ADCOCK. **X-Ray examination of chromium-iron alloys.** G. D. PRESTON. **Magnetic tests.** C. E. WEBB (Iron and Steel Inst., Sept., 1931. Advance copy, 48 pp.).—The liquidus of the Fe-Cr system falls from 1527°, the m.p. of Fe, to a min. at 1507° with 25 at.-% Cr, then rises in a flat curve to 1830°, the m.p. of Cr in vac.; the solidus coincides with the liquidus at 20–25 at.-% Cr, and is 3–4° lower than the liquidus with 0–20 at.-% Cr, but with higher Cr the gap widens to a max. of over 80° with 70 at.-% Cr. The  $\gamma$ -field extends to 12.3 at.-% Cr, the A<sub>3</sub> transformation point falling with increasing Cr to a min. at about 850° with 7–8% Cr. The interval between the Ac<sub>3</sub> and Ar<sub>3</sub> points increases from 16° with pure Fe to a max. of 25° with 7–8 at.-% Cr, and that between the Ac<sub>4</sub> and Ar<sub>4</sub> increases from 0° with 3 at.-% Cr to 132° with 11.9 at.-% Cr. The curve of the A<sub>2</sub> transformation point rises with addition of Cr to a max. with 3 at.-% Cr at 7° above the A<sub>2</sub> point of Fe, then falls with a gradually increasing slope to –140° with 76.2 at.-% Cr. In alloys with 40–70 at.-% Cr, quenching from any temp. above a line joining 600° with 40 at.-% Cr and 660° with 70 at.-% Cr depressed the temp. of the magnetic change by an amount depending on the Cr content and reaching a max. of 80° at 50 at.-% Cr, whilst after quenching from below a lower limit extending from 510° at 40 at.-% Cr to 640° at 70 at.-% Cr the magnetic change occurred at the same temp. as for annealed alloys; this behaviour is attributed to the occurrence of transformations of some kind at temps. within the above quenching limits. The Brinell hardness of Fe-Cr alloys rises in a smooth, almost linear curve from 50 for pure Fe to a flat max. of about 340 with 70–80 at.-% Cr, then falls sharply to 110 for pure Cr. The electrical resistivity of the annealed alloys rises steeply from 10.2 microhms/c.c. for Fe to 40.5 for the alloy with 15 at.-% Cr, then more slowly to a flat max. of 52 at 60 at.-% Cr, after which it falls slowly at first and then more rapidly to 13.1 microhms/c.c. for Cr. The lattice parameter of the  $\alpha$ -alloys rises in a slightly undulating curve from 2.860

for Fe to 2.879 for Cr. The magnetic properties of alloys with 0.70 at.-% Cr are tabulated and curves are given for the values of  $\mu$  at values of  $H$  between 0.2 and 100.

A. R. POWELL.

**Applications of the electric furnace for non-ferrous metals, particularly for bright annealing.** H. C. KLONINGER, G. KELLER, and H. MEUCHE (Inst. Metals, Sept., 1931. Advance copy, 18 pp.).—The application of electricity has enabled annealing furnaces to be located nearer to other processes. Suitable furnaces such as the Kenworthy, Hedderheim, Brown-Boveri, and Grünwald are described.

C. A. KING.

**Applications of the spectrograph to the analysis of non-ferrous metals and alloys.** H. W. BROWNSDON and E. H. S. VAN SOMEREN (Inst. Metals, Sept., 1931. Advance copy, 17 pp.).—A technique is described by which the quartz spectrograph may be employed in routine analyses of brass (for Pb, Sn, Fe, Ni, Al, and Mn) and of Pb (for Sn, Sb, Cd, As, Bi, Cu, Zn, Ag, and Th). Tables indicate relative intensities of selected spectral lines with varying impurity content. Fe has also been detected in Pb, but the results in this case are not under quant. control.

E. H. BUCKNALL.

**Brittleness in copper.** C. BLAZEY (Inst. Metals, Sept., 1931. Advance copy, 29 pp.; cf. B., 1929, 286).—Certain varieties of Cu become brittle when annealed at temps. below about 650°, cooled slowly from higher temps., or quenched from higher temps. and reheated at temps. below 650°. The brittleness is accompanied by intercryst. fracture. The types of Cu shown to be liable to brittleness include both the arsenical and As-free varieties. It appears to be necessary, for this defect to appear, that the material should contain Bi and be fairly heavily poled. Fairly prolonged annealing at high temps. in air reduces liability to brittleness of such metal.

E. H. BUCKNALL.

**Nickel-copper alloys of high elastic limit.** D. G. JONES, L. B. PFEL, and W. T. GRIFFITHS (Inst. Metals, Sept., 1931. Advance copy, 18 pp.).—The elastic limit of substantially pure Cu-Ni (50–100% Cu) alloys, though low in the fully annealed or the work-hardened condition, may be raised by low-temp. annealing after cold-working. Addition of Si to these alloys renders them capable of precipitation-hardening, and low-temp. annealing after cold-work develops high elastic limit together with good properties in other directions. For example, an alloy containing 0.17% Si and 1.26% Mn in 80:20 Cu-Ni gives: tensile strength 42.2 tons/sq. in., elastic limit 27.6 tons/sq. in., elongation 21% on  $4\sqrt{A}$ , reduction in area 57.4%.

E. H. BUCKNALL.

**Oxidation of some copper alloys.** J. S. DUNN (Inst. Metals, Sept., 1931. Advance copy, 24 pp.).—Cu and Cu:Zn (0–14% Zn) alloys strictly obey the oxidation law  $W^2 = Kt$ , where  $W$  is the mass of O absorbed in time  $t$ , at a given temp. The rate of oxidation is doubled for every 75° rise in temp. The oxide contains Cu and Zn in approx. the same ratio as in the alloy. Alloys containing >20% Zn show fair agreement with the law, but the oxide in this case is more or less pure ZnO, formed by the reduction of the

original CuO by Zn diffusing to the surface. The alloys containing 14–20% Zn yield a complex oxidation product and the velocity of oxidation does not conform to any known law. The presence of Cl<sub>2</sub> in the atm. greatly accelerates oxidation. Ni or As in the alloys has little effect, but Al reduces the velocity of oxidation. Si affords some protection at lower temps., but at 827° has little effect.

E. H. BUCKNALL.

**Failure of a high-strength brass.** J. E. NEWSON and A. WRAGG (Inst. Metals, Sept., 1931. Advance copy, 9 pp.).—Certain failures in extruded brass parts were due to excessive strains set up by straightening the metal through reeling machines. The stresses are tensional, decreasing from the centre of the bar to the surface, and may be removed by suitable annealing at low temp.

C. A. KING.

**Melting nickel-chromium alloys in hydrogen.** C. J. SMITHELLS, S. V. WILLIAMS, and E. J. GRIMWOOD (Inst. Metals, Sept., 1931. Advance copy, 12 pp.).—Sound ingots of Ni-Cr and Ni-Cr-Mo alloys are prepared by melting in H<sub>2</sub>. Reduction of oxide must be complete before casting, otherwise steam is formed by reaction with H<sub>2</sub> and blow-holes result. Resistance to oxidation of alloys from commercial materials is greater than that of those made from pure metals, possibly due to effects of Si and Al, additions of which to the pure alloys improved markedly this property.

E. H. BUCKNALL.

**Electrothermal production of zinc.** A. WEJNARTH (Diss., Stockholm, 1930, 146 pp.).—The electrical conductivities of numerous molten silicate slags of the types 2RO.SiO<sub>2</sub>, 4RO.3SiO<sub>2</sub>, and RO.SiO<sub>2</sub> have been determined in the systems involving FeO, FeO-CaO, FeO-CaO-MnO, and FeO-CaO-MnO-Al<sub>2</sub>O<sub>3</sub>. Increase in the SiO<sub>2</sub> content of the slags or replacement of FeO by CaO greatly reduces the conductivity. Al<sub>2</sub>O<sub>3</sub> has a greater effect on the conductivity than has CaO, but addition of MnO, which increases the conductivity, compensates for the effect of Al<sub>2</sub>O<sub>3</sub>. The m.p. and sp. gr. of many of these slags have been determined, and from the results the adjustment of a suitable slag for the electrothermal recovery of Zn from roasted ores is indicated. The metallurgical processes in this operation, viz., formation of slag and matte, and distillation and condensation of the Zn, are discussed at some length with especial reference to their dependence on the energy transformations.

A. R. POWELL.

**Comparison of rust protection of iron by zinc, cadmium, and zinc-copper alloys and electro-deposition of such alloys.** F. C. MATHERS and R. L. HARDY (Proc. Indiana Acad. Sci., 1928, 38, 183–185).—The presence of Cu in a Zn covering lessens the time of protection in a spray of 5% NaCl solution. Cd protects nearly twice as long as an equal wt. of Zn. The composition of electrodeposited alloys is controlled by using anodes of two different compositions.

CHEMICAL ABSTRACTS.

**Cadmium plating as a rust-preventive.** S. WERNICK (Ind. Chem., 1931, 7, 358–361).—The author has confirmed that Cd is ordinarily anodic to Fe, but the difference is less than 0.1 volt and in some solutions the relationship may be reversed. Variations in the base

metal may have the same effect. The rate of dissolution of Cd in HCl was found to be about uniform, with no change at the crit. thickness of film as with Zn. Tests with partly protected sheets in a saline atm. show that Cd is intrinsically less protective than Zn. A satisfactory Cd layer is produced by degreasing with NaOH and deposition from  $\text{Cd}(\text{CN})_2$ . The solution should contain about 3% Cd and an excess of free cyanide. and a c.d. of 100–150 amp./sq. in. should be used. The temp. must not exceed 30–40°. For external atm. protection a min. thickness of 0.0005 in. should be specified. Rather less is suitable for internal conditions where Cd is more efficient than Zn, owing to the formation of a protective coating. The crit. thickness of Cd deposits (*i.e.*, that at which it becomes non-porous) varies with the crystal size. It was determined by noting the time after which rust deposits appeared with coatings of different thicknesses, the curve showing a definite discontinuity. C. IRWIN.

**Corrosive effect of liquid fuels.**—See II. Sheet-Fe enamels.—See VIII.

See also A., Oct., 1118, Diffusion in metals. Sn-Sb, Au-Cu, Ca-Bi, and Fe-C-W alloys. Magnetic alloys. 1128, Fe-C-O system. 1133, Dissolution of Zn-Ag alloys in acids. Corrosion of Sn, Fe and steel, and other metals. 1135, Electrodeposition of Cu. Deposits of Cu on Bi. 1142, Determination of Mn in ferrosilicon. 1145, Fe ores from Madras and Mandi State.

#### PATENTS.

**Reverberatory smelting furnace and method of operation.** DAVIS STEEL PROCESS CORP. (B.P. 356,559, 31.10.30. U.S., 23.11.29).—The roof of the furnace is suspended from above and the side walls are absent, their place being taken by the ore or other material being treated, which is supplied downwards and inwards in such a way that the slope of the ore within the furnace is less than the angle of repose, thus providing a large surface upon which heating and melting take place. The correct slope of the ore is maintained by feeding it faster at the toe of the heap than at the top by means of a reciprocating curved plate which guides the ore round the bend from the vertical to the horizontal and promotes the flow of the outer and lower layers more than the inner. B. M. VENABLES.

**[Steel] tubes for use in highly heating molten metals, liquids, vapours, or gases.** F. KRUPP A.-G. (B.P. 354,965, 15.7.30. Ger., 20.7.29).—Steels with a high endurance limit and high strength at high temps. containing 0.12–0.17% C, 0.7–1% Cr, and 0.2–2 (0.3–0.6)% Mo are formed into tubes the inside of which is provided with a protective layer of Cr, Zr, or, preferably, Al. A. R. POWELL.

**Conversion of ferrophosphorus rich in silicon into ferrophosphorus of high phosphorus content free from or poor in silicon.** I. G. FARBENIND. A.-G. (B.P. 355,610, 28.11.30. Ger., 17.12.29).—Crude ferrophosphorus containing 18–45% Si is fused in an electric furnace with an excess of native  $\text{Ca}_3\text{P}_2\text{O}_8$ , whereby an alloy of Fe with 25–30% P and <1% Si is

obtained together with a silicophosphate slag and a little P. A. R. POWELL.

**Obtaining iron from iron carbonyl.** VEREIN. STAHLWERKE A.-G. (B.P. 355,302, 22.2.30. Ger., 8.3.29).— $\text{Fe}(\text{CO})_5$  vapours are passed over rounded, cylindrical, or lenticular particles of inert substances such as quartz, corundum, glass, or porcelain heated at 250–300°, and the resulting leaflets of Fe are separated by mechanical or magnetic means from the inert bodies. [Stat. ref.] A. R. POWELL.

**Treatment of copper matte.** UNITED VERDE COPPER CO., ASSEES. OF O. C. RALSTON AND C. R. KUZELL. (B.P. 355,078, 25.9.30. U.S., 25.9.29).—The matte is melted with Fe or with sufficient reducing agent to reduce its content of  $\text{FeO}$  and  $\text{Fe}_3\text{O}_4$  to Fe so that there is an excess of free metallic Fe in the matte. The powdered product dissolves readily in dil.  $\text{H}_2\text{SO}_4$  with the evolution of a steady stream of  $\text{H}_2\text{S}$ . A. R. POWELL.

**Zinc alloys.** ILLINOIS ZINC CO. (B.P. 355,422, 24.6.30. U.S., 18.10.29).—An alloy of Zn with 0.25–2% Cu and Ag or Sb equal to 0.1–20% of the Cu content is claimed. A. R. POWELL.

**Tungsten carbide [tools cemented with manganese].** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF F. C. KELLEY (B.P. 355,041, 29.8.30. U.S., 29.8.29).—A mixture of 91% of powdered WC and 9% of Mn is pressed into shape and sintered at 1300° in  $\text{H}_2$ . A. R. POWELL.

**Formation of sealed joints between tungsten or tungsten alloys and glass.** N. V. PHILIPS' GLASLAMP-ENFABR. (B.P. 355,119, 1.11.30. Holl., 13.12.29).—The wire is coated with an acid Na tungstate or with a mixture of  $\text{WO}_3$  and an alkali hydroxide, borate, silicate, or peroxide which, when the wire is sealed into the glass, forms a layer of W bronze between the wire and the glass. A. R. POWELL.

**Nickel-chromium alloys.** GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (B.P. 354,765 and 355,083, [A] 12.5.30, [B] 29.9.30).—(A) The addition of up to 1% Al to Ni-Cr alloys melted in  $\text{H}_2$  is claimed to increase appreciably the resistance of the alloys to atm. oxidation at high temps. (B) Up to 2% Si is added in place of Al. A. R. POWELL.

**Coating of metals.** J. H. GRAVELL (B.P. 355,286, 20.5.30. U.S., 4.3.30).—The coating mixture comprises a mixture of  $\text{H}_3\text{PO}_4$  in stearin pitch with solvent naphtha and kerosene as thinners; the metal surface is painted with the mixture and after the solvents have evaporated the metal is heated at 232° to harden the coating and cause it to adhere firmly. A. R. POWELL.

**Electrolytic production of [boron]-aluminium alloys.** D. R. TULLIS and P. OAKLEY (B.P. 356,290, 28.1.31).— $\text{B}_2\text{O}_3$  is added to the electrolyte consisting of a solution of  $\text{Al}_2\text{O}_3$  in a molten mixture of NaCl (90%) and  $\text{CaF}_2$  (10%), whereby pure B-Al is deposited at the cathode. H. ROYAL-DAWSON.

**Melting furnaces. Fusion of minerals. Welding outfits. Separating  $\text{CO}_2$  from gases.**—See I. Coverings for pipes etc.—See II. Treating V ores.—See VII. Metal-melting furnace. Propagating endothermic reactions.—See XI.

## XI.—ELECTROTECHNICS.

Solids in steam.—See I. Sizing.—See V. HCN from gases.—See VII. X-Ray analysis of steels. Magnetic Cr-Fe alloys. Annealing furnaces. Magnetic susceptibility of metals. Pb alloys [for accumulators]. Zn. Rust protection of alloys. Cd-plate.—See X. Electrodialysis cell for soils.—See XVI. Detecting added  $H_2O$ .—See XIX.

See also A., Oct., 1118, Magnetic alloys. 1119, Al and its alloys. Light-metal and Be alloys. 1129, Measurement of e.m.f. 1135, Electrodeposition of Cu. Deposits of Cu on Bi. Synthesis of  $NH_3$ . 1137,  $Co_2O$  photocell. 1138, Measurement of ultra-violet radiation. 1139, Formation of  $N_2$  oxides from  $N_2-O_2$  mixtures. Formation of  $O_2$  by electronic discharge. 1144, Measuring potential. Cu coulometer. Electro-osmosis of water. 1145, X-Ray apparatus for powder analysis. 1152, Prep. of *m*-aminocresol.

## PATENTS.

[Metal-melting] electric [induction] furnace. M. UNGER, ASST. to GEN. ELECTRIC CO. (U.S.P. 1,786,322, 23.12.30. Appl., 1.6.28).—The crucible is surrounded by powdered or granular refractory material which stops any metal leaking from cracks in the crucible lining. Flexible members are arranged between the heat-insulating walls of the furnace and the surrounding casing. J. S. G. THOMAS.

Propagation of endothermic reactions [in powders] in electric arc furnaces. BUFFALO ELECTRIC FURNACE CORP. (B.P. 355,700, 19.5.30. U.S., 27.6.29).—Pulverulent material, e.g., a mixture of Fe ore, C, and flux, is fused by being forced under pressure along a substantially horizontal heated conduit before entering an electric arc enclosed in a furnace arranged at the exit end of the conduit. J. S. G. THOMAS.

[Light-sensitive electrode for] photo-electric cells. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 356,395, 22.7.30. Holl., 25.2.30).—An electrode comprising Cs deposited on a layer of  $Cs_2O$  is claimed. J. S. G. THOMAS.

[Electrolyte for] electric cells. A. M. CODD (B.P. 356,259, 5.6.30).—A solution of  $(NH_4)_2S_2O_8$ , together with, if desired,  $H_2SO_4$ ,  $HNO_3$ , HCl, or salts of the alkali metals or of Hg, Mg, Al,  $NH_4$ , or Zn, is used. J. S. G. THOMAS.

Electric-discharge tubes. EDISON SWAN ELECTRIC CO., LTD., ASSEES. of H. J. SPANNER (B.P. 356,234, 5.5.30. Ger., 4.5.29).—In luminous tubes having a gas filling of A at about 2 mm. pressure and an envelope transparent to ultra-violet radiation, the surface of the cathode is covered partly with electron-emitting material, e.g., Cs, Rb, or Ba, and partly with material which is a poor conductor of heat, e.g., MgO,  $SrC_2$ , Zr amide, or silicides, whilst at least 90% of the total length of the tube is covered by a conducting strip, e.g., of metal or C. [Stat. ref.] J. S. G. THOMAS.

[Gas filling for] electric luminous discharge tubes. CLAUDE NEON LIGHTS, INC., ASSEES. of L. L.

BECK (B.P. 356,332, 17.6.30. U.S., 26.6.29).—The gas filling comprises Hg, He, Ne, and Kr or Xe. J. S. G. THOMAS.

Electric discharge tubes. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 356,407, 29.7.30. Holl., 27.9.29).—Deposited material, e.g., I vapour, is absorbed by, and subsequently liberated by heating from, material, e.g., active C, arranged on a part of the tube wall from which rays are not emitted. J. S. G. THOMAS.

Oxidising the cathode surfaces of electric-discharge tubes. VEREIN. GLÜHLAMPEN U. ELECTRICITÄTS A.-G. (B.P. 356,454, 27.8.30. Austr., 28.8.29).—Oxidation is effected by  $O_2$  liberated by the electrolysis of the heated glass envelope of the tube, produced by passage of current through the gas filling between an anode extending into the tube and an external cathode connected with the outside of the tube. J. S. G. THOMAS.

[Colour-matching] luminous electric-discharge tubes. CLAUDE NEON LIGHTS, INC., ASSEES. of L. L. BECK (B.P. 355,844, 5.6.30. U.S., 26.6.29).—A gas filling for a positive-column electric-discharge tube consists of He and not more than 5% of Ne, together with not more than 1% of A, Kr, or Xe. J. S. G. THOMAS.

Luminous electric-discharge tubes [containing cadmium vapour]. GEN. ELECTRIC CO., LTD., ASSEES. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 356,072, 30.12.30. Ger., 8.2.30).—Tubes of borosilicate glass containing, besides  $B_2O_3$ , alkalis and alkaline-earths, at least 10% of  $Al_2O_3$ , and <60% of  $SiO_2$  are claimed. If desired, a part of the  $SiO_2$  may be replaced by oxides of rare earths, e.g., of La, Pr, or Nd. J. S. G. THOMAS.

Electrolytic rectifiers and circuits therefor. H. J. PARKER, E. A. REA-PALMER, and A. G. SPENCE (B.P. 355,965, 11.9.30).—An electrolyte containing Na pyroborate (8 oz.) and  $KHCO_3$  (8 oz.) in 1 gal. of  $H_2O$  is claimed. J. S. G. THOMAS.

Apparatus for irradiating liquids. G. TIXIER (B.P. 356,083, 22.1.31. Fr., 22.1.30).—Liquid is supplied to an irradiation cell from a vessel provided with a const.-level device and is irradiated by an ultra-violet ray lamp etc. of const. intensity. J. S. G. THOMAS.

Manufacture of [cellulosic] electrical insulating materials. NON-INFLAMMABLE FILM CO., LTD., and V. E. YARSLEY (B.P. 355,654, 15.4.30).—A mixture of cellulose acetate, and/or butyrate, and/or benzoate, or benzylcellulose, with a relatively large % of plasticisers, e.g., 20—30% of trialkyl and/or triaryl phosphate, and/or an alkyl phthalate or tartrate, and/or glycerol ester, is claimed. J. S. G. THOMAS.

Insulating compositions [containing balata or gutta-percha]. ELECTRICAL insulating materials containing gutta-percha or balata. ELECTRICAL RES. PRODUCTS, INC., ASSEES. of A. R. KEMP and J. H. INGMANSON (B.P. 353,518—9, 25.4.30. U.S., [A] 18.1.30, [B] 3.2.30).—(A) Balata or gutta-percha, preferably purified or detersinated and with or without the addition of rubber which may have been deproteinised, is mixed with a hard paraffin wax, e.g., of m.p. 71—77°, the proportion

of the latter being 1—20% of the whole. A satisfactory mixture for insulating purposes is 45—50% of deresinated balata, 45—50% of deproteinised rubber, and 5—10% of wax. (B) The fluavil resins in gutta-percha and, particularly, in balata are more detrimental than the albane resins towards the electrical properties. To wholly or partly deresinated gutta-percha albane resins may therefore be reintroduced; for this purpose the albane resins may be separated from the removed mixture of resins or may be obtained from some other source such as chicle.

D. F. TWISS.

**Electric induction furnaces.** ASSOCIATED ELECTRICAL INDUSTRIES, LTD., and P. H. BRACE (B.P. 356,903, 13.8.30. U.S., 14.8.29).—See U.S.P. 1,768,881; B., 1931, 167.

**Separation of the constituent phases of disperse systems.** A. M. HERBSMAN (B.P. 356,926, 27.8.30).—See U.S.P. 1,783,471; B., 1931, 767.

**[Control means for] electric furnaces.** L. W. WILD, and WILD-BARFIELD ELECTRIC FURNACES, LTD. (B.P. 356,298, 10.6.30).

**[Joining sections of] furnace electrodes.** VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 357,047, 23.1.31. Ger., 23.1.30).

**[Grid structure for] electric accumulators.** H. SALMON, E. T. WILLIAMS, and EXPANDED METAL CO., LTD. (B.P. 356,780, 9.4.30).

**[Sealing of] dry cells.** W. W. TRIGGS. From NAT. CARBON CO., INC. (B.P. 356,271, 6.6.30).

**[Protection of] X-ray apparatus.** C. H. F. MÜLLER A.-G. (B.P. 356,622, 14.1.31. Ger., 17.1.30).

**Electron valves.** S. LOEWE (B.P. 356,109, 22.5.30. Ger., 22.5.29).

**Purifying hydrocarbons.**—See II. **Perylene dye.**—See IV. **Coated flexible materials.**—See V. **Fe from Fe(CO)<sub>5</sub>.** **W-glass joints.** B-Al.—See X. **Conc. latexes.**—See XIV.

## XII.—FATS; OILS; WAXES.

**Characterisation of fats in small amounts.** E. CHARGAFF (Z. physiol. Chem., 1931, 199, 221—224).—Micro-determinations are described of the I and sap. vals. of fats, using 2—5% of the usual quantity. For the hydrolysis a solution of NaOPr in PrOH is employed.

J. H. BIRKINSHAW.

**Rancidity [in fats] and stability in shortening products.** W. O. POOL (Oil and Fat Ind., 1931, 8, 331—336).—O<sub>2</sub>-absorption rates (possible volatile products were neglected) were approx. inversely proportional to the time taken to develop rancidity in the Schaal stability test. The intensity of the Kreis test increased smoothly with the time of exposure of the fat to free air (at 40—100°); when the fat was stored in sealed containers (in air, O<sub>2</sub>, N<sub>2</sub>, or vac.), however, the Kreis test rose to a max. and after 30—100 days fell to vals. as low as or below that of the original fat, although marked rancidity had developed. E. LEWKOWITSCH.

**Determination of butyric and hexoic acids in edible fats.** J. GROSSFELD and F. BATTAY (Z. Unters.

Lebensm., 1931, 62, 99—126).—The fat (5 g.) is saponified by heating with 2 c.c. of KOH (*d* 1.5) and 10 c.c. of glycerin and the authors' method then followed (B., 1927, 667). Of the resulting 110-c.c. distillate, 50 c.c. are titrated with 0.02N-NaOH (phenolphthalein) and the factor 0.616 used to obtain the butyric acid val. (*T*); 50 c.c. also are shaken with 50 c.c. of neutral petrol, and the aq. layer is titrated to give (allowing for any blank) the val. *T*<sub>1</sub>. Then *A* (relative increase in total acid concentration) = 100 (*T* — *T*<sub>1</sub>)/*T*%. From the formula *C* (hexoic acid as % of total titration) = 100 (*A* — *K*<sub>b</sub>) + (*K*<sub>c</sub> — *K*<sub>b</sub>) where *K*<sub>b</sub> and *K*<sub>c</sub> are the respective decreases in concentration of pure butyric and hexoic acids in the aq. layer, the % of hexoic acid is given by *P*<sub>2</sub>*CT*, and of butyric acid by *P*<sub>1</sub>(100 — *C*)*T*. The *P* factors depend on the fat; *P*<sub>2</sub> for butter fat 0.00482 (*P*<sub>1</sub> 0.00129), coconut oil 0.00548, cacao butter 0.00396 (*P*<sub>1</sub> 0.00127), lard 0.00542. Tables and curves are given for *K*<sub>b</sub> and *K*<sub>c</sub> at various concentrations of the respective acids, and in different solvents. Statistical examination of the data for 32 butter fats gave a mean butyric acid content of 3.73% and hexoic acid 1.72%, the mean deviations from the mean of a single value (method of least squares) being 6.6 and 16.8%, respectively. The correlation factor for the contents of the two acids was + 0.70 and the possible error ± 0.061%. Hexoic acid (0.61%) is the fatty acid of lowest mol. wt. in edible cacao fat. The method is therefore applicable to the detection of fats containing butyric acid in edible fats.

J. GRANT.

**Iodometric acidimetry in fat analysis.** I. H. P. KAUFMANN (Allgem. Oel- u. Fett-Ztg., 1931, 28, 225—227, 248—250; cf. B., 1930, 1118; A., 1929, 1271).—The acid vals. of saturated or unsaturated fatty acids or fats (pure or crude) may be determined accurately and directly as follows: 2—5 g. of fat (0.2 g. of fatty acid) are dissolved in 50 c.c. of Pr<sup>6</sup>OH; 25 c.c. of a 1:1 mixture of 10% aq. KI and 5% aq. KIO<sub>3</sub> and a measured excess (25 c.c.) of 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution are added. The reaction is completed in 2½ hr. at 55—60°. After cooling, excess (25 c.c.) of a fresh 0.1N-I solution is added and back-titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> in the presence of 5 c.c. of starch solution. The method is especially useful for dark oils, e.g., train oils.

E. LEWKOWITSCH.

**Determination of iodine value of liquid fats with mixtures of ether and water.** V. I. ESAFOVA (J. Appl. Chem., Russia, 1930, 3, 1207—1221).—Margosches' method is satisfactory for liquid fats, but in presence of unsaturated hydroxy-acids the results are high. If insufficient I is added HI unites with fats.

CHEMICAL ABSTRACTS.

**Aluminium salts of fatty acids and their swelling in organic solvents.** E. MARKOWICZ (Farben-Ztg., 1931, 36, 2050—2051, 2086—2087, 2122—2124; cf. B., 1929, 101).—The behaviour of Al palmitate, stearate, and linoleate in C<sub>6</sub>H<sub>6</sub>, xylene, turpentine, "Sangajol," "Dekalin," and linseed oil was studied, consideration being given to effects of temp. and time, Al content of the salts, both precipitated and fused salts being used. The results are classified with reference to the type of gel, paste, sol, etc. formed. This is a preliminary survey, introductory to more specialised work in progress. S. S. WOOLF.



**Solubility of dry-cleaning soaps.** C. L. BIRD (J. Soc. Dyers and Col., 1931, 47, 254—258).—The solubility at various temps. of the K and Na soaps of oleic, stearic, and palmitic acids in white spirit in the presence of free fatty acid, methylated spirit (64 O.P.), and  $H_2O$  is examined. K soaps are more sol. than Na soaps, and the oleates are the most suitable for sol. dry-cleaning soaps; solubility decreases in the order K oleate, K stearate, Na oleate, K palmitate, Na stearate, and Na palmitate. Na stearate and palmitate are definitely unsuitable for use as dry-cleaning soaps since they are almost insol. and their solutions set to jellies.  $H_2O$ , methylated spirit, and free fatty acid assist solubility to an extent increasing in the order named, the efficiency of methylated spirit being due to its  $H_2O$  content. When the solvent consists of methylated spirit or free fatty acid the ratio of solute to soap increases rapidly as the soap concentration decreases. The lower fractions of petroleum appear to be better soap solvents than the higher (e.g., white spirit). A. J. HALL.

**Determination of iodine and chlorine in iodised oils.** T. T. COCKING and G. MIDDLETON (Quart. J. Pharm., 1931, 4, 175—177).—I and Cl are converted into the inorg. condition by refluxing with glacial AcOH and Zn. The resulting I is titrated with 0.05N-KIO<sub>3</sub> in presence of excess of HCl. The total halides are determined by Volhard's method and Cl is obtained by difference.

T. McLACHLAN.

**Resoftening of linoxyn.** F. FRITZ (Farben-Ztg., 1931, 36, 2065—2068, 2103, 2140).—The resoftening of linoxyn to a tacky mass under the action of moisture in the absence of air and light is discussed, the literature on the subject being summarised. S. S. WOOLF.

**Friction and lubricating oils.**—See I. **Determining fat in milk. Protein analysis and fats.**—See XIX.

See also A., Oct., 1178, **Beef tallow**s, 1194, **Vitamin-A** in liver oil. 1195, **Liver oils** of fish and birds. **Vitamin-A** in cod-liver oil. 1199, **Lignoceric acid** from seeds of *Adenantha pavonina*. Seed oil of *Putranjiva Roxburghii*. Karité butter. Glycerides from cacao butter etc. Bark of *Xanthoxylum carolinianum*.

## PATENTS.

**Obtaining by distillation of solid or plastic materials products intended to replace vegetable oils as well as essence of turpentine in all their applications.** H. ARIAS (B.P. 356,203, 4.6.30. Fr., 20.9.29).—Hydrocarbons with a high isoprene content, e.g., balata, gutta-percha, rubber, Borneo gum, are distilled by indirect heat, the vapours produced being agitated before condensation. The products may be re-treated to obtain the light spirit. The products may be used in paints etc., and especially for the regeneration of vulcanised rubber, as rubber solvents, etc. [Stat. ref.] E. LEWKOWITSCH.

**Purification of [animal] waxes.** I. G. FARBERIND. A.-G. (B.P. 355,857, 12.6.30. Ger., 12.6.29).—Shellac wax, beeswax, etc., dissolved in liquid monohydric aliphatic alcohol(s) or their fatty acid esters (e.g., EtOH, BuOH, EtOAc) are treated with decolorising C activated by  $ZnCl_2$  (cf. B.P. 10,126 of 1914; B., 1915, 862).

E. LEWKOWITSCH.

**Purification of lubricating oil.**—See II. **Primary alcohols. Wetting agents.**—See III. **Wax-like products.**—See XIII. **Vulcanisation products.**—See XIV. **Butter and margarine.**—See XIX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Qualitative reactions of the turpentines.** I. M. KORENMAN (Pharm. Zentr., 1931, 72, 241—243).—Different samples of turpentine do not give the same coloration when tested under identical conditions. Known reactions are employed, and, in that of Kingzett and Hake, the substitution of other carbohydrates for sucrose gives greater sensitivity. A table is given showing the varying colour changes obtained at different dilutions when testing alcoholic solutions of turpentines with a 1% solution of vanillin in conc.  $H_2SO_4$ .

F. C. HARWOOD.

**Drying of oil-nitrocellulose mixtures.** H. WOLFF [with B. ROSEN] (Farben-Ztg., 1931, 36, 2047—2049).—The progressive oxidation of films of a nitrocellulose lacquer containing boiled linseed oil and of the oil itself was studied, by isolation of oxidised and non-oxidised fatty acids etc. It is shown that the oxidation process of linseed oil in such "combination lacquers" is considerably slower than the normal one of the oil. The effect of thickness of film is also less marked with the mixed lacquer than with the oil. S. S. WOOLF.

**Production of furfuraldehyde resins.** O. ROUTALA and O. KUULA (Suomen Kem., 1931, 4, 50—59).—Condensation of furfuraldehyde and PhOH in mol. quantities in the presence of 4N-NaOH, 3.6N-KOH, and 18% CaO yields an almost theoretical amount of readily sol. soft black resin capable of use in lacquers. A white resin results from condensation of furfuraldehyde and  $COMe_2$ , the quality being improved by substitution of part of the  $COMe_2$  by colophony. Heat converts furfuraldehyde-PhOH resins into insol. compounds similar to those obtained directly by the use of acid catalysts in the condensation. T. T. POTTS.

**Al salts of fatty acids.**—See XII.

See also A., Oct., 1124, **Viscosity of solutions of cellulose derivatives.**

## PATENTS.

**Treatment of oil paints and varnishes.** ELEKTROCHEM. WERKE MÜNCHEN A.-G. (B.P. 355,801, 29.5.30. Ger., 3.7.29).—Large amounts of paraffin wax etc. can be incorporated in the form of an aq. colloidal dispersion (prepared by emulsifying a xylene solution in  $H_2O$  with Na oleate) in an equal or greater vol. of paint; the  $H_2O$ -in-oil emulsion obtained dries well to resistant films. E. LEWKOWITSCH.

**Direct production of siccatives in solution.** I. G. FARBERIND. A.-G. (B.P. 353,783, 6.10.30. Ger., 10.10.29).—Oxides, hydroxides, or corresponding salts of readily volatile acids, e.g., AcOH, of one or more siccativ metals, e.g., Pb, Mn, Co, are treated, under reduced pressure if desired, in the absence of  $H_2O$  with one or more acids capable of forming siccatives, e.g., naphthenic, linoleic, benzoic acids, in the presence of a volatile org. solvent, e.g., cyclohexanol, at temps. (120—

140°) at which H<sub>2</sub>O of reaction continuously distills off, but below the b.p. of the solvent. S. S. WOOLF.

**Production of derivatives of resin acids.** CHEM. FABR. DR. K. ALBERT G.M.B.H. (B.P. 353,616, 4.6.30. Ger., 28.8.29).—Natural or artificial resin acids, *e.g.*, rosin, Congo copal, manila, salicylic acid-CH<sub>2</sub>O condensation products, are heated, *e.g.*, for 4 hr. at 300°, with esters of other acids boiling above 200°, *e.g.*, butyl phthalate, benzyl acetate, the acid of which is set free and may easily be eliminated, *e.g.*, by distillation in vac., washing with alkaline solutions, etc. either during or after the reaction. The need for "running" natural resins before esterification is obviated. S. S. WOOLF.

**Purification of resinous and wax-like products.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 354,775, 7.3.30).—Such products, *e.g.*, wool fat, shellac wax, etc., are mixed with at least 50 wt.-% of adsorptive C and the mixture is extracted with suitable org. solvents, *e.g.*, EtOH, C<sub>6</sub>H<sub>6</sub>, benzine; successive stages involving the use of different solvents and/or temps. may be employed. S. S. WOOLF.

**Manufacture of coating compositions and application thereof.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 353,589, 21.5.30).—Mixtures of cellulose esters, containing 10–75 wt.-% of the nitrate (which is sol. in the solvents of the pigmented nitrocellulose lacquers to be applied thereover), a common solvent softener for the esters, *e.g.*, tolyl phosphate (< 3% of the total), and a suitable solvent mixture, are claimed as aeroplane dopes. A preferred cellulose ester mixture contains 3 pts. of acetate to 2 pts. of nitrate. S. S. WOOLF.

**Cellulose derivative [pyroxylin] coating compositions.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 356,446, 21.8.30. U.S., 22.8.29).—Addition of, *e.g.*, 15–25% of low-leaded ZnO or SnO prevents "chalking" of cellulose ester or ether lacquers, which may contain dammar resin and Bu phthalate. E. LEWKOWITSCH.

**Manufacture of cellulose ester or ether enamels and the like.** A. B. MARTIN (B.P. 354,748, 11.4.30).—A suspension of pigment in a suitable liquid is forced, preferably centrifugally, into a more or less compressed mass of a cellulose ester or ether, which is moist with a liquid with which the pigment-suspending liquid is miscible. S. S. WOOLF.

**Manufacture of resin-like condensation products [from carbazole compounds].** A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 354,840, 17.5.30).—Aldehydes or substances yielding them are caused to react with *C*-alkylcarbazoles (cf. B.P. 327,746; B., 1930, 625) in presence of acid condensing agents, *e.g.*, HCl, giving pale transparent resins, sol. in org. solvents. S. S. WOOLF.

**Resinoid compositions.** BAKELITE CORP., Assees. of H. KLINE (B.P. 353,430, 17.4.30. U.S., 19.4.29).—A phenol (1 mol.) is treated with a methylene-containing compound (2 mols.) in the presence of an acid catalyst, *e.g.*, oxalic acid, and, if desired, a solvent (b.p. above 125°) to aid reaction control, *e.g.*, Et phthalate, and the dehydrated reaction product is incorporated with filling materials in glass, porcelain, enamel-lined, or Cr-plated apparatus throughout. The surfaces in contact with

the resinoid should be free from metals having  $d > 8$  and subject to attack by acids of concentration equiv. to 1% aq. HCl, *e.g.*, Fe and Cu. Substantially colourless resinoids usable in white moulding compositions are obtained. S. S. WOOLF.

**Manufacture of mixed esters [of alkyd-resin type].** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. H. KIENLE (B.P. 356,340, 20.6.30. U.S., 21.6.29).—A drying oil, *e.g.*, linseed oil, is bodied by heating, the alcohol ingredients, *e.g.*, glycerol, of an alkyd resin are added, and the mixture is heated till completely blended, after which the acid components of the resin are added and completely esterified by continued heating.

E. LEWKOWITSCH.

**Resinous cementing compounds.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. V. ADAMS (B.P. 353,713, 1.8.30. U.S., 9.8.29).—The alcohol and acid components of an alkyd resin, *e.g.*, glycerol and phthalic acid, are heated with drying or semi-drying oils or their fatty acids until a definite curing stage is reached. *E.g.*, when a sample of the resin will cure in 1½ min. at 200°, a small amount of a degelled drying or semi-drying oil, *e.g.*, degelled linseed oil, is added, and the whole heated until a completely blended cement is obtained. S. S. WOOLF.

**Synthetic resins.** AMER. CYANAMID CO. (B.P. 356,141, 28.4.30. U.S., 6.6.29).—Inclusion of coconut oil fatty acids (or oil) in the preparation of a resin of the type glycerol-phthalic anhydride with stearic and/or palmitic acid improves the drying properties (in a lacquer coating) without impairing the fastness to light. Resinification at 200–290° in an inert atm. is preferred.

E. LEWKOWITSCH.

**Manufacture of (A) moulding materials, (B) impregnated products, [from coal].** KOPPERS CO., Assees. of H. J. ROSE and W. H. HILL (B.P. 353,493 and 353,495, 17.4.30. U.S., 13.5.29).—(A) Coking coal is dissolved at 200–400° in high-boiling coal- or water-gas tar oil or cracked petroleum oils, inert fillers (asbestos fibre, mineral wool, graphite, rubber), thinning agents (tar oils or H<sub>2</sub>O), pigments, and/or plastic materials (resins, waxes, bitumens) are incorporated, and the mixture is moulded, the coal solvent being subsequently removed. (B) Fibrous or other porous materials, *e.g.*, wood, cardboard, brick, or coke, impregnated with such coal solutions or with coal precipitated therefrom are claimed. Rubber or latex (together with S compounds as tougheners, and accelerators, *e.g.*, diphenylguanidine), fillers, resins, preservatives, etc. may be incorporated.

S. S. WOOLF.

**Combination of hardenable phenol-aldehyde condensation products with [China] wood oil.** BAKELITE GES.M.B.H. (B.P. 355,827, 30.5.30. Ger., 31.5.29. Addn. to B.P. 349,931; B., 1931, 769).—The condensation products in the resole stage (or acetylated etc. derivatives of the resoles) are combined with tung oil by heating at, *e.g.*, 130–160° in the presence of a solvent. E. LEWKOWITSCH.

**Grinding of paint.**—See I. Products [resins] from coal.—See II. Varnishes etc. from polymerised vinylphthalenes.—See III. Azo pigment.—See IV. Cellulose acetate. Coated flexible

materials.—See V. Abrasive implements.—See VIII. Coating of metals.—See X. Substitutes for vegetable oils.—See XII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Colour reactions of latex for identification of *Hevea* clones.** W. BOBILIOFF (Arch. Rubbercultuur, 1931, 15, [7]; Med. Proefstat. Rubber, 1931, [65], 289—308).—The oxidase enzymes present in *Hevea* latex lead to the development of discoloration of the latex and the serum; they are activated by salts of Ca or Mg, a 1% solution of  $\text{CaCl}_2$  being most effective. The intensity and tone of the colour and the time necessary for its development are characteristic of the clone. The test is best made in the shade on drops of latex collected from the stems of healthy half-grown leaves.

D. F. TWISS.

**Active filling materials [for rubber].** W. BACHMANN, J. BEHRE, and P. BLANKENSTEIN (Kolloid-Z., 1931, 56, 334—349).—For the purpose of studying the effects of fillers, especially different forms of  $\text{SiO}_2$ , methods of measuring the plasticity, "nerve," surface tension in solution, filtering power, swelling, adsorption capacity, particle size, tensile strength, hardness, elasticity, resistance to wear, and fatigue of raw and vulcanised rubber are described. Experiments on the effects of filling materials are only preliminary.

E. S. HEDGES.

**Luminescence of accelerators and preservative agents used in the rubber industry.** R. DITMAR (Caoutchouc et Gutta-Percha, 1931, 28, 15,685—15,686).—The appearance of the luminescence of a number of accelerators and antioxidants illuminated by a quartz-Hg lamp with an ultra-violet filter is described. In some cases it is possible to distinguish between supplies of the same accelerator from different manufacturers.

D. F. TWISS.

#### PATENTS.

**Concentration of latexes, particularly latexes from rubber plants.** J. AUMARECHAL and G. ROBRIEUX (B.P. 353,876, 25.4.30. Fr., 2.5.29).—Latex passing at high speed from one or more upper tubular members to one or more lower tubular members is subjected to evaporation. The formation of drops in the stream of liquid is prevented by providing smoky flames so disposed that soot and oily material are condensed on the liquid surface, by introducing traces of org. substances such as oils, stearic acid, or  $\text{NH}_4\text{Ph}$ , or by disposing a source of static electricity in proximity to the latex stream.

D. F. TWISS.

**Colloidal emulsion [rubber cement] and process.** T. WHITTELEY (U.S.P. 1,793,983, 24.2.31. Appl., 6.11.22).—It is possible to emulsify  $\text{H}_2\text{O}$  in solution of rubber in an org. solvent without destroying the viscosity of the solution, but, in the presence of Na oleate or similar  $\text{H}_2\text{O}$ -sol. dispersing agent, the primary emulsion becomes inverted and an aq. emulsion of the rubber solution is formed. By homogenisation of the latter >95% of the dispersed droplets may be reduced to <0.001 mm. in diam. An emulsion ranging from a thin fluid to a stiff dough can be obtained also by other methods, e.g., by mixing rubber latex with a rubber

solvent or rubber solution in the presence of Na oleate. Such emulsions have marked penetrative power towards fibrous materials and also are directly applicable to wet leather for adhesive purposes. Similar emulsions can be produced with pyroxylin solution instead of rubber solution.

D. F. TWISS.

**Manufacture of goods of or containing rubber or similar material.** DUNLOP RUBBER Co., LTD., ANODE RUBBER Co., LTD., H. C. YOUNG, and C. HEMM (B.P. 353,656, 1.7.30).—An aq. emulsion or dispersion, e.g., a conc. latex mixing, is applied to a suitable backing, such as of Pb or a rubber composition, and is then partly or completely set by application of a coagulant, e.g.,  $\text{AcOH}$ . By placing the coated backing cold in a hot press and causing a hot patterned plate to descend on it, perforations can be produced in the moist coagulum.

D. F. TWISS.

**Manufacture of articles or materials, particularly of organic materials such as rubber, provided on their surfaces with mottled patterns or ornamentations.** DUNLOP RUBBER Co., LTD., ANODE RUBBER Co., LTD., and F. H. LANE (B.P. 353,524, 26.4.30).—The articles etc. are given a mottled pattern or ornamentation by treating them with a coarse aq. dispersion or emulsion of suitably coloured rubber solutions.

D. F. TWISS.

**Manufacture of articles of rubber or similar material.** DUNLOP RUBBER Co., LTD., ANODE RUBBER Co., LTD., and A. S. KING (B.P. 353,895, 22.4.30).—Supports or backing strata, coated with a fluid mixture of a coagulant, e.g., an acid or a salt of a bi- or ter-valent metal, and a wetting agent, e.g., casein or saponin, are contacted with an aq. dispersion such as compounded rubber latex. The coagulated layer is eventually removed and dried.

D. F. TWISS.

**Colouring of rubber and of artificial rubber-like masses.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 354,769, 13.5.30).—Complex Al lakes of the hydroxy-, aminohydroxy-, and *N*-substituted aminohydroxy-anthraquinones containing one or more  $\text{SO}_3\text{H}$  groups in the anthraquinone nucleus are employed as colouring materials for rubber. They are fast to light and soap and resistant to hot or cold vulcanisation and do not "bleed."

D. F. TWISS.

**Making a rubber composition [and carbon black therefor].** E. B. SPEAR and R. L. MOORE, ASSRS. to THERMATOMIC CARBON Co. (U.S.P. 1,794,558, 3.3.31. Appl., 16.8.28).—A modified thermatomic black ("P-33") is made by decomposing hydrocarbon gas in a retort without substantial combustion, the hydrocarbon gas being previously mixed with an inert gas. The product differs from earlier types of thermatomic black in its colour, greater bulkiness, and ability to form mixtures in much higher proportions with rubber with beneficial results. The vulcanised rubber containing it exhibits a high tensile strength comparable with that of a mixture of similar composition containing C black, but has a lower degree of stiffness and a higher ultimate elongation than is given by C black or even the older thermatomic black. (Cf. B.P., 307,743; B., 1930, 626.)

D. F. TWISS.

**Preparation of vulcanisation products from rubber.** J. Y. JOHNSON. From I. G. FARBENIND.

A.-G. (B.P. 354,760, 8.5.30).—Strong vulcanised products are obtained from rubber containing 10–250% of ZnO prepared from a by-product of a wet chemical reduction process, *e.g.*, from  $\text{ZnCO}_3$ , which has been purified by elutriation.  
D. F. TWISS.

**Manufacture of vulcanised rubber.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 353,871, 23.4.30).—Vulcanisation is accelerated by the reaction products of mercaptothiazoles, dithiocarbamates, or alkylxanthic acids containing 4 or more C atoms with chloroformic or chloroacetic esters. The products exhibit less scorching tendency than the parent accelerators.  
D. F. TWISS.

**Manufacture of vulcanised rubber and the like.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 353,692, 24.7.30).—Compounds of the formula  $\text{HO}\cdot\text{R}\cdot\text{O}\cdot\text{R}'\cdot\text{Y}$ , where R and R' are bivalent aromatic hydrocarbon residues and Y is H or OH, improve the ageing resistance of rubber without affecting the rate of vulcanisation or causing substantial discoloration on exposure to sunlight. Examples cited are *p*-hydroxy- and *pp'*-dihydroxy-diphenyl ether.  
D. F. TWISS.

**Manufacture of [rubber] vulcanisation products.** J. Y. JOHNSON. From I. G. FARBERIND, A.-G. (B.P. 352,167, 19.5.30).—For the softening of rubber, stearic acid and similar agents can be advantageously replaced by the products obtained by destructive oxidation of difficultly volatile paraffin hydrocarbons, *e.g.*, paraffin wax, these products, consisting mainly of carboxylic acids, being free from resins and/or asphaltic matter, and preferably also being freed from neutral products.  
D. F. TWISS.

**Reclaiming vulcanised rubber-fibre products.** RUBBER REGENERATING CO., Assees. of A. W. BULL (B.P. 356,519, 1.10.30. U.S., 2.10.29).—Vulcanised rubber fibre, such as a motor-tyre casing, is broken into small pieces and the smaller freed particles of rubber are separated, *e.g.*, by screening. The remaining disintegrated material is then beaten with  $\text{H}_2\text{O}$  to strip more rubber from the fabric and to reduce the latter to fibre; the rubber settles and is removed. The wet mixture of fibre and rubber, after being drained and dried, is then loosened and separated by mechanical means.  
D. F. TWISS.

**Patterned material of rubber or containing rubber as its principal constituent and manufacture thereof.** DUNLOP RUBBER CO., LTD., and F. W. WARREN (B.P. 356,849, 27.6.30).

**Manufacture of moulded rubber goods.** DUNLOP RUBBER CO., LTD., and F. W. WARREN (B.P. 354,862, 21.5.30).

**[Heating by electrical induction moulds for] vulcanisation of rubber and rubber-like substances.** HUNGARIAN RUBBER GOODS FACTORY, LTD., P. KLEIN, and S. GOTLÉB (B.P. 356,966, 13.10.30).

**Rubbered paper.**—See V. Coated fabric.—See VI. Insulating materials.—See XI. Substitutes for vegetable oils.—See XII. Moulding materials from coal.—See XIII. Chewing gum.—See XIX.

## XV.—LEATHER; GLUE.

**Colorimetric determination of tannin.** A. B. SHAKHKELDIAN (J. Appl. Chem., Russia, 1931, 3, 1117–1124).—The solution should contain 0.5–2.0 g. of tannin per litre. To 100 c.c. are added 0.315 g. of pure, dry tannin; to 10 c.c. of the solution are added 10 c.c. of 2%  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$  solution, and the mixture is diluted to 100 c.c. 10 c.c. of the unknown solution are also treated with 2%  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$  solution (10 c.c.), diluted, and compared with the former after 15 min. It is assumed that Kahlbaum tannin contains 0.015 g. of impurities in 0.315 g. The method is rapid, but only 0.1 as sensitive as Menaul's method.

CHEMICAL ABSTRACTS.

**Determination of pyrocatechol tannins in presence of pyrogallol tannins.** A. PONTE and G. GUALDI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1931, 9, 323–334).—50 c.c. of the filtered tan liquor of analytical concentration are boiled with  $\text{CH}_2\text{O}$  (40% solution) and conc. HCl, cooled, filtered through a tared filter, and dried at 100° to const. wt.  
D. WOODROFFE.

**Deterioration of chestnut- and quebracho-tanned leathers by sulphuric acid.** R. C. BOWKER (J. Amer. Leather Chem. Assoc., 1931, 26, 444–461).—Greater deterioration was obtained in leathers tanned with chestnut extract than with those tanned with quebracho extract and respectively after-treated with amounts of  $\text{H}_2\text{SO}_4$  varying from 0.5 to 4% and subsequently aged for 2, 4, or 6 months. The deterioration of the quebracho-tanned leather was the same for both ordinary and sulphited extract. The deterioration was slightly greater for highly-tanned leathers.  
D. WOODROFFE.

**Removal of lime from animal skin by acids and acid salts.** A. KÜNTZEL and R. BIEDERMANN (Kolloid-Z., 1931, 56, 207–225).—Phenolphthalein detects only the free CaO in skin, the remainder being bound. When the free CaO is removed by washing or by neutralisation, the equilibrium is disturbed and more CaO is set free. The bound CaO can be set free only by acids having a dissociation const.  $> 2 \times 10^{-6}$ . Weak acids cannot remove CaO completely;  $\text{NH}_4$  salts occupy an intermediate position between these and strong acids. There is a relation between loss of CaO and swelling.

E. S. HEDGES.

**Tannin in brewing.**—See XVIII.

See also A., Oct., 1141, Measurement of  $p_{\text{H}}$ . 1174. Action of emetics on gallic and tannic compounds. 1178, Chemistry of leather.

## PATENTS.

**Depilation of hides.** WALLERSTEIN CO., INC. (B.P. 355,306, 22.3.30. U.S., 2.4.29).—Hides are treated with an alkaline solution at  $p_{\text{H}}$  7–10 and then with an antiseptic liquor at 27–32° produced by the action of enzyme-forming bacteria of the *mesentericus* or *subtilis* group on a carbohydrate-containing medium.

D. WOODROFFE.

**Dehairing and fleecing of skins to be tanned or dressed.** SOC. FINANCIÈRE DE CRÉDIT MOBILIER ET IMMOBILIER (B.P. 355,368, 27.3.30. Fr., 28.3.29).—The skins are dipped into a solution of skin waste in NaOH

or other alkali or alkaline-earth solution, except  $\text{Ca}(\text{OH})_2$ , to which resin soap and  $\text{PhOH}$  or resorcinol have been added, and the whole is kept at  $25^\circ$  for 24 hr.

D. WOODROFFE.

**Tanning processes and apparatus.** G. B. STOOLE (B.P. 354,772, 30.4.30).—The hides etc. are drawn through tanning liquors while hanging parallel to the discs of a rotating apparatus, and are attached at one end only so that they are subjected to bending movements.

H. ROYAL-DAWSON.

**Tanning of hides and the like.** G. V. WRANGE, and AKTIEBOLAGET FRIBERG'S HÖGVACUUMPUMP (B.P. 355,713, 26.5.30. Swed., 28.5.29).—Hides are tanned in two or more closed evacuated tanks, containing tanning liquors of different concentrations, which are cut off from each other, but are provided with separate pumps for circulating the tanning liquors in them or for transferring the liquors from one tank to another.

D. WOODROFFE.

**Treatment of [bark-]tanned leather waste.** ORANIENBURGER CHEM. FABR. A.-G. (B.P. 355,282, 12.5.30. Ger., 13.5.29).—The waste is treated with sulphonic acids of high mol. wt. or their salts, or with aq. solutions of inorg. or org. acids, to which may be added a reducing agent. The product is then precipitated to form a plastic mass.

D. WOODROFFE.

**Rubber cement.**—See XIV.

## XVI.—AGRICULTURE.

**Soils of the Nile and Gash.** III. P. VAGELER and F. ALTEN (Z. Pflanz. Düng., 1931, 22A, 21—51; cf. B., 1931, 939).—Theories of the mineral nutrition of plants are discussed, together with the application to Egyptian soils of recognised methods of determining soil-nutrient values, exchangeable bases, and certain relevant physical properties.

A. G. POLLARD.

**Soil examination.** H. ELLEDER (Z. Pflanz. Düng., 1931, 10B, 417—427; cf. B., 1930, 630).—Numerous methods for determining the P status of soils are compared. The problem of translating results into terms of practical application remains unsolved, and values obtained have a qual. rather than a quant. significance.

A. G. POLLARD.

**Colloid content and related soil factors as indicators of [forest] site quality.** I. T. HAIG (Yale Univ. School Forestry Bull., 1929, 24, 33 pp.).—Soils in the vicinity of New Haven, Conn., U.S.A., were studied. The org. matter content was 2—10% and had no significant influence on soil fertility. CHEMICAL ABSTRACTS.

**Use of hydrogen peroxide for determining humification [in soils or decomposed organic materials].** H. L. RICHARDSON (Soil Sci., 1931, 32, 167—171).—Undecomposed plant materials undergo considerable decomp. on boiling with 6%  $\text{H}_2\text{O}_2$ , the effect being enhanced by the presence of soil. The use of 6%  $\text{H}_2\text{O}_2$  in the examination of partly humified matter can give only approx. results.

A. G. POLLARD.

**Influence of liming chernozem on the yield and composition of wheat.** V. N. KHARCHIKOV (Udobr. Urozhai, 1930, 2, 751—759).—At a  $\text{H}_2\text{O}$  content of 40% of the total  $\text{H}_2\text{O}$ -holding capacity the yield

was sometimes increased by Ca or  $\text{Ca} + \text{P}$ . At 60% there was an increase on the unfertilised plots and on those fertilised with N or P. With  $\text{N} + \text{P}$ , liming gave negative results. The N content of the wheat increased whenever the Ca depressed the yield, and *vice versa*.  $\text{CaO}$  was more effective than  $\text{CaCO}_3$  in connexion with  $\text{NaH}_2\text{PO}_4$  when the amount of phosphate did not exceed the amount added with manure. With  $(\text{NH}_4)_2\text{SO}_4$  the  $\text{CaO}$  produced little effect.  $\text{CaO}$  increased the N and P content of the grain.

CHEMICAL ABSTRACTS.

**Nitrates and ammonium salts as sources of nitrogen for higher plants at constant  $p_{\text{H}}$ .** K. PIERSCHLE (Z. Pflanz. Düng., 1931, 22A, 51—86; cf. A., 1930, 262).—In sand cultures with 8 different crops, the utilisation of  $\text{NH}_3\text{-N}$  and of  $\text{NO}_3'$  in neutral and slightly acid solutions was approx. the same, slight variations occurring with the different plant types. With alkaline and strongly acid solutions,  $\text{NH}_3\text{-N}$  was less easily assimilated. Mixtures of  $\text{NH}_3\text{-N}$ ,  $\text{NO}_3'$ , and urea produced better results than either  $\text{NH}_3\text{-N}$  or  $\text{NO}_3'$  alone. The intake of N, P, and K measured as % of dry matter was not directly related either to the growth rate or to the  $p_{\text{H}}$  of the solution. An increased P intake in the presence of  $\text{NH}_3\text{-N}$  and an increased K intake from  $\text{NO}_3'$  solutions was indicated. The difficulty of ascertaining the optimum nutrient ratios for various  $p_{\text{H}}$  values is emphasised in this type of work.

A. G. POLLARD.

**Effect of nitrogen fertilisers on the yield and quality of sugar beets.** A. G. MIKHALOVSKI (Udobr. Urozhai, 1930, 2, 633—635).—On degraded chernozem  $(\text{NH}_4)_2\text{SO}_4$  was most effective, being followed by urea,  $\text{NH}_4\text{Cl}$ , and Leuna-saltpetre;  $\text{NaNO}_3$  and  $\text{CaCN}_2$  gave negative results.  $\text{NaNO}_3$  and urea increased, whilst Leuna-saltpetre and  $\text{NH}_4\text{Cl}$  decreased, the sugar content.  $\text{CaCN}_2$  and  $\text{NaNO}_3$  gave the greatest amount of melassigenic N. Without P the increase for N was 5%; with P, 15%.

CHEMICAL ABSTRACTS.

**Effect of mineral fertilisers and manure on the quality of sugar beet.** J. A. SIRACHENKO (Nauk Zapiski Tzuk. Prom., 1930, 10, 477—490).—With repeated application of mineral fertilisers the  $p_{\text{H}}$  of the soil decreases. The fertility of the lower layers is increased. In comparison with manure, mineral fertilisers decrease the sucrose and maltose content of the beet.

CHEMICAL ABSTRACTS.

**Manuring pedigree grasses for seed production.** G. E. EVANS and R. A. CALDER (Welsh J. Agric., 1931, 7, 195—208).—Of the common plant nutrients, N was most active in inducing seed production in leafy strains of grasses. P and K applied together with or without N depressed the seed yield from fescues and had varied effects on other species. P and K reduced the number of both barren and fertile tillers, but N increased the production of fertile tillers in many species. Seed yields were greater when N was applied early.

A. G. POLLARD.

**Recovery of nitrogen in pastures from the application of nitrogenous manures. II. Ordinary swards.** T. W. FAGAN and R. O. DAVIES (Welsh J. Agric., 1931, 7, 268—276).—On grass land receiving

periodical dressings of N fertilisers the N recovered in the herbage cut at monthly intervals varied very considerably. Where clover was abundant, application of N suppressed its growth sufficiently to reduce the net N recovery in the crop to a negligible amount. High N recovery was associated with certain upland pastures containing much moss on soils of high org. content. The increased N content of the crop resulting from the use of N fertilisers is obtained partly from the fertiliser itself and partly through the biochemical activities of the soil.

A. G. POLLARD.

**Effect of various manures on the yield of hay and on the botanical composition of the herbage of meadow land.** R. A. and E. J. ROBERTS and I. G. LEWIS (Welsh J. Agric., 1931, 7, 142—155).—Annual dressings of farmyard manure produced greater hay yields than any combination of artificial fertilisers examined or an alternation of farmyard manure and basic slag. Annual applications of basic slag alone reduced yields and when used in conjunction with K and N fertilisers no increase resulted from the slag. K fertilisers definitely increased yields. On land receiving complete artificials, triennial dressings of CaO did not increase the hay crop.

A. G. POLLARD.

**The "many-necked" condition of swedes in relation to varietal and manurial trials.** W. M. DAVIES (Welsh J. Agric., 1931, 7, 319—332).—A significant reduction in the proportion of "many-necked" swedes resulted from early applications of  $\text{NaNO}_3$  (e.g., 1 cwt. per acre in the seed drills). Later treatment with top dressings was ineffective.

A. G. POLLARD.

**Bordeaux mixture as a factor increasing drought injury.** J. D. WILSON and H. A. RUNNELS (Phytopath., 1931, 21, 729—738).—Ginseng plants sprayed with Bordeaux mixture succumbed to drought more readily than unsprayed controls.

A. G. POLLARD.

**Chemical composition of eleven species and strains of grasses at different stages of maturity.** T. W. FAGAN and W. E. J. MILTON (Welsh J. Agric., 1931, 7, 246—255).—With advancing growth the protein and  $\text{H}_3\text{PO}_4$  contents of grasses decrease and there is an increase in the fibre, ash, CaO, and  $\text{SiO}_2$ . These changes are particularly rapid as the grass passes from the pasture to the hay stage, and the significance of this in the making of hay and in the overwintering of pasturage is emphasised.

A. G. POLLARD.

**Action of hydrogen and metallic sulphides on the higher plants.** E. HILTNER (Arb. biol. Reichsanstalt f. Land- u. Forstwirts., 1931, 19, 211—225).—In small proportions  $\text{H}_2\text{S}$  favoured the growth of the aerial portion of peas, although root development was much restricted. Metallic sulphides increased the yield of crops in a number of cases, notably  $\text{MgS}$  on mustard and  $\text{Na}_2\text{S}$  on rye grass and potatoes. In the latter case the growing period of the plant was prolonged. The manurial action of  $\text{NH}_4$  salts was increased where a portion of the N was applied as  $\text{NH}_4\text{HS}$ . In sand cultures, sol. sulphides proved injurious and only  $\text{FeS}$  exerted a favourable effect. In soils, potato yields were considerably increased by  $\text{Na}_2\text{S}$ , but  $\text{K}_2\text{S}$  in equiv. amounts was slightly injurious. The limit of toxicity of sulphides is dependent

on the nature of the associated cation, the nature of the plant, and of the medium in which it is growing.

A. G. POLLARD.

**Decomposition of the green parts of lupin in the soil.** Z. P. USPENSKAYA (Udobr. Urozhai, 1930, 2, 635—638).—Results with millet grown on a podsolised loam are recorded.

CHEMICAL ABSTRACTS.

**Lucerne "inoculation" and the factors affecting its success.** H. G. THORNTON (Imp. Bur. Soil Sci., Tech. Comm., 1931, No. 20. 39 pp.).—A summary and discussion of published work including a consideration of soil conditions as affecting inoculation and nodulation.

A. G. POLLARD.

**Decrease and increase of nicotine in the tobacco plant.** P. KOENIG (Z. Unters. Lebensm., 1931, 62, 87—93).—The nicotine content of green tobacco leaves increases (from 1.61% on the dry tobacco) with ascending height on the stem to a max. (4.45%) and decreases slightly (to 3.75%) for the top leaves. In general, it is decreased by a dry season and if the plant is growing in the shade (e.g., hop-shading), and is increased by N fertilisers and Cl manures. Methods of harvesting also influence it, and diseased plants may have a greater proportion in the leaf than sound plants. During the ripening period the nicotine content may be doubled in 2—3 weeks. The stem contains about 0.3 (lower) to 0.8% (upper), but less when dried; the root contains none, the sap 0.3, and the ripe green seeds 0.2%. On drying, fermentation occurs, and the half-dried leaf may contain more nicotine than the final product. It is considered that the nicotine (Pfyl and Schmitt, B., 1927, 955), protein, and non-protein contents, the colour, aroma, and burning powers are sufficient to characterise a tobacco. German patents for treating tobacco are summarised.

J. GRANT.

**Superphosphate.**—See VII.

See also A., Oct., 1136, Formation of N compounds in soil. 1188, Toxicity of acid Pb arsenate to larvæ of the Colorado potato beetle. 1199, Fertiliser requirement of plants. Effect of manurial treatment on tomato foliage. 1200, P deficiency and the metabolism of the tomato.

PATENTS.

**$\text{H}_3\text{PO}_4$  and  $\text{CaCN}_2$ . Leaching phosphate rock.**—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Manufacture of glucose from manihot root.** A. E. WILLIAMS (Ind. Chem., 1931, 7, 375—377).—The crushed root (from *Manihot utilissima* or *M. aipi*, the latter containing HCN) is boiled with  $\text{H}_2\text{O}$  to eliminate HCN, and heated in a converter under pressure with dil. HCl. The liquid is neutralised by  $\text{Na}_2\text{CO}_3$ , and, after filtration from fibrous matter, is decolorised and conc. in a vac. evaporator. In a second process, the starch is extracted from the coarsely ground root with aq. NaOH and this is treated (instead of the root) as in the first process. The crystallisation of the sugar in the first process is retarded by the high content of inorg. salts, and by mannose derived from the hydrolysis of cellulose.

C. RANKEN.

**Caramel preparation from molasses.** D. J. AKKERMANN (Arch. Suikerind. Nederl.-Indië, 1930, 38, 677—682; Internat. Sugar J., 1931, 33, 245).—Molasses is diluted to  $d$  1.26, subsided, treated with  $\text{SO}_2$  for 3 hr. at  $110^\circ$ , heated for  $\frac{1}{2}$  hr. at the same temp., mixed with 1—4 litres of milk of lime at  $d$  1.15 per hectolitre of the original molasses, and again heated for  $1\frac{1}{2}$  hr. at  $125^\circ$ . The caramelised syrup thus obtained when diluted is suitable for darkening high-polarising sugars.

J. P. OGILVIE.

**Errors in the determination of the tenacity of starch.** A. PARLOW (Z. Spiritusind., 1931, 54, 236—237).—The value of the tenacity of starch solutions is very sensitive to the length of time the solution is retained at  $70$ — $100^\circ$  during heating and cooling. If the starch solution which has been boiled for 3 min. is cooled to  $20^\circ$  in 2 hr., the % tenacity may approximate to 90 less than when the same solution is cooled to  $20^\circ$  in 5—6 min. Similarly, if 1 hr. is required to raise the starch solution to  $100^\circ$ , the % tenacity approximates to 150 less than when heating for a few min. is sufficient to reach  $100^\circ$ . For the determination the starch solution should be raised to  $75^\circ$  on a water-bath and thereafter to  $100^\circ$  over a naked flame. During the 3-min. boil, the temp. must be kept exactly at  $100^\circ$ , and cooling to  $20^\circ$  should be as rapid as possible through the first  $50^\circ$ .

C. RANKEN.

[Starch for] desizing textiles.—See VI. Sugar beet.—See XVI. Detection of benzylidenesorbitol.—See XVIII. Lemonade syrup.—See XIX.

See also A., Oct., 1124, Sol. starch. 1145. Filter tubes [for sugar determinations].

#### PATENTS.

Saccharification of cellulose.—See V.

### XVIII.—FERMENTATION INDUSTRIES.

**Tannin [in brewing]. III. Precipitation of protein in beer by hop tannin.** H. FINK and W. RIEDEL (Woch. Brau., 1931, 48, 391—394. Cf. B., 1930, 212; 1931, 85).—Measured quantities of clear, centrifuged boiled hop extract and of beer were mixed and kept in the dark for 2 days before centrifuging and determining the content of the several forms of protein-N in the clear solution. From these values and the corresponding values in the original hop extract and beer the amounts precipitated by the hop tannin were calc. Of the total precipitated N, 59.2% consisted of amino-N, 25.4% of albumose-N, and 16.9% of coagulable protein-N, whereas no peptone-N was precipitated. Referred to the amounts of the several N compounds originally present in the mixture, the precipitation removed 41.7% of the coagulable protein-N, 25.9% of the amino-N, and 10.3% of the albumose-N.

C. RANKEN.

**Detection of benzylidenesorbitol [obtained] by Werder's sorbitol method, by means of a colour reaction with acetone.** G. REIF (Z. Unters. Lebensm., 1931, 62, 82—86).—The benzylidenesorbitol, prepared using 50%  $\text{H}_2\text{SO}_4$  and PhCHO free from BzOH (B., 1929, 619), is shaken with 100 c.c. of cold  $\text{H}_2\text{O}$ , filtered,

washed according to the size of the residue with 50—200 c.c. of  $\text{H}_2\text{O}$ , then twice with 3—10 c.c. of petroleum spirit containing 30% abs. EtOH at  $0^\circ$ , and finally twice with 10 c.c. of petroleum spirit. It is dried for 1 hr. at  $75^\circ$  and 0.025 g. is shaken with 0.9 c.c. of  $\text{H}_2\text{O}$  and 0.3 c.c. of  $\text{COMe}_2$ , and 0.52 c.c. of  $\text{H}_2\text{SO}_4$  ( $d$  1.84) added from a pipette. PhCHO liberated from benzylidenesorbitol gives an orange-red colour due to dibenzylideneacetone, whilst benzylidenemannitol is insol. and gives a pale yellow colour. The colour is proportional to the amount of benzylidenesorbitol present, and is produced, though slowly, with 0.01 g. (or with 5% of fruit wine in grape wine). Tartaric, malic, citric, oxalic, succinic, cinnamic, lactic, and benzoic acids, tartar,  $\text{CaSO}_4$ , mannitol, sorbitol, dextrose, sucrose, and dextrose give at the most a pale yellow colour, and invert sugar and laevulose a brown tint (B., 1930, 737), whilst certain related ketones, aldehydes, and phenols and  $\text{NH}_2\text{Ph}$  may give the full reaction.

J. GRANT.

**Sauerkraut.** C. S. PEDERSON (New York State Agr. Expt. Sta. Bull., 1931, No. 595, 23 pp.).—A description of the bacteria concerned in the fermentation is followed by a discussion of the effect of NaCl concentration, temp., etc. No satisfactory starter has yet been produced. The causes of various types of spoilage are indicated.

E. B. HUCHES.

**Excœcaria agallocha as a source of power alcohol.** H. K. SEN, S. B. GHOSH, and P. P. PAL (Proc. XV Indian Sci. Cong., 1928, 163).—Simonsen's process affords approx. 40% of reducing sugar; of this about 70% is readily fermentable. CHEMICAL ABSTRACTS.

[Starch for] desizing textiles.—See VI. Marzipan containing "Convertit" etc.—See XVII. Determining EtOH in wines.—See XX.

See also A., Oct., 1154, Yeast sterols. 1188, Honey catalase. Dextrose dehydrogenase. Amylase from rice. Dialysis of cereal amylases. 1190, Stability of malt enzymes.

#### PATENTS.

**Preparing and using materials [hop derivatives] for brewing beer.** G. M. CLARK. From HANSENA A.-G. (B.P. 355,391, 2.6.30).—Air is expelled by passing  $\text{CO}_2$  through compressed dry hops slowly heated to  $70^\circ$  in an extractor. A mixture of steam and  $\text{CO}_2$  is then passed through at  $90$ — $98^\circ$  and distillates containing various grades of hop oil are collected. Moisture absorbed by the hops is removed by the passage of dry  $\text{CO}_2$ , and the hops are pressed and packed into airtight metal cylinders filled with  $\text{CO}_2$ . The hop oil and hops remain stable.

C. RANKEN.

**Control of fermentation, particularly of brewers' wort.** W. SCOTT, R. SELIGMAN, and ALUMINIUM PLANT & VESSEL Co., LTD. (B.P. 355,355, 24.5.30).—The space above fermenting wort is subjected to a pressure higher or lower than that of the atm. The yeast purges sooner with lower pressures, whilst purging is delayed with higher pressures.

C. RANKEN.

Saccharification of cellulose.—See V. Depilating hides.—See XV.



## XIX.—FOODS.

**"Rope" spore content of flour and its significance.** A. J. AMOS and D. W. KENT-JONES (Analyst, 1931, 56, 572—586).—The authors recommend a modification of the method of Hoffman (B., 1930, 583) and extracting 20 g. of flour with 400 c.c. of  $H_2O$  and 20 g. of sand in a glass-stoppered bottle to obtain an even distribution of spores of the *B. mesentericus* group. The spores are not destroyed by ordinary dry-heat sterilisation, and treatment of the apparatus with  $HgCl_2$  solution is employed. The no. of spores in a flour is no criterion as to whether the resulting bread will be ropy or not; this is dependent on the technique employed in the bakery. Long systems of fermentation, low temps. in doughs, sparing use of yeast, slack doughs, insufficient baking, and slow cooling of the bread tend to aggravate rope trouble. T. McLACHLAN.

**Measurement of flour colour by the Pulfrich photometer.** R. RÜTER (Mühlenlab., 1931, No. 4, 26—35).—This instrument is not sufficiently sensitive for measuring the colour of dry flour. Whilst it is more useful in the Pekar test, the results are difficult to reproduce owing to numerous factors beyond control. The relation of colour to fat content and to flour grade has been studied photometrically. A method is described whereby the carotene content can be determined photometrically without using a standard solution. The author prefers the Pulfrich photometer to that devised by Kent-Jones. Comparison of the crumb colours of loaves made from the flours is difficult owing to the effect of differences in texture. W. J. BOYD.

**Choice of methods for the determination of fat in sterilised milk.** F. E. NOTTBOHM and O. BAUMANN (Z. Unters. Lebensm., 1931, 62, 164—169).—Recommended methods (Vereins Deutscher Lebensmittelchemiker) are: (1) For unsweetened condensed, dried, and homogenised milks and for cheese (after Schmid-Bondzynski): 5 g. of liquid (1 g. of solid) sample are heated in a flask with 10 c.c. of boiling  $HCl$  ( $d$  1.124) for 20 min., and the warm liquid is mixed with 10 c.c. of  $EtOH$  and 25 c.c. of  $Et_2O$  in a Röse-Gottlieb tube and extracted with 25 c.c. of petroleum spirit, an aliquot portion of the extract being evaporated in a tared flask and weighed. (2) For sweetened milks (after Weibull): a solution of 10 g. in 20 c.c. of  $H_2O$  and 30 c.c. of  $HCl$  ( $d$  1.124) are boiled for 20 min., 50 c.c. of  $H_2O$  added, and the residue after filtration and removal of  $HCl$  in hot  $H_2O$  is dried at  $100^\circ$  and extracted with  $Et_2O$  for 1 hr. in a Soxhlet apparatus. Method (1) gives higher results than the Röse-Gottlieb method for full and skim condensed milks, and similar results for cream and milk powder. J. GRANT.

**Van Slyke method for protein analysis as affected by fats.** S. M. HAUGE (J. Amer. Chem. Soc., 1931, 53, 3049—3052).—Increases in acid-sol. humin, phosphotungstic acid humin, and non-amino-N, and decreases in amino-N and arginine val. are found when casein (Hammersten) admixed with butter fat or glycerol is analysed by the Van Slyke method. With gelatin in presence of glycerol, decreases in amino-N and arginine are found. Carnauba wax or stearic (and oleic) acid

has little or no effect. Hydrolysis of tyrosine or tryptophan in presence of glycerol causes increased formation of acid-sol. humin. H. BURTON.

**Cryoscopy [in milk analysis].** S. ROTHENFUSSER (Z. Unters. Lebensm., 1931, 62, 219—221).—Refractometric methods are preferable for the detection of added  $H_2O$  in milk, but cryoscopic methods are recommended when it is desired to establish the absence of added  $H_2O$ . Details of technique are given, and a tapping apparatus (the "cryohammer") is described which consists essentially of a soft rubber cylinder mounted on a spiral support. It is intended to overcome the disadvantages of adjusting the Beckmann thermometer by striking with the finger. J. GRANT.

**Detection of the heating of milk.** S. ROTHENFUSSER (Z. Unters. Lebensm., 1931, 62, 210—214).—In applying the author's method (B., 1931, 315), 5 c.c. of milk may be placed in a cylinder, and a glass tube carrying a loosely-fitting central rod, the bottom of which is a fairly close fit and is ground, is dipped below the surface of the milk. Solid reagent is inserted in the open, top interstitial space and added in small quantities as required by rotating the central rod, the whole being mixed by shaking and any development of colour observed. J. GRANT.

**Detection of added water in milk and other substances.** B. DAVIES (J. Sci. Instr., 1931, 8, 160—164).—Cells containing standard milk (etc.) and the sample under test form two arms of an a.c. Wheatstone bridge. The sp. resistance of pure milk from individual cows and of average milk from a herd varies largely from day to day, rendering the detection of added  $H_2O$  difficult. C. W. GIBBY.

**Supplements to a milk diet for dairy calves.** C. Y. CANNON (Iowa Agric. Exp. Sta. Res. Bull., 1931, No. 136, 67—103).—Lucerne hay and straw markedly reduced rickets and anemia in calves receiving a whole-milk ration. The use of lucerne and grain for a period prior to feeding a whole-milk ration allowed a sufficient storage of Fe to prolong the period of apparent good health. Calves on milk diets showed greater skeletal development and larger live-wt. increases than normal. A. G. POLLARD.

**Dried buttermilk for growing and fattening pigs.** J. M. EVVARD, C. C. CULBERTSON, and Q. W. WALLACE (Iowa Agric. Exp. Sta. Bull., 1931, No. 278, 121—148).—The inclusion of dried buttermilk in pig rations produced satisfactory growth and decreased the equiv. feed required per unit live-wt. increase. The proteins of buttermilk are more efficient than tankage proteins. Analyses and results of feeding trials are recorded. A. G. POLLARD.

**Factors affecting the viscosity of cream.** C. J. BABCOCK (U.S. Dept. Agric. Tech. Bull., 1931, No. 249, 19 pp.).—The viscosity of cream increases with the butter fat content, with the % of solids-not-fat, and also with falling temp. Pasteurisation lowers the viscosity; this rises subsequently, but to a less extent than that of raw cream. Standardising cream to a sp. butter fat content does not affect its viscosity. Freezing lowers the viscosity, which does not return to the initial value on storage. Acidity, unless  $>0.3\%$ , has little effect

on viscosity. Homogenisation of cream increases its viscosity in direct relation to the pressure used.

A. G. POLLARD.

**Evaluation of mineral waters and artificial mineral waters.** J. TILLMANS (Z. Unters. Lebensm., 1931, 62, 138—147, 156—160).—The conclusions of the Verein Deutscher Nahrungsmittelchemiker and representatives of the industries concerned (Jan., 1931) were discussed and revised. They deal with the salt and gas contents of the products and with their description, bottling, etc. Proposed limits for mineral waters are: not less than 1 g. of total solids/kg. (or 1 mg. Li<sup>+</sup>, I<sup>-</sup>, HAsO<sub>2</sub>, and total S; 1.3 mg. HAsO<sub>4</sub>; 2 mg. F<sup>-</sup>; 5 mg. Br<sup>-</sup> and HBrO<sub>2</sub>; 10 mg. Sr<sup>++</sup>; total Fe and Mn; alkalinity 4 mg.-equiv.); CO<sub>2</sub> 0.25 g.; radon 50 Mache units.

J. GRANT.

**Evaluation of effervescing lemonades and effervescing lemonade syrups.** A. BEYTHIEN (Z. Unters. Lebensm., 1931, 62, 221—228, 239—242).—The rules drawn up by the Verein Deutscher Nahrungsmittelchemiker and representatives of the industries concerned (Dec., 1930) were discussed and revised. They refer to the description of the beverages, and to their natural and chemical constituents. At least 7% of sugar should be present in effervescing lemonades and 60% in syrups. Starch sugars and syrups, foaming agents, and harmful preservatives are forbidden. J. GRANT.

**Acids in edible fats.**—See XII. Sauerkraut.—See XVIII.

See also A., Oct., 1178, Beef tallow. EtOH-sol. proteins from milk products. 1188, Honey catalase. Amylase from rice. Dialysis of cereal amylases. 1194—7, Vitamins [various]. 1198, Phosphatide from carrots. 1199, Karité butter. 1200, Determination of SiO<sub>2</sub> in vegetables.

#### PATENTS.

**Milk or cream preparation.** W. O. FROHRING (B.P. 354,535, 29.7.30).—Whole milk is separated, and to the cream is added Na caseinate (to about 1% on the wt. of final product). The fat content (usually about 40%) is reduced to about 18% by addition of whole milk or skim milk, and the mixture is then pasteurised, homogenised, tinned, and sterilised. The product may be used direct from the tin without dilution.

E. B. HUGHES.

**Whipping cream preparation.** W. O. FROHRING (B.P. 355,913, 30.7.30).—Cream containing 40% of fat is mixed with 1% of Na caseinate, pasteurised, homogenised at a pressure not above 750 lb./sq. in. and then packed in tin cans. The product whips readily.

E. B. HUGHES.

**Manufacture of butter and margarine.** E. LANGFELDT (B.P. 354,187, 30.4.30. Holl., 1.5.29).—In order to retain their characteristic flavour, these products are prepared by incorporating a mixture of lactic and tartaric acids and H<sub>3</sub>PO<sub>4</sub> so that the final *p*<sub>H</sub> of the aq. phase is 3—5.

E. B. HUGHES.

**Preparation of fresh fruit [and vegetables] for market.** E. M. BROGDEN and M. L. TROWBRIDGE, Assrs. to BROGDEN Co. (U.S.P. 1,795,275 and 1,794,346, [A] 3.3.31, [B] 24.2.31. Appl., [A] 18.1.26, [B] 2.3.25).—The usual 5—20% loss in shipping and storage of citrus

fruits etc., largely due to blue mould, is prevented by washing at 46° with a solution of alkaline hydroxide or Na<sub>2</sub>CO<sub>3</sub>. An application of a film of wax gives further immunity. Suitable apparatus is described.

E. B. HUGHES.

**Acceleration of meat-pickling processes.** J. GERT and S. R. FRITZ (GERT & Co.) (B.P. 356,000, 8.10.30. Austr., 4.4.30).—About 2% of H<sub>3</sub>PO<sub>2</sub> or H<sub>3</sub>PO<sub>3</sub> or their salts is added to the pickling-salt mixtures (e.g., NaCl, KNO<sub>3</sub>, sugar).

II. ROYAL-DAWSON.

**Modified [chewing] gum and its manufacture.** C. E. DELLENBARGER, Assr. to P. CARPENTER (U.S.P. 1,786,831, 30.12.30. Appl., 24.4.22).—A mixture of paraffin wax and rubber is heated, e.g., to 145°, whereby a bland, tasteless mass is obtained, and the consistency is then adjusted by addition of resinous material such as pontianak or ester gum and a bituminous hydrocarbon. Cocoa or chocolate can be used as a flavouring material, although not applicable to gum chicle. D. F. TWISS.

**Milling of cereals.** F. STACEY (B.P. 356,789, 13.5.30).

**[Cooking] ovens, particularly of the continuous working or conveyor type [for food products].** BRIT. THOMSON-HOUSTON Co., LTD., Asses. of J. L. SHROYER (B.P. 356,101, 1.4.30. U.S., 1.4.29).

**[Funnel-shaped] milk coolers.** N. E. H. HJORTH (B.P. 356,636, 30.1.31. Denm., 1.2.30).

**Refrigerating apparatus particularly for cooling milk.** PULSOMETER ENG. Co., LTD., and J. B. CLEWS (B.P. 356,336, 19.6.30).

**Drying of milk. Drying of tea. Cooling of liquids. Grinding of chocolate.**—See I.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Indirect determination of residue and alcohol content in tinctures and wines.** H. TER WEE and P. VAN DER WIELEN (Pharm. Weekblad, 1931, 68, 900—921).—The method is based on the determination of the refractive index of the liquid. Graphical methods of interpreting the results and of making the necessary allowance for the influence of the solute are described. Data are given for a number of tinctures and wines.

H. F. GILLBE.

**Medicines for injection. II. Decomposition of novocaine solutions during sterilising and keeping.** J. ABILDGAARD (Dansk Tidsskr. Farm., 1931, 5, 129—142).—Novocaine solution (2%) in 0.001*N*-HCl undergoes no appreciable decomp. either after 30 min. steam-sterilisation at 100° or by 20 min. autoclaving at 120°. Under similar conditions, using a 1% aq. sol., 2% and 5% of the novocaine, decomposes and in presence of a phosphate buffer, at *p*<sub>H</sub> 5.5 and 6.5, the respective amounts of decomp. are 6%, 8% and 19%, 35%. A series of commercial solutions, 1—5 years old, showed good agreement between the found and declared novocaine contents. In most samples about 2% decomp. had occurred. The decomp. products were determined by addition of NaHCO<sub>3</sub> and extraction with Pr<sup>2</sup>OH—CHCl<sub>3</sub> (1:3) mixture, which extracted novocaine and diethylaminoethanol. This extract was shaken with 0.1*N*-HCl and excess acid titrated with 0.1*N*-NaOH,

using methyl-red and methylene-blue. The HCl absorbed was equiv. to the novocaine originally present. The residual  $\text{NaHCO}_3$  liquor, after adjustment of the  $p_{\text{H}}$  to 3.3–3.5, was extracted with the same solvent and the extracted *p*-aminobenzoic acid, after removal of the solvent, was titrated with 0.1*N*-NaOH. The difference between the two titrations gave the actual amount of novocaine present.

E. H. SHARPLES.

**Nicotine content of tobacco.** PETRI (Z. Unters. Lebensm., 1931, 62, 126–128).—Recommendations as follows are made to the present (German) Committee of manufacturers: (1) "Nicotine-free" tobacco should contain not more than 0.1% of nicotine (on the dry tobacco). (2) "Poor in nicotine" should denote a nicotine content of  $\frac{1}{2}$  the normal amount (e.g., not more than 0.75% for cigars and 0.5% for cigarettes or cut tobacco, on the dry substance). (3) Other descriptions (e.g., "denicotinised") should be rejected as misleading; this applies also to descriptions referring to the nicotine content of the smoke, since this is dependent on the method of analysis. (4) Pfyl and Schmitt's method of analysis (B., 1927, 955) is recommended. No distinction is apparent between treated tobaccos and those naturally poor in nicotine.

J. GRANT.

**Nicotine content of tobacco smoke.** C. PYRIKI (Z. Unters. Lebensm., 1931, 62, 95–98).—Oriental tobaccos were blended to give the mixtures containing 0.06–1.08% nicotine, which were made into cigarettes, 6.5 cm. long and 5.2 cm., artificially smoked with pauses of 6 sec. every 4 sec. The apparent nicotine content of the smoke (Pfyl and Schmitt, B., 1927, 955) was equal to that of the blank (5 mg./100 g. of dry tobacco) on nicotine-free tobacco for tobaccos containing up to 0.3 and 0.5% of nicotine, smoked as described in 5 and 8 min. respectively. For 0.6% in the tobacco the respective amounts in the smoke were 55 and 39; for 1%, 235 and 174 mg./100 g. of dry tobacco. The proposed limiting nicotine contents for cigarette tobaccos described as "nicotine-free" (0.08%) and "poor in nicotine" (0.20%) are therefore unduly low, and 0.1–0.15 and 0.5–0.6% are suggested, respectively.

**Determination of mercury content of grey mercury ointment.** L. W. WINKLER (Pharm. Zentr., 1931, 72, 609–610).—1 g. of the sample is dissolved in  $\text{CCl}_4$  (5 c.c.), 10 c.c. of 50%  $\text{HNO}_3$  are added, and, when all the Hg has dissolved, 25 c.c. of  $\text{H}_2\text{O}$  are added, followed by so much  $\text{KMnO}_4$  solution (1:19) that the mixture remains red after vigorous shaking.  $\text{FeSO}_4$  is then added in small quantities at a time until the solution is colourless and, after the introduction of 0.5 g. of cryst.  $\text{Fe}(\text{NO}_3)_3$ , the liquid is titrated with 0.1*N*- $\text{NH}_4\text{CNS}$  (1 c.c.  $\equiv$  1% Hg).

E. H. SHARPLES.

**Al(OAc)<sub>3</sub>.**—See VII. Tobacco plant.—See XVI.

See also A., Oct., 1138, Measurement of physiologically active ultra-violet radiation. 1152, "Musc ambrette." 1162, Polyhydroxy-chalkones, -hydrochalkones, and -flavanones. 1167, Derivatives of substituted cinchonic acids. 1169, Murexide reaction. 1171, Cocaine and allylcocaine in narcotic poisons. Physiologically active constituents of yew. Alkaloids of ergot. Behaviour of solanine on microsublimation. Yohimbine. 1172, Action

of sol. iodides on strychnine sulphate. Alkaloids of gelsemium. 1174, Narceine. 1194, Cryst. insulin. Cryst. female sexual hormone. 1194–7, Vitamins (various). 1198, Phosphatide from carrots. 1199, *Senecio aureus*. Ephedrine from yew leaves. 1201, Mosaic disease of tobacco.

#### PATENT.

**Manufacture of aminoalkoxy-compounds useful as antiseptics.** SOC. CHEM. IND. IN BASLE (B.P. 351,605, 7.4.30. Switz., 5.4.29).—Antiseptics are obtained by introducing halogen into aminoalkoxy-derivatives of the aromatic or quinoline series. Examples are:  $\beta$ -diethylaminoethyl ethers of 2-chlorothymol, b.p. 142–143°/3 mm., 2:4:6-tribromophenol, b.p. 170–171°/3 mm. (hydrochloride, m.p. 163–164°), bromo- $\alpha$ -naphthol, m.p. 179–180° (hydrobromide, m.p. 174–175°), 4-chloro-*o*-aminophenol, b.p. 158–160°/3 mm., 5-chloro-7-iodo-8-hydroxyquinoline (B,HCl, m.p. 187–188°; B,2HCl, m.p. 153–154°), 5:6:7-trichloro-8-hydroxyquinoline (B,2HCl, m.p. 134–135°), 7-chloro-2-hydroxy-4-methylquinoline (B,2HCl, m.p. 154–155°; methanesulphonate, m.p. 133–135°), 5-chloro-8-hydroxyquinoline, b.p. 165–169°/0.6 mm. (B,HCl, m.p. 191–193°), 5-bromo-8-hydroxyquinoline (B,2HCl, m.p. 158–159°), 5-chloro-7-bromo-8-hydroxyquinoline (B,2HCl, m.p. 142–143°), 5-bromo-7-iodo-8-hydroxyquinoline (B,2HCl, m.p. 142–143°), 5:7-dibromo-8-hydroxyquinoline (B,2HCl, m.p. 165–166°), and 5:7-diiodo-8-hydroxyquinoline (B,2HCl, m.p. 168–169°);  $\beta$ -dicyclohexylaminoethyl ethers of 4-chlorophenol (B,HCl, m.p. 198–199°) and 5-chloro-8-hydroxyquinoline (B,2HCl, m.p. 239–240°) are also prepared.

C. HOLLINS.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Development of a latent image on pure [untreated] paper.** R. E. LIESEGANG (Z. wiss. Phot., 1931, 30, 156–157).—The resin in the sizing of ordinary paper, on exposure to strong light, produces oxidising compounds, and the latent image is developable with  $\text{FeSO}_4$  and gallic acid, the  $\text{Fe}^{+++}$  salt being formed. Long exposure enables "watermark" images to be developed with NaOH solution.

J. LEWKOWITSCH.

**Photographic printing processes by means of hardened gel layers dehardened by light.** E. STENGER (Z. wiss. Phot., 1931, 30, 108–112; cf. Phot. Ind., 1925, 23, 429).—A discussion is given of all types of pigment processes applicable to gelatin or gum arabic containing tartaric acid and  $\text{FeCl}_3$ , which hardens the layer, but is reduced by light to  $\text{FeCl}_2$ , thereby dehardening the gelatin.

J. LEWKOWITSCH.

See A., Oct., 1109 and 1110, Cu salts as light filters. 1136, Study of the photographic process. 1137, Latent image. Sensitisation.

#### PATENTS.

**Manufacture of gelatin films for imbibition printing.** TECHNICAL MOTION PICTURE CORP., and I. G. FARBENIND. A.-G., Assecs. of W. H. CARSON and L. T. TROLAND (B.P. 356,243, 2.6.30. U.S., 10.6.29).—The celluloid base is coated twice: (a) with gelatin emulsion,

(b) with hardening (e.g., chrome alum) solution, preferably containing gelatin and saponin. A uniformly hardened surface is obtained. J. LEWKOWITSCH.

**Production by photography of composite sound-record films.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 356,237, 29.5.30. Ger., 30.5.29).

**[Synchronisation marking of] sound and picture films.** I. G. FARBENIND. A.-G. (B.P. 354,941, 1.7.30. Ger., 1.7.29. Addn. to B.P. 354,455).

## XXII.—EXPLOSIVES; MATCHES.

**Effect of impurities on the f.p. of glyceryl nitrate.** A. SAPOSCHNIKOV and K. SNITKO (J. Appl. Chem., Russ., 1930, 3, 1077—1085).—The isomerides have f.p. 12.2—12.3° and 1.3—1.5°, respectively; use of  $\text{NaNO}_3$  affords the former, and of  $\text{KNO}_3$  the latter. The f.p. may be lowered by addition of the dinitrate; that of dinitrochlorohydrin or tetranitrodiglycerol has only a slight effect. CHEMICAL ABSTRACTS.

### PATENTS.

**Moisture-proof match-igniting composition and matches prepared therewith.** S. II. LEDIN (B.P. 355,901, 15.7.30. Swed., 20.7.29).—The binding agent comprises a solution containing 8—15% of cellulose or cellulose derivatives and a natural or artificial resin. In addition to the chlorate, there is added a readily sol. metal salt, e.g., dichromate, having the same positive ion as the chlorate, to reduce dissociation and solubility in  $\text{H}_2\text{O}$  of the latter.  $\text{PbO}_2$  is employed as an additional O carrier. To secure good adhesion of the coating, the match-head is coated first with the binding agent. [Stat. ref.] W. J. WRIGHT.

**Firedamp-proof blasting time fuse.** E. KAROLLUS (B.P. 356,511, 29.9.30. Austr., 30.9.29).

**Cellulose nitrate.**—See V.

## XXIII.—SANITATION; WATER PURIFICATION.

**Moulds successfully deodorise undigested sewage sludge.** C. G. WIGLEY (Eng. News-Rec., 1931, 107, 448).—Fresh sewage solids at Atlantic City can be air-dried on shallow sand beds without nuisance. Part of this air-dried material is crushed and stored in shallow rows in a dark building; moulds quickly develop and the cured deodorised material can be sold for \$20 a ton (cf. U.S.P. 1,730,489; B., 1929, 998). C. JEPSON.

**Ammonia-chlorine [water] treatment at Cleveland.** W. C. LAWRENCE (J. Amer. Water Works' Assoc., 1931, 23, 1382—1387).—When this treatment was applied at Cleveland, no increase in the amount of  $\text{Cl}_2$  previously applied was necessary when  $\text{NH}_3$  was added to produce as good a bacterial effect as when  $\text{Cl}_2$  was used alone; the cost of disinfection increased from 22 to 34 cents per million gals. Satisfactory results have been obtained as no chlorophenolic nor chlorinous tastes have been observed, even with residuals as high as 0.55 p.p.m., efficient sterilisation has been maintained, and as the min. residual throughout the distribution system is 0.1 p.p.m., aftergrowths and algae have been eliminated. C. JEPSON.

**Ammonia-chlorine [water] treatment at Beaver Falls and New Brighton, Pa.** E. C. GOEHRING (J. Amer. Water Works' Assoc., 1931, 23, 1372—1381).—

Comparative trials with two plants situated on the same stream and 3 miles apart have substantiated the claims made for this treatment, viz., more efficient sterilisation than with  $\text{Cl}_2$  alone, given sufficient contact period; minimisation of chlorophenolic tastes and odours; and prevention of algal growths in settlement basins and filters when applied to raw water. Application of the dose to raw water is advisable when phenols are absent and when the  $\text{Cl}_2$  demand of the raw water is generally low, but if this demand is high, as when phenols are present, it is better to apply it to the filter effluent, allowing sufficient detention for sterilisation before distribution. C. JEPSON.

**Bactericidal efficiency of the ammonia-chlorine treatment [for water].** H. H. GERSTEIN (J. Amer. Water Works' Assoc., 1931, 23, 1334—1356).—When used with Chicago water from Lake Michigan ( $\text{pH}$  7.8—8.0), the  $\text{NH}_3$ - $\text{Cl}_2$  treatment is effective in preventing the chlorophenolic tastes produced when  $\text{Cl}_2$  is used alone. There is, however, a lag period in the bactericidal effect, increasing with increased proportion of  $\text{NH}_3$ , so that contact for 2 hr. is advisable, as low proportions of  $\text{NH}_3$ , which give rapid bactericidal action, do not prevent the chlorophenolic tastes. The efficiency of the bactericidal treatment also decreases with fall in temp. and increased turbidity of the raw water. C. JEPSON.

**Ammonium salts in taste elimination [from water].** H. E. JORDAN (J. Amer. Water Works' Assoc., 1931, 23, 1366—1371).—At Indianapolis, for reasons of economy,  $(\text{NH}_4)_2\text{SO}_4$  has been substituted for anhyd.  $\text{NH}_3$ , the phenolic content being too low to produce objectionable tastes. The salt is fed in solution along with  $\text{Al}_2(\text{SO}_4)_3$  about 10 sec. before the pre- $\text{Cl}_2$  dose is applied, the total  $\text{Cl}_2$ - $\text{NH}_3$  ratio being 2.25 to 1. The effects of pre-ammoniation are not limited by the presence of free  $\text{NH}_3$ , but an increased  $\text{Cl}_2$  tolerance is found in the finished water whether  $\text{NH}_3$  be present or not in the final stage. C. JEPSON.

**Chloroamines.**—See VII.

See also A., Oct., 1144, "Distilled water" by electro-osmosis. 1188, Industrial poisons. 1192, Improved ferrocyanide-citrate agar. Tests for colon-aërogenes.

### PATENTS.

**Sewage-purifying device.** H. E. ELROD (B.P. 356,401, 25.7.30).—A form of aeration chamber, suitable for the activated-sludge process, is claimed in which economy in power consumption is secured by introducing the air at some distance above the floor of the tank. The stream of air bubbles is separated from the main body of liquid by a baffle which extends from the surface almost to the bottom and thus an air-lift effect is obtained which circulates the tank contents, especially if the upward stream of sewage and air be directed across the surface of the tank by means of a suitably placed deflector. C. JEPSON.

**Breathing apparatus.** DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of HANSEATISCHE APPARATEBAU GES. FORM. L. VON BREMEN & Co. (B.P. 356,547, 23.10.30. Ger., 11.12.29).

**Antiseptics.**—See XX.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 20 and 27, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Formation and growth of crystals.** W. E. GIBBS (Trans. Inst. Chem. Eng., 1930, 8, 38—56).

**Rôle of surface energy in chemical engineering.** W. E. GIBBS (Proc. Chem. Eng. Group, 1929, 11, 20—34).

**Design and operation of gas-heated furnaces.** C. M. WALTER (Proc. Chem. Eng. Group, 1929, 11, 35—43).

**Insulation of heated and cooled surfaces.** J. S. F. GARD and R. F. ROBINSON (Proc. Chem. Eng. Group, 1930, 12, 96—112).

**Heat-transfer calculations, including recent methods of correlating convection data.** O. A. SAUNDERS (Trans. Inst. Chem. Eng., 1930, 8, 209—213).

**High-pressure reactions.** W. A. BONE (Trans. Inst. Chem. Eng., 1930, 8, 98—106).

**Hardness of water.**—See XXIII.

### PATENTS.

**Rotary-hearth furnace.** E. G. DE CORIOLIS, J. R. MOSER, and A. L. LARSEN, Assrs. to SURFACE COMBUSTION CORP. (U.S.P. 1,796,144, 10.3.31. Appl., 26.4.29).—The furnace is provided with a removable roof so that the hearth, and the supports and mechanism for the hearth, can be removed vertically in turn. Provision is made to admit gases through a hollow vertical shaft. B. M. VENABLES.

**Rotary kiln.** L. S. PETERSEN, Assr. to F. L. SMITH & Co. (U.S.P. 1,796,664, 17.3.31. Appl., 7.8.29).—A method of securing transverse chains in a cement or other kiln is described; metallic blocks are buried in the joints between the ceramic lining blocks or bricks. B. M. VENABLES.

**Rotary dryer.** W. A. HARTY and F. W. MOORE, Assrs. to HARMOR & Co., Inc. (U.S.P. 1,796,651, 17.3.31. Appl., 24.10.27).—A rotary dryer having a heated axial flue has the latter flexibly supported by radial rods extending through the wall of the dryer and secured by external springs. After passing through the axial flue, the products of combustion are passed backwards through the dryer in direct contact with the material. B. M. VENABLES.

**Furnace structures.** J. E. KENNEDY (B.P. 356,819, 14.6.30).—The wall of a furnace, *e.g.*, a boiler furnace, is divided into panels each composed of bricks supported on metallic brackets and frames. B. M. VENABLES.

**Increasing the durability of linings of furnaces or ovens and the like.** F. J. MALHERBE (U.S.P. 1,797,780, 24.3.31. Appl., 20.7.28. Belg., 29.7.27).—The linings etc., especially the soles of flattening furnaces for reheating

glass sheets, are impregnated with an emulsion containing K and/or Na silicate and petroleum. L. A. COLES.

**Handling of furnace residues.** T. G. COCHLAN (B.P. 356,733, 3.4.30. U.S., 22.4.29).—Comminuted ash or fused slag is moistened and/or disintegrated by a primary supply of liquid which transports it along a sluiceway to an ejector device in which a secondary supply of liquid enters the stream at an angle of about 22.5° to the axis and suffices to transport the material any reasonable distance through a pipe. [Stat. ref.] B. M. VENABLES.

**Thermal controlling system.** C. C. BOARDMAN, Assr. to THERMATOMIC CARBON Co. (U.S.P. 1,797,256, 24.3.31. Appl., 24.9.27).—When a number of gases are generated in separate furnaces and a mixture is desired at a const. temp., the gases are individually cooled, first, *e.g.*, by water sprays, under manual control, and again before mixing by thermostatically controlled means, the thermostat being situated in the receiver for the mixture. B. M. VENABLES.

**Centrifugal dryer.** C. O. LAVETT (U.S.P. 1,795,495, 10.3.31. Appl., 31.10.28).—The material is spun in a basket with open ends and having hollow trunnions through which extends a hollow shaft driven at a different speed and carrying rakes to discharge the collected solid matter. Washing liquid is admitted through one and heating medium through the other end of the hollow shaft to distributing devices within the basket. B. M. VENABLES.

**Fluid separators.** (A) H. E. BOYRIE, (B) G. D. BRADSHAW and R. N. ROBERTSON, [A, B] Assrs. to BLAW-KNOX Co. (U.S.P. 1,796,434—5, 17.3.31. Appl., [A] 2.1.25, [B] 20.5.27).—Modifications to the steam separator described in U.S.P. 1,381,767 are made. B. M. VENABLES.

**Making heat exchangers.** C. STEENSTRUP, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,797,030, 17.3.31. Appl., 15.11.29).—An air-cooling device which may be used as the condenser in a refrigerating machine is described. B. M. VENABLES.

**Aqueous or aqueous-alcoholic solutions suitable for use in cooling systems, storage vessels, etc.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 352,311, 5.9.30. U.S., 7.9.29).—An aminocarboxylic acid or sol. salt thereof (phenylglycine, anthranilic acid, hydrolysed casein) is added to aq. alcohols, glycol, etc. to prevent the rusting of iron containers. The addition also of ore-flotation collecting agents, *e.g.*, 2-thiolbenzthiazole, xanthates, benzyl mercaptan, thiocresol, diisomylthiocarbamide, thioformaldehyde, etc., prevents discoloration of such liquids in presence of Cu or brass. C. HOLLINS.

\* The remainder of this set of Abstracts will appear in next week's issue.

**Pulveriser.** C. R. TAYLOR, ASSR. to GRINDLE FUEL EQUIPMENT CO. (U.S.P. 1,796,545, 17.3.31. Appl., 11.10.29).—In a pulveriser having a vertical shaft and comprising a fan chamber above a beater chamber, the two chambers are connected by a comparatively small neck in which are provided subsidiary rotating vanes to prevent coarse particles being drawn upwards; the feed of raw material is around the neck, and additional air is admitted through adjustable openings in the upper end of the fan. B. M. VENABLES.

**Pneumatic separator for pulverising mills.** W. L. and H. L. McLAUGHLIN (U.S.P. 1,797,434, 24.3.31. Appl., 19.12.27).—In a conduit for an upward air-borne stream of pulverised material is placed a trough-shaped collector to which are attached shoots leading to the walls of the casing above the pulverising zone, the coarse particles which have been lifted in the middle of the conduit being able to slide down the walls where the current is less. The classification of the material is effected by the stream of air dividing into two while passing the trough and reuniting above it. B. M. VENABLES.

**Mixer.** A. MOYER (U.S.P. 1,796,659, 17.3.31. Appl., 18.1.30).—A mixer for stiff mixtures, *e.g.*, concrete, comprises a vertical fixed receptacle in which are rotated an outer worm, driving the material downwardly and inwardly, and an inner worm of the same hand but opposite rotation, driving the material upwardly and outwardly. B. M. VENABLES.

**Submerged (A) drum filter, (B) continuous [disc] filter.** [A] E. J. SWEETLAND, [B] E. J. SWEETLAND, J. V. ZENTHOEFER, and J. T. HOYT, [A, B] ASSRS. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,796,491—2, 17.3.31. Appl., [A] 24.12.26, [B] 16.2.27).—The filters are operated entirely submerged. No attempt is made to obtain a solid cake, but the thick pulp is blown off a short arc or single segment (as the case may be) at the top of the rotation. Partitions are provided to separate the thick pulp from the prefilter as far as possible, and the former is removed by a conveyor. B. M. VENABLES.

**Filter element.** W. W. NUGENT (U.S.P. 1,796,532, 17.3.31. Appl., 18.10.26. Renewed 4.11.29).—Filter bags are operated with outward flow; they are attached to frames at the mouth, which is sufficiently wide for the bags to be turned inside out for cleaning, and elsewhere are supported by U-shaped rods. B. M. VENABLES.

**Filter.** A. BOULADE, ASSR. to SOC. DU CARBURATEUR ZENITH (U.S.P. 1,797,399, 24.3.31. Appl., 27.12.26).—A stack of filter elements is assembled loosely on metal strips and the ends of the latter are bent over, forming a bundle in which every element is easily cleaned. When placed in a housing the elements are clamped together, forming an edge-filter. B. M. VENABLES.

**[Rotating-leaf] filters.** E. W. W. KEENE (B.P. 356,806, 13.6.30).—The leaves of the filter are fixed at an angle to the axis, and the casing is provided with doors in the end for inspection and in the circumference for changing the leaves. B. M. VENABLES.

**Separation of [dissolved] air from liquids.** W. S. ELLIOTT (U.S.P. 1,797,405, 24.3.31. Appl.,

17.2.23).—The liquid, *e.g.*, make-up water for boilers, is treated by direct contact with steam (or its own vapour) in two chambers in succession. In the upper chamber the temp. is maintained at a predetermined const. val., usually the b.p., and in the lower the steam is bubbled under the water in sufficient vol. to reduce the partial pressure of the harmful gases to a very small figure, the mixed gases being allowed to escape. B. M. VENABLES.

**Atomising or disintegrating liquids, and apparatus therefor.** A. J. ADAMS (B.P. 356,720, 5.4.30).—An apparatus producing a conical spray diverging from the axis of a cylindrical stream of air is described. The spray is operated by air from a bellows driven by the same motor that drives the fan. B. M. VENABLES.

**Evaporator.** B. C. SPRAGUE, ASSR. to L. E. HANKISON (U.S.P. 1,795,743, 10.3.31. Appl., 27.7.26).—The scale-forming liquid which is to be evaporated is caused to flow successively through a number of compartments of a vessel having a common vapour space and common supply of steam or other heating fluid. B. M. VENABLES.

**Evaporation of liquids in films.** CHEM. FABR. BUDENHEIM A.-G. (B.P. 357,050, 24.1.31. Ger., 25.1.30).—Evaporation is effected by direct flame in a rotating cylindrical kiln which has an enlarged diam. at the burner end; the flow of liquid may be either con- or counter-current to the hot gases. The kiln is suitable for  $H_3PO_4$  and other scale-forming and/or corrosive liquids. B. M. VENABLES.

**Apparatus for concentrating solutions.** J. HAMILL, J. F. TADDIKEN, and G. W. CONNOR (U.S.P. 1,795,601, 10.3.31. Appl., 4.12.25).—In a calandria having vertical tubes, endeavour is made to give the steam a const. velocity in all parts. The entry of steam is at one side near which the tubes are widely spaced apart, the spaces diminishing towards the opposite side, where an outlet for permanent gases is provided, preferably behind a baffle forming a further contraction of the passage for the gases. The outlet for condensate is in the form of a gutter surrounding the axial downflow passage for liquid being concentrated. B. M. VENABLES.

**Distillate-chilling apparatus.** H. TORRANCE, ASSR. to CARBONDALE MACHINE CO. (U.S.P. 1,796,772, 17.3.31. Appl., 16.6.27).—An apparatus suitable for the separation of paraffin wax and similar purposes comprises a number of water-cooled straight conduits connected by headers: each of the former is provided with a rotating worm which scrapes the walls but permits free flow in an annular space between the shaft and blade of the worm. B. M. VENABLES.

**Gas scrubber.** A. G. MCKEE (U.S.P. 1,797,271, 24.3.31. Appl., 21.12.25).—The gas is passed upwards through a number of centrifugally produced sprays in succession; the uppermost sprayer is surrounded by a number of parallel plates upon which the spray impinges at a small angle without much shock, so that no mist is produced at that zone. B. M. VENABLES.

**Air cleaner.** C. A. WINSLOW and E. J. HALL (U.S.P. 1,798,510, 31.3.31. Appl., 25.9.24).—An air filter for the intakes of compressors, internal-combustion engines, etc. is described. C. JEPSON.

**Bubbling apparatus for treating gases with liquids.** H. KOHL (B.P. 356,985, 27.10.30. Austr., 20.11.29).—The gas is bubbled under the liquid from a single conduit situated within a bell, and from the bell it passes through a number of conduits which dip under the liquid outside the bell in an inclined direction so that the liquid is set in rotation. B. M. VENABLES.

**Collection and consumption of flue dust.** BABCOCK & WILCOX, LTD. FROM FULLER LEHIGH CO. (B.P. 356,773, 11.6.30).—The flue dust is separated from the gases in a dry dust catcher and delivered to the slag floor of the furnace. B. M. VENABLES.

**Viscosity-indicating instruments.** L. A. MAPEL (B.P. 356,721, 8.4.30).—A device for testing the oil from the lubricating system of a motor engine etc. is described. B. M. VENABLES.

**Apparatus for supplying reagents under pressure.** UNITED WATER SOFTENERS, LTD., and H. G. MARIGNY (B.P. 356,796, 10.6.30).—The apparatus is suitable for supplying a regulated flow of reagent into a main liquid flowing under pressure. It comprises a closed tank divided into two compartments by a horizontal partition, the upper parts of each compartment being in communication through an air-pipe. While main liquid is admitted to the upper compartment the air cushion is compressed and reagent forced out of the lower compartment. The difference in pressure between the inlet and outlet sides of the apparatus is produced by an orifice inserted in the main pipe, and this difference, besides causing the flow of reagent, also operates a diaphragm-and-needle valve to control the flow.

B. M. VENABLES.

**[Antifriction] bearing.** B. H. MCQUEER, ASSR. to NAT. CARBON CO., INC. (U.S.P. 1,797,833, 24.3.31. Appl., 12.8.29).—Bearings made from, *e.g.*, petroleum coke are steeped in a solution containing a B compound, preferably  $\text{Na}_2\text{B}_4\text{O}_7$ , and, if desired, a polyhydric alcohol, and are dried at 105–350°.

L. A. COLES.

**Producing [high] vacua [in radio valves etc.].** H. S. COOPER, ASSR. to KEMET LABORATORIES CO., INC. (U.S.P. 1,800,134, 7.4.31. Appl., 31.12.29).—An alloy of Mg with 5–30% of Ba (or Sr) is introduced as a pellet into the envelope (*e.g.*, valve) and is flashed after the latter has been partly evacuated and sealed.

L. A. COLES.

**[Mechanical] removal of deposits from boilers and prevention of formation of scale and apparatus therefor.** A. CZECZOTT (B.P. 356,869, 14.7.30. Poland, 13.7.29).

**Heat-insulating material.**—See IX. **Effecting reactions between gases. Precipitators for gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Petrography and the classification of coal.** C. A. SEYLER (Proc. S. Wales Inst. Eng., 1931, 47, 549–555).—The methods of classification of the constituents of bituminous coal due, respectively, to Stopes, Thiessen, and Wheeler are discussed. A. B. MANNING.

**Fuel technology and the classification of coal.** C. A. SEYLER (Proc. S. Wales Inst. Eng., 1931, 47,

557–592; cf. B., 1924, 544).—Coals are classified according to the positions of the points obtained when the H content is plotted against the C content (both calc. to the “pure coal” basis) on a suitable chart. The position of any such point determines many of the properties of the coal, *e.g.*, the calorific val., the volatile matter content, etc., such properties being conveniently represented on the chart by lines of equal calorific val. (“isocals” or “isocaustics”), equal volatile matter content (“isovols”), etc. The isocals are plotted by the aid of Dulong’s formula, which has also been used to deduce the linear relation, derived empirically by Rosin, between the vol. of combustion gases and the nett calorific val. This relation permits some other related quantities to be represented on the chart, *e.g.*, vol. of air required for combustion, flame temp., etc. It is believed that properties such as liability to oxidation, ignition temp., yields of products of carbonisation, etc. may also be represented. The softening points of bright coals are related to the ultimate composition and can be expressed as “isotectic” lines on the chart. The relation between the softening points of coals and their coking properties is discussed.

A. B. MANNING.

**Behaviour of solid fuels during oxidation.**  
**VII. Differentiation of coals as regards their tendencies to spontaneous ignition.** B. MOORE (Fuel, 1931, 10, 394–400; cf. B., 1931, 999).—The relation between the  $\text{O}_2$  temp. and the time of occurrence of (a) glowing of the coal, and (b) ignition of volatile matter, has been studied by the methods and apparatus described previously. The tendencies for ignition to occur were greatest with coals from seams associated with fires in the mine, and the highest ignition factors were obtained with these coals.

A. B. MANNING.

**Swelling pressure of coal and formation of “spongy” coke.** J. TAYLOR (Fuel, 1931, 10, 390–393).—A thin layer, *e.g.*, 2 mm. thick, of freshly powdered dry coal was heated in a cylindrical retort provided with a piston, which, when the coal reached the plastic stage, was loaded sufficiently to keep the vol. of the coal const. The coal was held between layers of asbestos paper and the base of the retort was perforated. The rate of heating was 10°/3 min. The max. load gave a measure of the actual swelling pressure in the plastic layer, whereas in other methods, using cylinders of coal 2–3 cm. high, this swelling pressure may be partly neutralised by the secondary contraction of the coke formed. With coals of high agglutinating val. the magnitude of the swelling pressure was a guide to the tendency of the coal to form spongy coke in the oven, and the blending of coals to avoid spongy coke formation could be controlled by this method of examination.

A. B. MANNING.

**Determination of volatile matter in coal. Temperature of the preliminary heating, and an electric furnace therefor.** T. SŌMIYA and Y. NAKAMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 282–283 B, 283 B).—Variations in the loss of wt. of a number of coal samples on heating for 7 min. at 950°, with or without a pre-heating for 6 min. at 500°, 600°, 700°, or 800°, are tabulated. A temp. of 700° is recommended as giving the



most concordant results in the final determination. Dimensions of a suitable furnace are given. C. IRWIN.

**Production and use of "Dryco" [smokeless fuel].** R. E. GIBSON (Gas J., 1931, 195, 739–741).—A method of preparing high-temp. coke, suitable for use as a domestic fuel, is described. Coke of the desired quality is obtained by blending suitable coals, carbonising them in retorts for 9–10 hr. at about 1000°, and cooling the coke in a Sulzer plant. The coke is then crushed to 2-in. size and screened to remove fines below  $\frac{1}{2}$  in. C. B. MARSON.

**Table for the calculation of the calorific value of solid fuels.** W. ZWIEG (Chem.-Ztg., 1931, 55, 723).—For a  $\text{H}_2\text{O}$  equiv. of the calorimeter +  $\text{H}_2\text{O}$  of 3450 a table has been constructed giving the total cal. evolved by 1 g. of coal for a rise in temp. of 1.740–2.229°, i.e., the calorific val. of the coal. A. R. POWELL.

**Properties of cokes in relation to their reactivity.** T. J. DRAKELEY and E. T. WILKINS (J.S.C.I., 1931, 50, 331–342 r).—Homogeneous cokes, prepared from crushed and briquetted coal by carbonisation at various temps. between 450° and 1100°, were recarbonised rapidly at 1000° in the laboratory. A microscopical examination of the coke sections showed that the structure became more open as the temp. of coking increased and that certain changes in the appearance of the low-temp. cokes took place during recarbonisation. These observations are illustrated by means of photomicrographs. The "real" and "apparent" density of the cokes showed that the porosity of the recarbonised samples was almost const., as were the C and H contents. The approx. ignition temp. was found to exhibit a tendency to vary with the reactivity of the coke samples towards  $\text{CO}_2$ . The adsorptive capacity of the cokes for methylene-blue, which is probably dependent on the extent of the readily accessible surfaces, was found to bear no relation to the reactivity, although many experiments were conducted in an endeavour to discover a disturbing factor. The adsorptive capacity for  $\text{CO}_2$  at 30 in. pressure and 15° was measured before and after recarbonisation, and the effect of "activation" by  $\text{CO}_2$  at 1000° was also examined. There appears to be some relationship between this adsorptive capacity and the reactivity, but all the variations in reactivity cannot be thus explained. The ignition temp. and reactivity of a coke are considered to be largely influenced by the condition of the micropores.

**Reactivity of coke.** T. J. DRAKELEY (J.S.C.I., 1931, 50, 319–330 r).—The passage of gas through crushed coke in a tube is principally governed by the law of capillary flow, and only about 2% of the gas may be supposed to pass through the actual pores of the coke. A large variation in the porosity of cokes prepared at the same temp. had only a slight influence on their capacity to reduce  $\text{CO}_2$ . The law of mass action is shown to be applicable to the reaction between the cokes and  $\text{CO}_2$  at 900°, 1000°, and 1100°, and vals. are respectively calc. for the direct and reverse reaction velocity coeffs. The samples of coke were prepared from the same coal at temp. varying from 450° to 1100°, and the direct reaction velocity coeff. was found at each of the three

reaction temp. to diminish slowly as the temp. of carbonisation was increased from 450°. At 700° the coeff. diminishes rapidly and again more slowly between 900° and 1100°. The velocity coeff. of the reverse reaction, in which  $\text{CO}$  is decomposed to give C and  $\text{CO}_2$ , shows an abrupt rise at about 700°, after which increased temp. of carbonisation has little effect. A rise in the temp. of the reaction increases the direct reaction velocity coeff., but diminishes the coeff. for the reverse reaction with all cokes. On the assumption that the reaction proceeds as it commenced, the % of  $\text{CO}$  in the hypothetical equilibrium mixtures have been calc. The vals. so obtained are exceptionally low. Continued heating of the sample of coke gives data which indicate that the hypothetical equilibrium mixture will then contain more  $\text{CO}$  than with the original sample of coke. This effect is due to a considerable decrease in the reverse reaction velocity coeff., which more than counterbalances the decrease in the direct reaction coeff. Hence the reactivity of a sample of coke cannot be expressed solely in terms of the velocity coeff. for the direct reaction; the reverse reaction velocity coeff., which is particularly sensitive to heat effects, must also be stated. The formation of  $\text{CO}$  and the rates of the reaction are explained on the assumption that coke contains C which becomes more cryst. as the temp. of carbonisation increases. Thus the production of  $\text{CO}$  and the rate of the direct reaction decrease as the temp. of carbonisation increases. On the other hand, the more cryst. is the C, the more rapid becomes the reverse reaction, probably due to catalytic influences. At higher reaction temp. the formation of  $\text{CO}$  and the rate of the direct reaction increase for all samples of coke. On the other hand, the rate of the reverse reaction diminishes. This may be due to diminished absorption, and therefore decreases decomp. of the  $\text{CO}$  by a relatively smaller amount of cryst. C consequent on the promotion of cracks in the coke surface.

**Desiccation of lignite by the Fleissner process.** H. KLEIN (Internat. Bergwirts. u. Bergtechn., 1930, 23, 377; Fuel, 1931, 10, 385–389).—The process, which is particularly suitable for drying woody lignites, is carried out in large tanks and comprises four steps: (1) preheating, (2) heating with live saturated steam under pressures of 10–20 atm., (3) expansion of the steam to atm. pressure, and (4) final drying of the material by circulation of air. On heating under pressure with saturated steam,  $\text{H}_2\text{O}$  in the liquid form flows out of the lignite; this, together with  $\text{H}_2\text{O}$  from condensation of part of the steam, is continuously drained away. The steaming period occupies 75–80 min. The steam leaving the tank during expansion is utilised in preheating the lignite in another tank. The total heat consumption is about 460 kg.-cal. per kg. of  $\text{H}_2\text{O}$  removed. The product is a solid fuel of high calorific val. and good storing properties. A plant at Köflach, Austria, having 16 tanks each of 13,500 kg. capacity, and producing 1100 tons of desiccated fuel per day, is described. The raw coal is a typical lignite of 35–38%  $\text{H}_2\text{O}$  content, which is reduced to 13–17%, the calorific val. being raised from 3600 to 5000 kg.-cal./kg. A. B. MANNING.

**Modern methods of investigation in gas technology.** H. BROCHE (Brennstoff-Chem., 1931, 12, 312—315).—Recent developments in the methods of determining tar and dust, S, O<sub>2</sub>, C<sub>10</sub>H<sub>8</sub>, etc. in coke-oven and other gases, of evaluating gas-purifying material, and of controlling gas purification are briefly discussed.

A. B. MANNING.

**Effect of temperature and moisture content of [iron] oxides [for use in purification of coal gas].** D. V. REID (Gas J., 1931, 195, 742—745).—A method for determining the purifying efficiency of Fe oxides under varying conditions is described, the apparatus and oxides used being those described by Dunkley and Leitch (B., 1925, 487). It is shown that each oxide has an optimum range of H<sub>2</sub>O content, and that increasing or decreasing the H<sub>2</sub>O content beyond this range lowers its activity. The activity of some oxides is confined to a narrow range of H<sub>2</sub>O content, but with others (e.g., the precipitated oxide) it is const. over a wide range; the activity increases with rise in temp., up to a max., and then decreases. Curves relating rate of gas purification and (a) % H<sub>2</sub>O, (b) rise in temp., for the various oxides are given. C. B. MARSON.

**Determination, transformation, and removal of organic sulphur in gases.** O. ROELEN (Brennstoff-Chem., 1931, 12, 305—312).—The S in gases of low S content has been determined by the catalytic combustion of the gas over a Pt spiral, followed by absorption of the SO<sub>2</sub> produced. The apparatus had a throughput of 100 litres/hr. The results were low, and a more suitable method, to be described in a later communication, has been worked out. For the catalytic production of benzene from water-gas the S content of the latter must be reduced to 0.1—0.2 g./cu. m. Initial attempts to purify water-gas to this degree by the catalytic conversion of the org. S into H<sub>2</sub>S, and absorption of the latter, failed with intermediate-scale apparatus, using as catalysts CuO—PbCrO<sub>4</sub>, finely-divided Ag deposited on MgO, or Fe wool; desulphurisation of the gas was satisfactory at first, but later the efficiency decreased, owing mainly to conditions which brought about a re-formation of org. S compounds, e.g., COS and CS<sub>2</sub>, from the H<sub>2</sub>S and CO in the gas. Thus with Fe as catalyst this reaction occurred in the cooler parts of the purifier, being accelerated by the catalytic action of a deposit containing Fe, C, and S, which was formed on the walls of the apparatus. In producing an H<sub>2</sub>:CO mixture from coke-oven gas (cf. Fischer, B., 1931, 231) the greater part of the org. S was simultaneously converted into H<sub>2</sub>S. Such a gas mixture, freed from H<sub>2</sub>S by passage over luxmasse, was passed through a heated Fe tube, then cooled by direct contact with a stream of H<sub>2</sub>O, and finally washed with an alkaline solution of K<sub>4</sub>Fe(CN)<sub>6</sub>. The rapid cooling prevented the re-formation of org. S compounds and the final washing removed the residual H<sub>2</sub>S. This arrangement has given a gas of the required purity over long working periods, requiring only an occasional cleaning out of the Fe tube. Luxmasse is unsuitable for the final removal of H<sub>2</sub>S, for it also can bring about the formation of COS and CS<sub>2</sub> from H<sub>2</sub>S and CO.

A. B. MANNING.

**Removal of nitric oxide from coke-oven gas by its catalytic reduction to ammonia.** H. TROPSCH

and R. KASSLER (Brennstoff-Chem., 1931, 12, 345—348).—The gas is passed at 200—250° over a MoS<sub>2</sub> catalyst formed by impregnating "Stuttgart mass" with NH<sub>4</sub> thiomolybdate and reducing the latter in H<sub>2</sub> at 210°. The rapidity with which the NO was reduced was greatly increased by carrying out the process under pressure, e.g., 13—16 atm. A similar W catalyst was much less effective. The NO was converted almost completely into NH<sub>3</sub>, little or no N<sub>2</sub> being formed. Small quantities of NO in coke-oven gas were determined by treatment of the gas with excess of O<sub>2</sub>, absorption of the NO<sub>2</sub> in NaOH solution, treatment of the neutralised solution with Ilosvay's reagent, and comparison of the colour produced with standards corresponding with known amounts of NO.

A. B. MANNING.

**Hydrogenation of nitrogen compounds of primary tar.** G. ROBERTI (Atti R. Accad. Lincei, 1931, [vi], 13, 527—531).—When hydrogenated at 350°/100 atm. in presence of CoS, pyridine yields pentane, amylene, hexane, hexene, heptane, and an unsaturated hydrocarbon,  $d^{12}$  0.7503,  $n_D^{15.3}$  1.4223, formed apparently by hydrogenation of a condensation product of 2 mols. of pyridine. Similarly, quinoline gives propylcyclohexane, propylcyclohexene, etc. The N is liberated as NH<sub>3</sub> in each case (cf. B., 1931, 833).

T. H. POPE.

**Formation of oil from dilute acetylene.** D. BINNIE (J.S.C.I., 1931, 50, 297—299 r).—Ni carbonyl or Ni used in conjunction with CO is a suitable catalyst for condensing dil. C<sub>2</sub>H<sub>2</sub> (4%) in the presence of H<sub>2</sub> into a complex olefinic oil of  $d$  0.69. Yields of 60% (wt. of oil on wt. of C<sub>2</sub>H<sub>2</sub>) were obtained. Ni alone hydrogenates the C<sub>2</sub>H<sub>2</sub> to C<sub>2</sub>H<sub>6</sub>.

**Determination of bitumen in natural asphalt and asphaltic materials by centrifuging.** M. ILMENEV (Nef. Choz., 1930, 19, 614—615).—The sample (1.5—2 g.) is refluxed for 10—15 min. with C<sub>6</sub>H<sub>6</sub> (100 g.); after cooling, 70 c.c. are centrifuged for 5 min. at 2500 r.p.m., 50 c.c. of this being then evaporated, and the residue of bitumen dried and weighed. H<sub>2</sub>SO<sub>4</sub> in "kontakt" is determined iodometrically by the use of BaCrO<sub>4</sub>.

CHEMICAL ABSTRACTS.

**Bitumen from Ural crude oil.** A. N. PARSHIN (Nef. Choz., 1930, 19, 560—564).—A report and discussion.

CHEMICAL ABSTRACTS.

**New oil fields in the Benoisk district.** V. SELSKI (Groznski Neft., 1930, 1, No. 1, 15—21).—Oil from a well 150 km. S.E. of Grozni contained 30% b.p. < 200°,  $d$  0.774, and 40% b.p. 200—315°,  $d$  0.814. The bottoms were almost pure gas oil,  $d$  0.866. The crude oil contains up to 10% of paraffin.

CHEMICAL ABSTRACTS.

**Crude oil from the Benoisk district.** A. Y. GANCHIKOVA (Groznski Neft., 1930—1931, 1, No. 2—3, Suppl. 1, 1—5).—Characteristics of the crude oil and its fractions are recorded.

CHEMICAL ABSTRACTS.

**Refining Ural crude oil.** D. M. MARKO (Nef. Choz., 1930, 19, 560—561).—Characteristics of the crude oil and its fractions are recorded.

CHEMICAL ABSTRACTS.

**General constituents of Japanese petroleum. I. The fraction of petroleum from Nisayama distilling**

under 200° at ordinary pressures. R. KOBAYASHI (J. Soc. Chem. Ind., Japan, 1931, 34, 289—294 B).—The characteristics and constitution of each fraction having a boiling range of 10° from 50° to 200° are described.

E. H. SHARPLES.

**Gasoline fractions of representative Japanese crude petroleum.** III. M. MIZUTA (J. Soc. Chem. Ind., Japan, 1931, 34, 288 B).—Halogen absorption methods cannot be adapted to the determination of unsaturated hydrocarbons in gasoline; 80%  $\text{H}_2\text{SO}_4$  is the most suitable reagent and completely removes these hydrocarbons without affecting other substances. Japanese gasolines contain < 1% of unsaturated hydrocarbons.

E. H. SHARPLES.

**Total heat and sp. heat of a series of fractions of petroleum oil, and their relation to other properties (Persian oil fractions).** VI. Preparation of samples and determination of their mean mol. wt. and other properties. VII. Temperature variation between 0° and 100° of the sp. heat. II. R. LANG and R. JESSEL. VIII. Total heat of the liquids. H. R. LANG (J. Inst. Petroleum Tech., 1931, 17, 572—576, 577—581, 581—584).—VI. Mean mol. wts. in  $\text{PhNO}_2$ , b.p.,  $n_D^{20}$ , coeff. of expansion, and sp. gr. of crude oil have been determined.

VII. Sp. heats have been measured from 0° to 100° by the continuous-flow method.

VIII. The total heats are calc. C. W. GIBBY.

**Doctor test and silver nitrate test for the detection of sulphur compounds in gasoline.** M. MIZUTA (J. Soc. Chem. Ind., Japan, 1931, 34, 287—288 B).—Variations between the doctor test and the  $\text{AgNO}_3$  test with S compounds are described. The  $\text{AgNO}_3$  test gave the highest no. of "positive" reactions and is recommended for straight-run gasolines, whilst the doctor test gives better results for the detection of mercaptans in cracked gasolines, mercaptans giving a positive reaction on the addition of flowers of S. E. H. SHARPLES.

**Mol. wts. and sp. gr. of gasoline fractions obtained in the Vickers unit.** L. SELSKI and A. SOMOV (Groznski Neft., 1930—1931, 1, No. 2—3, Suppl. 1, 7—8).—Vals. are tabulated.

CHEMICAL ABSTRACTS.

**Sediments in the condensers of the Foster-Wheeler unit.** B. BONDARENKO (Groznski Neft., 1930—1931, 1, No. 2—3, Suppl. 1, 5—7).—The sediment is removed by injection of HCl. The temp. of the cooling  $\text{H}_2\text{O}$  should not exceed 50°.

CHEMICAL ABSTRACTS.

**Uncondensed refinery gases and their utilisation.** A. DOLADUGIN and S. ISAEVA (Groznski Neft., 1930, 1, No. 1, 54—56).—The composition of the gas is recorded; the quantity per ton is calc. for various units.

CHEMICAL ABSTRACTS.

**Removing sulphur from Chusovskie Gorodki (Ural) [oil] distillates.** I. Y. POSTOVSKI and V. G. PLUSNIN (Neft. Choz., 1930, 19, 561—564).—The fractions are passed with steam over Fe ore at 350—450°. The S content of the first 100 c.c. is thus reduced from 1.25 to 0.25%, 5% of the distillate being lost. The S content was reduced to 0.14% by treatment with 2% of conc.  $\text{H}_2\text{SO}_4$ . The activity of the catalyst was lowered by formation of C.

CHEMICAL ABSTRACTS.

**Breaking crude oil emulsions.** F. SCHNEIDER and A. V. HEIFETZ (Azerbaid. Neft. Choz., 1930, No. 12, 138).—Crude oil emulsions (up to 50%  $\text{H}_2\text{O}$ ) formed from Bibi-Eibat "emulsifying" crude oil are broken by treatment with acid sludge obtained in the treatment of the vaseline oil distillate with fuming  $\text{H}_2\text{SO}_4$ .

CHEMICAL ABSTRACTS.

**Knocking properties of motor fuels and knock prevention.** E. ENDO (J. Fuel Soc. Japan, 1931, 10, 57—58).—Using an Armstrong fuel testing plant it was found that (1) the variation of engine speed between 550—850 r.p.m. was not effective on the knocking rate, (2) the highest useful compression ratio (H.U.C.R.) decreased by about 0.5 when the ignition timing was advanced from  $-12^\circ$  to  $-31^\circ$ , (3) the knocking tendency, which decreased with throttling, increased with temp. rise of the intake air and cooling water, (4) the fuel-air ratio at the max. knocking intensity was nearly the same as that at the max. power output. The most important influence of knocking on engine performance is power drop. Nineteen varieties of gasoline were tested and their  $\text{C}_6\text{H}_6$  vals. were found to depend mainly on their volatility. The knocking rate of common antiknocks was investigated and three classes were recognised. The first contains those only powerful in a rich concentration in gasoline, e.g.,  $\text{C}_6\text{H}_6$ ; the second, those having antiknock power proportional to their concentration, e.g., PhMe; and the third is effective in a low concentration, but does not increase its antiknock power proportionally to its concentration, e.g.,  $\text{PbEt}_4$ . Initial results, using a N.A.C.A. universal test engine in comparison with the Armstrong engine, indicate that the same knocking val. will be obtained with different engines under the same measuring conditions.

H. S. GARLICK.

**Motor-fuel testing.** O. ENOCH (Brennstoff-Chem., 1931, 12, 348—352).—Methods of evaluating motor fuels, and in particular the recent developments in methods of determining the antiknock value, are discussed.

A. B. MANNING.

**Fuel testing in slow- and high-speed Diesel engines.** L. J. LE MESURIER and R. STANSFIELD (J. Inst. Petroleum Tech., 1931, 17, 387—418).—Standard engines have been used to determine fuel consumption, fuel-system leakage, the combustion process from the point of injection through the delay period to the beginning of expansion, combustion stock, ease of starting, and the effects of dopes, nozzle alterations, and changes in speed in relation to combustion and shock. The characteristics of 14 different fuels are tabulated and the behaviour of the fuels was observed on engines of the Robey, Petter, Benz, and Junkers type under standardised conditions of running. Very little difference was found in the consumptions of the engines, although fuels of paraffinic, aromatic, and naphthenic types were used. Viscosity was found to affect only the fuel-system leakage and not the consumption. The process of combustion may be divided into 3 stages: delay period between injection and commencement of pressure rise, a period of rapid uncontrollable combustion, and a period during which the remainder of the fuel burns as injected at the nozzle. The effect of the delay period

is shown to be least with paraffinic fuels, which give less combustion shock. No relationship was found between ease of starting and self-ignition temp. The addition of materials which promote knocking in petrol engines was found to be of some value in promoting smoother running in the Diesel engines, and in shortening the delay period. Finer sprays were found to give more rapid pressure rise in the earlier stages of combustion. Slower speeds were found to increase the tendency to combustion shock. In general, the Diesel fuels from crudes which give the highest anti-knock petrols are likely to be worst for use in Diesel engines. T. A. SMITH.

**Determination of mean mol. wts. of lubricating oils by improved cryoscopic methods.** N. G. GULLICK (J. Inst. Petroleum Tech., 1931, 17, 541—571).—Mol. wts. of such oils can be determined cryoscopically to  $\pm 1-1.5\%$ , using  $C_6H_6$  or  $PhNO_2$  as solvent. The presence of  $Na_2SO_4$  as a drying agent does not impair the accuracy of the method. To obtain consistent results it is essential to maintain a const. temp. difference between the temp. of the cooling bath and the f.p. Differentiation may be made between different types of oil. C. W. GIBBY.

**Preparation of lubricating oil by once-through distillation of fuel oil.** Y. E. EMMUL and R. M. SHATIROV (Nef. Choz., 1930, 19, 582—585).—The characteristics of the source and products (obtained with little steam and much fuel, or *vice versa*) are recorded. CHEMICAL ABSTRACTS.

**Dewaxing paraffinic lubricating oils.** ARISTOV (Groznski Neft., 1930, 1, No. 1, 57).—Various methods are discussed. Dilution of the cylinder oil residue with ligroin or other solvent and addition of a fine porous solid is the method preferred. CHEMICAL ABSTRACTS.

**Prevention of explosion danger in oil-tankers. Optical analysis of very weak mixtures of petroleum vapour [with air].** Z. TUZI and O. KADITA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 16, 147—158).—Doi's modification of Jamin's refractometer has been used with satisfactory results for the determination of small quantities of petroleum vapour in air. C. W. GIBBY.

**Preparing petroleum soap in Grozni refineries.** E. MUSHKIN (Groznski Neft., 1930—1931, 1, No. 2—3, 44—46).—The following percentages of naphthenic acids are recorded: kerosene from paraffin-base crude oil 0.012, from semi-paraffin crude oil 0.090, from asphalt-base crude oil 0.35, from lubricating-oil crude oil 1.01, from Maikop crude oil 0.66; gas oil from asphalt-base crude oil 0.72, from heavy crude oil 0.60; heavy gas oil 1.94 and 1.95; gas oil from Maikop crude oil 0.72; spindle-oil distillate 1.60; spindle-oil distillate from asphalt-base crude oil 0.90; machine oil distillate 0.50. The naphthenic acids are first removed with NaOH, because they are partly sulphonated by  $H_2SO_4$ . These acids are less suitable for soap-making and are difficult to recover. CHEMICAL ABSTRACTS.

**Naphthenic acid soaps.** R. A. VIRABYANTZ and O. A. ARTEMIEV (Groznski Neft., 1930—1931, 1, No. 2—3, Suppl. 1, 9—13).—The naphthenic acids are determined by dissolving the product in a mixture of

$EtOH$  and  $C_6H_6$  and titrating with alcoholic alkali in presence of alkali-blue. The naphthenic acid content of various oil fractions is recorded.

## CHEMICAL ABSTRACTS.

**Asphalt paving materials.**—See IX. **Determination of CO.**—See XIII. **Oil sprays.**—See XVI.

## PATENTS.

**Preventing explosions in [coal] mines.** E. WITTE (B.P. 355,412, 14.6.30. Ger., 14.6.29).—The coal dust in the mine is mixed with such a proportion of mineral dust of const. colour shade that the grey mixture is lighter in shade than a crit. mixture, determined by experiment, which fixes the explosive limit for the mine. A. B. MANNING.

**Apparatus for washing coal or the like.** G. E. DEAN (B.P. 355,269, 20.5.30).—The apparatus comprises a water tank containing two conveyors arranged side by side and having a lateral extension wherein is a breaking mechanism. The crushed material falls on to an adjustable chute which delivers it into the tank. One or more nozzles, disposed immediately below the chute, produce a deflecting current of water, so regulated as to carry the material of lower sp. gr., *e.g.*, coal, on to the further conveyor, whilst that of higher sp. gr., *e.g.*, slate and other impurities, is deposited on to the nearer conveyor. The conveyors discharge the materials, after affording opportunity for drainage, from the tank on to separate chutes. A. B. MANNING.

**Dissolution of coal.** KOPPERS Co., Assees. of H. J. ROSE and W. H. HILL (B.P. 356,239, 31.5.30. U.S., 1.6.29).—The process described in B.P. 268,372 (B., 1928, 882) is carried out by mixing the coal and solvent in a porous container at a temp. at which the coal is dissolved without decomp., and under such conditions that the insol. material forms a filter layer on the inside of the container which facilitates separation of the coal solution from the foreign solid matter. Means may be provided for recirculating the heated oil through the container or for vaporising the oil and condensing the vapours directly above the container. A. B. MANNING.

**Manufacture of briquettes and like agglomerated bodies or lampblack from coal dust.** H. HARDY (B.P. 356,236, 7.5.30. Belg., 22.5.29).—Caking coals, alone or admixed with non-caking coals, are heated in the granular state and with agitation, *e.g.*, in a horizontal tubular retort provided with a spiral conveyor, until the particles swell and assume a spheroidal shape, the heat treatment being arrested before the particles burst. The material is then cooled, moistened with  $H_2O$ , reheated to about  $100^\circ$ , and briquetted without the addition of tar or other binder. The cooling may be carried out in such a manner that, on moistening, the particles burst with the production of lampblack, which may be separated. The briquettes may subsequently be carbonised. A. B. MANNING.

**Working of coal-distillation ovens.** C. STILL (B.P. 355,956, 2.9.30).—Ducts or hollow spaces are formed in the compressed coal charge by means of suitable forming members, *e.g.*, tubes, which are then withdrawn from the coal. The volatile products of

distillation are withdrawn from the retort, under a relatively high vac., *e.g.*, 40 mm. water gauge or higher, through tubes connected with the ducts in the coal. The ducts may be strengthened by being filled with lumps of coke. A. B. MANNING.

**Coke ovens and the like.** C. STILL (B.P. 356,079, 9.1.31. Ger., 14.1.30. Cf. B.P. 355,956, preceding abstract).—Ducts may be formed in the charge by rods which are pushed in from the oven roof; these are then withdrawn and tubes of slightly larger diam., tapered at the ends, are jammed into the openings of the ducts to form therewith as gastight a connexion as possible. The tubes communicate with a common gas-collecting pipe, through which the volatile products are withdrawn, preferably under vac. (at least 50 mm. water gauge). A. B. MANNING.

**Furnaces for burning waste materials and the like.** STETTINER CIAMOTTE-FABR. A.-G., VORM. DIDIER (B.P. 356,278, 6.6.30. Ger., 10.6.29).—The waste material is fed on to an inclined grid on which it is divided into thin layers for drying and sifting purposes, and after being dried and freed from its finer constituents is conveyed to the combustion space and burned, all the steps taking place within a single furnace. A. B. MANNING.

**Gas-producing apparatus.** J. U. McDONALD (U.S.P. 1,798,617, 31.3.31. Appl., 13.6.25).—The producer consists of a double shell, the annular space acting as a steam boiler. Solid fuel (with or without the addition of oil), steam, hot (preheated by the producer gas) and cold air enter the producer at the top. The gas is withdrawn at the bottom through a pipe inside a conical ash discharger. The discharger is attached to a rotary vessel which holds water. D. K. MOORE.

**Apparatus for manufacturing carburetted water-gas.** F. B. DEHN. From GAS MACHINERY CO. (B.P. 355,490, 8.8.30).—The generator is connected with the carburettor by a single duct which opens centrally into and co-axially with the top of the latter. The oil-supplying duct and the secondary air duct are also arranged co-axially with the opening of the gas duct. By these means uniform heating of the chequerbrick and lining of the carburettor is obtained. A. B. MANNING.

**Manufacture of gas from liquid fuels.** O. O. THWING, ASSR. to GEN. OIL GAS CORP. (U.S.P. 1,796,733, 17.3.31. Appl., 1.4.25).—The composition and calorific val. of gas manufactured by the partial combustion of hydrocarbon oil are kept const. by keeping the temp. of the combustion zone of the retort const. This is done by thermostatic control of the oil supply. D. K. MOORE.

**Automatically controlling the calorific value of gas.** R. ASHWORTH and W. R. D. TILDESLEY (B.P. 356,425, 12.8.30).—The lever admitting a diluent to the gas is operated by a governor, the position of which is maintained normally by an exhaustor, but is controlled by an air-supply valve, operated in turn by the arm of a recording gas calorimeter. A. B. MANNING.

**[Continuous] manufacture of oil gas.** E. A. DIETERLE (U.S.P. 1,798,372, 31.3.31. Appl., 22.4.24).—Atomised oil, with or without the addition of steam, is passed over the outside of vertically spaced banks of

tubes through which hot gases of combustion are being passed. D. K. MOORE.

**Recovery of pressure gas from oil wells.** L. N. YATES, ASSR. to SINCLAIR OIL & GAS CO. (U.S.P. 1,798,774, 31.3.31. Appl., 19.6.30).—In the automatic intermittent pumping of oil from wells by means of gas pressure, the gas released when the well chamber has been emptied is divided into two parts, one released at high pressure and one at low pressure. The former is free from, but the latter contains, oil vapours. D. K. MOORE.

**[Synthetic] production of acetylene.** D. A. BRADING, ASSR. to M. BRADING (U.S.P. 1,797,400, 24.3.31. Appl., 26.3.25).— $C_2H_2$  is produced by passing preheated  $H_2$  and finely-divided C through an electric arc. The  $H_2$  employed is preferably that derived from the electrolysis of  $H_2O$ . D. K. MOORE.

**Carrying out catalytic oxidations.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 355,308, 14.4.30).—Compounds of C, *e.g.*, CO, or the exhaust gases from Diesel engines etc., are oxidised by passing them, admixed with air, at the ordinary or slightly raised temp. over catalysts consisting of the manganites of the heavy metals, the earth metals, or the rare-earth metals, *e.g.*, of Cu, Fe, Ni, Zn, etc. The manganites may be mixed with one another or with other substances, *e.g.*, Co oxide,  $Fe(OH)_3$ , or metals such as Fe, Zn, or Al, in order to produce a catalyst of any desired activity. A. B. MANNING.

**Separation of carbon monoxide from mixed gases containing hydrogen.** OESTERR.-AMERIKANISCHE MAGNESIT A.-G. (B.P. 355,861, 16.6.30. Austr., 18.6.29).—CO is eliminated from water-gas etc. by passing the gas, admixed with steam, over a mixture of C and  $MgO$  or  $MgCO_3$  at a temp. above the decomp. temp. of  $MgCO_3$ , but not above  $500^\circ$ . The reaction  $CO + H_2O \rightarrow CO_2 + H_2$  may be accelerated by heating the first portion of the contact material to  $750^\circ$ , provided that the gases afterwards pass over the second portion at not above  $500^\circ$ . A. B. MANNING.

**Collection of sulphur from gases.** A. M. CLARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 355,832, 31.5. and 18.8.30).—The mist or fume of finely-divided S obtained by cooling a gas containing S vapour is maintained at a moderately high temp., *e.g.*, between the initial condensation temp. and  $100^\circ$ , for sufficiently long to allow the S particles to attain a coarser state of division; the S is then separated from the gas by filtration or settling. It is desirable to adjust the dust content of the initial gas, *e.g.*, by filtration followed by the addition of a regulated quantity of the unfiltered gas, in order to provide the optimum number of condensation nuclei for the process. A. B. MANNING.

**Obtaining a condensate from gases.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 355,413, 16.6.30. Holl., 29.6.29).—In separating a condensate from a gas, either by compression or adsorption, the efficiency of the process is increased by first adding to the gas a substance which has a b.p. below that of the lowest-boiling of the components of the desired condensate, and is also to a considerable extent condensed therewith; it is subsequently removed from the condensate and again utilised in a further separation. Thus in the separation of

benzine from casing-head gas the added substance is preferably  $C_3H_8$ . A. B. MANNING.

**Treatment of gas-purification products.** F. W. SPERR, JUN., ASSR. to KOPPERS Co. (U.S.P. 1,798,912, 31.3.31. Appl., 7.10.26).—The froth, consisting of S and Fe oxide, obtained by the aeration of the spent alkaline liquid used for the absorption of  $H_2S$  in gas manufacture, is subjected to violent aeration in a flotation machine. The tailings, containing Fe oxide, is returned to the first aeration vessel, whilst S in a high state of purity is removed from the froth by filtration. A recovery of 90% of the S and 80% of the Fe or other compound is attainable. D. K. MOORE.

**Boilers for heating tar and tar compounds.** G. S. CLARK (B.P. 356,235, 7.5.30).—The boilers or heating tanks are provided with electrical heaters detachably mounted within tubes fixed horizontally in the tanks. The resistance of the elements is such that they do not get red hot at the voltage for which they are designed to work. A. B. MANNING.

**Treatment of asphalts.** H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 355,430, 30.6.30).—The stickiness or tackiness of asphalts is decreased by treating them in the molten condition with 3–10% of a resinate, sulphonate, naphthenate, or linoleate etc. of Mn, Co, Pb, or other heavy metal. A. B. MANNING.

**Extraction of phenol, phenol homologues, and other hydroxy-aromatic compounds from tar oils and mineral oils.** J. G. PEAKE (B.P. 355,445, 9.7.30).—Aq. alkaline solutions containing the impure phenoxides etc. are distilled under reduced pressure to remove volatile impurities, and then acidified to liberate the phenols etc., which are subsequently fractionated at appropriate temp. and pressures. A. B. MANNING.

(A) Production of tar compositions for road construction and otherwise for use as coatings.

(B) Production of bituminous compositions. AMBER SIZE & CHEM. Co., LTD. (B.P. 355,937 and Addn. B.P. 356,003, [A] 18.8.30, [B] 13.10.30. Ger., [A] 16.8.29, [B] 11.10.29).—(A) Tar compositions of increased "wetting capacity," liquid at room temp. and usable in any weather, are prepared by adding to the usual prepared tars 3–20% of materials, e.g., Dippel's oil, bone oil, or shale oil, containing aromatic hydroxy-compounds and N-bases. (B) Bituminous substances other than tar, e.g., petroleum pitches, may be similarly treated. A. B. MANNING.

**Treatment [cracking] of [petroleum] oils.** C. B. BUERGER, ASSR. to GULF REFINING Co. (U.S.P. 1,796,138, 10.3.31. Appl., 23.2.23).—Oil is maintained under cracking temp. and pressure in a vertical column and a portion is continuously removed, forcibly circulated through heating tubes, and returned to the main bulk of oil at a point below that of withdrawal and under conditions allowing C, tar, and heavy residue oil to settle and be withdrawn. The vapours before final condensation are scrubbed with replenishing oil, which is supplied to the heating coil in admixture with that portion of the oil being circulated from the main supply. H. S. GARLICK.

**Treatment [cracking] of hydrocarbon oils.** J. W. COAST, JUN., and G. T. GRANGER (U.S.P. 1,786,053, 23.12.30. Appl., 11.12.26).—Crude oil is pumped into the top of a distilling column (A), the vapours from which pass into a fractionating column (B) near the bottom, whilst the vapours from B are condensed to yield motor spirit. The liquid from the bottom bubbler of A is pumped through a skimming still in which it is heated, and the liquid and vapour are passed into an expansion vessel (C) from which the liquid flows into a storage tank to be used as fuel. The vapours from C pass into A below the bubbler plates, the vapours rising through the bubblers, whilst the liquid, mixed with the condensate from B, which also flows into A below the bubbler plates, is pumped through a still in which it is cracked under pressure. The vapours and liquid therefrom are forced into a second expansion vessel from which the vapours enter A below the bubbler plates, whilst the liquid passes into C. The liquid in the bottom of A is recirculated through the cracking still. The process considerably reduces the accumulation of coke in the stills and pipes. D. K. MOORE.

**Treatment [cracking] of petroleum oils.** W. M. CROSS, ASSR. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,785,073, 16.12.30. Appl., 28.8.25).—Oil heated to 315–370° is pumped into a vessel under 600–2000 lb./sq. in. pressure. Air is forced into this vessel through a perforated pipe and the combustion of a little of the oil raises the temp. to 370–540° and cracking occurs. After a period of digestion the oil is discharged into a still, and on releasing the pressure about 90% passes over as vapour and is condensed. The introduction of the air enables the oil to leave the heating coil at a lower temp., thereby reducing coke formation, minimises the production of unsaturated hydrocarbons, and promotes the formation of anti-detonating compounds, e.g., hydroxy- and aromatic compounds. D. K. MOORE.

**Apparatus for distilling [crude] oil.** R. F. GILDEHAUS, JUN. (U.S.P. 1,788,947, 13.1.31. Appl., 10.8.25).—Vac. stills, heated by radiant heat instead of by contact with furnace gases, are arranged in series so that a gravity flow through the series can be obtained. Good heat transference is obtained by maintaining a circulation of oil in each still. Equalising lines are provided so that the same vac. is maintained in the stills and charging tanks. T. A. SMITH.

**Apparatus for distilling [hydrocarbon] oils.** J. R. SCHONBERG, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,791,937, 10.2.31. Appl., 28.5.25).—Two drums are mounted one above the other with heating flues extending through them. Combustion gases are passed through the flue in the lower drum prior to any substantial contact of the gases with the outside shells of the drums, then through the flue in the upper drum, and finally around the upper drum to a stack. The upper drum has an inlet for liquid, a pipe for conveying liquid from the upper to the lower drum, and a vapour line communicating with each drum. H. S. GARLICK.

**Distillation of hydrocarbon oils.** P. L. KRAUEL, ASSR. to STANDARD OIL Co. (U.S.P. 1,789,004, 13.1.31. Appl., 3.1.27).—Oil, after being heated in a conduit,

is passed into a vaporising drum, where the volatile fractions are removed. The residual oil is passed along with inert gas to a fractionating column where further vaporisation is brought about by means of steam. The vapours from the column are returned to the oil stream before it enters the vaporising drum.

T. A. SMITH.

**Separation of [petroleum oil] emulsions.** F. R. MOSER, Assr. to N. V. DE BATAAFSCHE PETROLEUM MAATS. (U.S.P. 1,788,684, 13.1.31. Appl., 3.9.24. Holl., 19.7.24).—High-boiling mineral oil, after pretreatment with  $\text{H}_2\text{SO}_4$  ( $d$  1.83) to remove resin-forming materials, is sulphonated by means of oleum. The reaction products form colloidal suspensions with  $\text{H}_2\text{O}$  and can be used to bring about the separation of petroleum emulsions.

T. A. SMITH.

**Conversion of hydrocarbons.** J. J. JAKOWSKY, Assr. to C. & C. DEVELOPING Co. (U.S.P. 1,792,744, 17.2.31. Appl., 22.7.24).—A substantially saturated vaporous mixture of mineral oil hydrocarbons is passed through a walled zone maintained above the crit. gasifying point of the motor fuel it is desired to produce, but below that of materially higher-boiling products, while simultaneously passing a high-tension, silent electrical discharge through the vapours. By such means the non-gaseous constituents are driven towards the walls and are converted, the gaseous constituents pass through the zone substantially without interruption, and a motor fuel is condensed from the volatile products leaving the converting zone.

H. S. GARLICK.

**Refining of mineral oils.** A. LACHMAN, Assr. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,790,622, 27.1.31. Appl., 22.6.27).—Cracked gasoline vapours at  $150^\circ$  are passed upwards through a mass of porous material over which flows a continuous stream of 70–85%  $\text{ZnCl}_2$  solution. Steam is added to replace the  $\text{H}_2\text{O}$  removed by the vapours and prevents the clogging of the apparatus. Stable water-white distillates are obtained without the use of  $\text{H}_2\text{SO}_4$ , and the loss does not exceed 2%. The product may require treatment with a small quantity of alkali to remove  $\text{H}_2\text{S}$ .

T. A. SMITH.

**Purification of cracked petroleum vapours.** C. B. WATSON and R. C. OSTERSTROM, Assrs. to PURE OIL Co. (U.S.P. 1,793,885, 24.2.31. Appl., 21.8.28).—Cracked petroleum vapours are continually passed to a fractionating zone into the upper part of which finely-divided fuller's earth or similar material is introduced and allowed to fall in substantially dry state in counter-current relation to the vapours. The heavier particles of oil and purifying agent are removed from the bottom of the fractionating zone, whilst the partly purified vapours are removed from the top to a separating zone. Finely-divided purifying agent is introduced into the vapours between the fractionating and separating zones from which the purified vapours are removed.

H. S. GARLICK.

**Refined viscous hydrocarbon oil.** T. H. ROGERS, Assr. to STANDARD OIL Co. (U.S.P. 1,793,134—5, 17.2.31. Appl., 7.11.28. Renewed 31.7.30).—Acid formation and increase of emulsibility in use of highly refined viscous hydrocarbon oils are prevented by the addition of (A)

small proportions of a naphthol, *e.g.*, 0.2% of  $\beta$ -naphthol, or (B)  $<0.1\%$  of an aromatic compound having more than one OH group attached to the ring, *e.g.*, 0.04% of pyrogallol.

H. S. GARLICK.

**Treatment of sulphur-containing [hydrocarbon] oils.** H. G. M. FISCHER and W. J. ADDEMS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,789,335, 20.1.31. Appl., 8.5.26).—Hydrocarbons containing mercaptans are refined by treatment with a heavy-metal sulphide, alkali, and sufficient S to convert the major portion of the mercaptans into polysulphides. After treatment the oils are distilled to obtain distillates practically free from S. A final treatment with a small quantity of "doctor" solution may be necessary.

T. A. SMITH.

**Continuous method for sweetening petroleum.** H. H. CANNON and W. W. GARY, Assrs. to CANNON-PRUTZMAN TREATING PROCESSES, LTD. (U.S.P. 1,789,167, 13.1.31. Appl., 21.1.29).—Oil containing mercaptans is refined by treatment with slaked lime or the hydroxide of metal of group II,  $\text{PbO}$ , diatomaceous earth, and S. The refining material is separated in the form of a filter cake, which will purify further large quantities of unrefined oil.

T. A. SMITH.

**Treatment of hydrocarbon oil with metallic halides.** J. L. COOLEY, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,795,761, 10.3.31. Appl., 1.12.26).—The oil is passed through a heating coil where it is raised to reaction temp. and anhyd.  $\text{AlCl}_3$  or equiv. metal halide catalyst is forced into the stream of hot oil. Low-boiling oils are removed by passage through a flashing chamber from which the main bulk of oil is introduced into one end of a conversion chamber or still maintained at sufficient temp. and pressure to cause decomp. of the oil. Simultaneously the separated vapours from the flashing chamber are withdrawn and discharged into the reaction chamber through a distributor at a lower point so that they rise upwardly through the contents of the still, which thereby are agitated. Mechanical agitators may also be provided to break up tarry matter and coke in the chamber. Part of the mixture in the still is passed to a stratifying zone from which the spent  $\text{AlCl}_3$  residue or tar is withdrawn.

H. S. GARLICK.

**Petroleum-treating system.** S. J. DICKEY and E. W. ROTH, Assrs. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,792,003, 10.2.31. Appl., 15.11.26).—Oil is passed under pressure through a series of tanks into the last of which is introduced a mixture of purified oil and fresh treating reagent. A mixture of purified oil and reagent sludge is drawn in each instance from the relatively next succeeding tank of oil and is introduced into each of the preceding tanks.

H. S. GARLICK.

**Conversion of residues obtained on sweetening petroleum distillates.** H. G. M. FISCHER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,795,278, 3.3.31. Appl., 25.8.27).—Residue oil containing alkyl polysulphides obtained by catalytic treatment of sour petroleum distillates with S followed by distillation under reduced pressure to produce a sweetened distillate and a residue oil containing hydrocarbon-S compounds,



is heated to  $>177^\circ$ , but below the temp. of vaporisation of a substantial part of the residue oil, to form a vapour comprising  $\text{H}_2\text{S}$  and hydrocarbon-S compounds of lower b.p. than those originally present, which may be separated by appropriate means. H. S. GARLICK.

**Treating [dewaxing of] viscous hydrocarbon oil.** R. E. MANLEY, ASSR. to TEXAS CO. (U.S.P. 1,792,234, 10.2.31. Appl., 15.4.27).—Wax-bearing, hydrocarbon lubricating oil is mixed with a suitable more volatile carrier oil, derived from a naphthenic-base crude and less volatile than kerosene, and distilled under reduced pressure, thereby removing the carrier oil and more volatile low-melting wax together. The remaining oil is cooled and chilled to ppt. the high-melting wax.

H. S. GARLICK.

**Separation of paraffin from petroleum oils.** J. VERNER, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,789,347, 20.1.31. Appl., 16.8.27).—Cylindrical filters are arranged in a container which is also fitted with heating tubes. Chilled wax-containing oil is pumped through the vessel, the scale being retained. After sufficient wax has collected, steam is passed through the heating tubes and the molten slack separately collected. T. A. SMITH.

**Purification of [lubricating] oil.** L. H. CLARK, ASSR. to SHARPLES SPECIALTY CO. (U.S.P. 1,788,911, 13.1.31. Appl., 16.12.25).—Used lubricating oil is treated at  $80$ – $95^\circ$  with a dil. solution of  $\text{Na}_2\text{SiO}_3$ ,  $\text{Na}_2\text{Al}_2\text{O}_4$ , or  $\text{Na}_3\text{PO}_4$  containing a small proportion of colloidal substance. The mixture is then centrifuged and the oil freed from light constituents by distillation in steam. T. A. SMITH.

**Production of hydrocarbon vapour or gas under pressure for burners and the like.** H. GAMARD (B.P. 355,683—4, [A] 22.5.30, [B] 23.5.30. Fr., [A] 30.5.29).—(A) The gasifying device consists of a tube depending into a reservoir of the liquid hydrocarbon, heated either electrically or by heat conveyed from the burner by a surrounding conducting tube. An insulating layer of air separates the heated tube from the bulk of liquid in the reservoir. The outlet orifice for the gas is formed by a jet disposed above the level of the liquid in the reservoir. (B) The gasifying tube and electrical heater are designed to form a separate unit which can be removed, exchanged, or replaced in the reservoir as desired. A. B. MANNING.

**Resolution of hydrocarbon vapour.** F. A. HOWARD and N. E. LOOMIS, ASSRS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,797,145, 17.3.31. Appl., 25.10.27).—A mixture of hydrocarbons, chiefly compounds of the gasoline range, from a cracking process is subjected to partial condensation under superatm. pressure to produce a first condensate requiring the usual chemical treatment with  $\text{H}_2\text{SO}_4$  or "doctor" solution, and a second vapour which requires no chemical treatment and has an end-point of approx.  $150$ – $215^\circ$ . This is separated into a gas and liquid products substantially free from normally gaseous hydrocarbons which are subsequently blended with the chemically treated first condensate. H. S. GARLICK.

**Separation of the oxidation products of solid hydrocarbons, waxes, etc.** J. Y. JOHNSON. From

I. G. FARBENIND. A.-G. (B.P. 352,512, 13.1.30).—The crude oxidation product is centrifuged, with or without diluent, at a temp. at which the constituents to be removed are liquid; preferably high-speed sieve centrifuges lined with wool or flannel filters or with fine-meshed Al sieves are used. C. HOLLINS.

**Refining of wax-containing oil by distillation.** SHARPLES SPECIALTY CO., ASSEES. of L. D. JONES (B.P. 356,172, 28.4.30. U.S., 14.6.29).—The vapour of the oil is passed into a fractionating tower consisting of groups of bubble trays forming alternately rectifying and stripping sections. Part of the reflux from any rectifying section is passed to the next lower rectifying section, whilst the remainder is passed into the stripping section and then withdrawn from the tower. By regulation of the temp. and quantity of reflux, part of the wax is obtained in one of the fractions from which it is deposited on cooling in a cryst. form removable by filter-pressing, whilst the remainder is obtained in an adjacent fraction from which it is deposited in an amorphous state removable by centrifuging.

D. K. MOORE.

**Recovery of paraffin wax from materials containing the same.** J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 365,205, 4.6.30. Addn. to B.P. 291,817; B., 1928, 613).—Substances containing paraffin wax, e.g., hydrogenation and distillation products of coal, are treated with a mixture of a lower alkyl formate, e.g.,  $\text{HCO}_2\text{Me}$ , and one or more org. solvents, e.g.,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ ,  $\text{CCl}_4$ , to produce white paraffin wax.

D. K. MOORE.

**Lubricant.** D. P. BARNARD, ASSR. to STANDARD OIL CO. (U.S.P. 1,796,857, 17.3.31. Appl., 19.10.27).—A lubricating oil suitable for the lubrication of an internal-combustion engine under operating conditions is mixed with a relatively small amount (5–10%) of a volatile, substantially non-inflammable diluent, preferably a chlorinated hydrocarbon derivative, b.p. below  $120^\circ$ , e.g.,  $\text{CCl}_4$ , in order to ensure ease of starting with elimination of the diluent without fire risk after a relatively short time of full-load operation. H. S. GARLICK.

**Manufacture of sulphonic lubricating material.** H. L. JOHNSON, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,795,491, 10.3.31. Appl., 8.9.24).—A lubricant especially adapted for use in the die-drawing of metals comprises a metallic (Na) salt of the green sulphonic acid obtained from the sludge formed by treating petroleum lubricating oil with conc.  $\text{H}_2\text{SO}_4$  (15–30%), a normally solid, non-mineral, substantially unsaponified oleaginous material, e.g., tallow (1–15%), and  $\text{H}_2\text{O}$ , the materials being heated to  $82^\circ$  to effect a homogeneous mixture. H. S. GARLICK.

**Slushing grease.** A. H. BRANSKY, ASSR. to STANDARD OIL CO. (U.S.P. 1,795,993, 10.3.31. Appl., 2.3.25).—A rust-preventative consists of a mixture of wax (e.g., montan or carnauba) of high m.p., or saponification products thereof, and an oil-sol. compound (20–60%) derived from the sulphonation of hydrocarbon oils with conc.  $\text{H}_2\text{SO}_4$ , brought to suitable consistency by the addition of hydrocarbon oil. H. S. GARLICK.

**Filter element [for oil].** Distillate-chilling apparatus. Gas scrubber. Viscosity indicators.

Antifriction bearing.—See I. Treatment of basic or acidic substances.—See III. HCN.  $(\text{NH}_4)_2\text{SO}_4$ .—See VII. Material for building etc.—See IX.

### III.—ORGANIC INTERMEDIATES.

Determination of *m*-xylene in mixed xylois. H. P. REICHEL (Chem.-Ztg., 1931, 55, 744).—To 5 c.c. of the mixed xylois are slowly added 20 c.c. of glacial AcOH, 25 c.c. of  $\text{HNO}_3$  (*d* 1.5), and 40 c.c. of  $\text{H}_2\text{SO}_4$  (*d* 1.83), and the whole is kept at 15° for 30 min. by ice water. The mixture is heated on the water-bath for 1–2 hr., cooled, and diluted with 450 c.c. of  $\text{H}_2\text{O}$ , and, after keeping for  $\frac{1}{2}$  hr., the mixed crystals are filtered and washed with  $\text{H}_2\text{O}$ . These, consisting of 2 : 4 : 6-trinitro-*m*-xylene and 2 : 3 : 5-trinitro-*p*-xylene, are digested on a water-bath for 30 min. with 100 c.c. of  $\text{COMe}_2$ . The 2 : 4 : 6-compound which crystallises from the cooled solution is collected on a filter and dried; 0.6 g. is added to the observed wt. to allow for its solubility in  $\text{COMe}_2$ . C. RANKEN.

Naphthenic acid soaps.—See II. Determination of  $\text{CH}_2\text{O}$ .—See XX.

#### PATENTS.

Manufacture of alcohols [from oxidised hydrocarbons]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,537, 11.4.30).—The products obtained by destructive air- or  $\text{O}_2$ -oxidation of liquid hydrocarbons or fractions of such products are heated under pressure with large excess of  $\text{H}_2$  in presence of a catalyst (Ni or Co). Mixtures of alcohols of high m.p. containing  $\text{C}_{10}$ – $\text{C}_{15}$  and useful in ointments and for prep. of wetting agents (by sulphonation) are obtained. C. HOLLINS.

Production of amides. SOC. FRANÇ. DE CATALYSE GÉNÉRALISÉE (B.P. 352,517, 5.3.30. Fr. 7.12.29).—Formamide, prevented from dehydration to HCN by the presence of  $\text{NH}_3$  under pressure, is heated at 150–300° with MeOH to give  $\text{NH}_2\text{Ac}$ , and, generally, with R-OH to give R-CO-NH<sub>2</sub>. C. HOLLINS.

Acetaldehyde condensation reactions. E. J. BOAKE and L. W. E. TOWNSEND (B.P. 352,056, 29.3.30).—In the conversion of MeCHO into aldol or paraldehyde the heat of reaction is wholly or partly controlled by supplying the necessary  $\text{H}_2\text{O}$  by endothermic dehydration of a hydrated salt, which is preferably a catalyst for the condensation. Pure MeCHO, *e.g.*, is stirred at 10° with an equal wt. of  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ , or with a mixture of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  and a little anhyd.  $\text{Na}_2\text{CO}_3$  at 15–16°, to give aldol. For paraldehyde a mixture of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  and  $\text{NaHSO}_4$  at 12–18° is used. C. HOLLINS.

Preparation of aliphatic acid anhydrides especially acetic anhydride. G. B. ELLIS. From C. F. BOEHRINGER & SÖHNE G.M.B.H. (B.P. 352,176, 22.5.30).— $\alpha\alpha'$ -Diacetoxydiethyl ether, or the crude product from  $\alpha\alpha'$ -dichlorodiethyl ether and AcOH or an acetate, is heated (preferably in a solvent, *e.g.*, decahydronaphthalene,  $\text{COMe}_2$ , or EtOH) in presence of a non-basic catalyst ( $\text{ZnCl}_2$ ,  $\text{SnCl}_2$ ,  $\text{SO}_2$ ), whereby the ether is decomposed smoothly into MeCHO (2 mols.) and  $\text{Ac}_2\text{O}$  (1 mol.), which are separated by distillation. Thus  $\alpha\alpha'$ -dichlorodiethyl ether is converted into acetate by

heating with AcOH in  $\text{C}_6\text{H}_6$  at 80° for 4–5 hr., the  $\text{C}_6\text{H}_6$  is distilled off, and the residue heated with 0.2% of  $\text{ZnCl}_2$  in AcOH; the yield of  $\text{Ac}_2\text{O}$  is 75% of the theoretical. C. HOLLINS.

Manufacture of [ $\beta$ ]-chloropropionic esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,802, 8.9.30).—Acrylonitrile is treated with HCl and the  $\beta$ -chloropropionitrile obtained is converted into esters by means of an alcohol and HCl. The Me, Et,  $\text{CH}_2\text{Ph}$ ,  $\text{OH} \cdot \text{CH}_2 \cdot \text{CH}_2$ ,  $\text{ClCH}_2 \cdot \text{CH}_2$ , and  $\text{EtO} \cdot \text{CH}_2 \cdot \text{CH}_2$  esters are thus prepared in 92–98% yields. C. HOLLINS.

Production of pure high-grade vinyl chloride. I. G. FARBENIND. A.-G. (B.P. 352,842, 13.10.30. Ger., 18.10.29).— $\alpha\alpha\beta$ -Trichloroethane is treated above 50° with fine or coarse Zn, Fe, or Al in presence of  $\text{H}_2\text{O}$ ; *e.g.*, it is passed into an aq. suspension of Zn dust at 50–60°, or is heated with  $\text{H}_2\text{O}$  and Fe filings at 100–120°/5–6 atm., or is passed with steam over Fe filings at 300°. C. HOLLINS.

Manufacture of acetals. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,474, 4.4.30).—Non-carboxylated mono- or poly-hydric alcohols or phenols (except MeOH) are converted into acetals by combination with vinyl ethers, preferably in presence of acid catalysts ( $\text{H}_2\text{SO}_4$ ,  $\text{BF}_3$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{SOCl}_2$ ,  $\text{NaHSO}_4$ ,  $\alpha$ -chlorodiethyl ether,  $\text{CaCl}_2$ ) and a solvent ( $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , or excess of the alcohol). The following preps. are described: ethylidene Et *n*-Bu ether, b.p. 151–152°, from vinyl Et ether, BuOH, and  $\text{H}_2\text{SO}_4$ ; ethylidene Ph Et ether, b.p. 83–84°/10.5 mm., from vinyl Et ether, PhOH, and  $\text{EtO} \cdot \text{C}_2\text{H}_4\text{Cl}$ ; ethylidene Et  $\beta$ -chloroethyl ether, b.p. 53–56°/10 mm., from vinyl Et ether, ethylene chlorohydrin, and  $\text{SOCl}_2$ ; glycol  $\alpha\alpha'$ -diethoxydiethyl ether, b.p. 97°/13 mm., from vinyl Et ether and  $\text{EtO} \cdot \text{C}_2\text{H}_4\text{Cl}$ ; ethylidene cholesteryl Et ether from vinyl Et ether, cholesterol, and  $\text{EtO} \cdot \text{C}_2\text{H}_4\text{Cl}$ . C. HOLLINS.

Manufacture of alkyl- or aryl-oxyethylidene esters [ $\alpha$ -alkoxy- or aryloxy-alkyl esters]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,070, 4.4.30).—A vinyl aryl or alkyl ether is treated with an org. or a non-oxidising inorg. acid in presence of accelerating agents ( $\text{BF}_3$ ,  $\text{H}_2\text{SO}_4$ , HCl,  $\text{SOCl}_2$ ,  $\text{NaHSO}_4$ ,  $\text{KHF}_2$ ,  $\text{CaCl}_2$ ,  $\alpha$ -chlorodiethyl ether, etc.), at raised temp. and pressure, if desired, and in a diluent. Vinyl Et ether and HCl at 0° give  $\alpha$ -chlorodiethyl ether, b.p. 92–95°;  $\alpha$ -chloroethyl *n*-Bu ether, b.p. 120–130°, is similarly prepared and gives with KOAc  $\alpha$ -*n*-butoxyethyl acetate, b.p. 166–172°. Ph  $\alpha$ -chloroethyl ether, b.p. 79–81°/12 mm.,  $\alpha$ -ethoxyethyl acetate, b.p. 128–130°, and salicylate, b.p. 84–85°/0.025 mm., and  $\alpha$ -*n*-butoxyethyl salicylate, b.p. 92–93°/0.006 mm. (decomp.), are also described. C. HOLLINS.

Treatment of basic or acidic substances produced by destructive hydrogenation. IMPERIAL CHEM. INDUSTRIES, LTD., W. R. MADEL, and E. W. FAWCETT (B.P. 355,213, 18.2.30).—The crude basic or acidic substances, *e.g.*, phenols, mixtures of quinoline and isoquinoline, are separated into their components by selective extraction or fractional crystallisation in light petroleum at low temp., *e.g.*, –10° to –40°.

A. B. MANNING.

**Manufacture of complex salts [of pyrocatechol-disulphonic acids].** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 351,971, 1.4.30).—An alkali salt of pyrocatecholdisulphonic acid or its OH derivatives is treated in aq. solution with aq.  $\text{CaCl}_2$  or  $\text{Ca}(\text{NO}_3)_2$  and either  $\text{Ca}(\text{OH})_2$  or caustic alkali; or the alkali salt is acidified and then neutralised with  $\text{Ca}(\text{OH})_2$ . The salts contain Na and Ca, the latter being in part attached to phenolic and in part to sulphonic O. They are suitable for injection. C. HOLLINS.

**Manufacture of *m*-hydroxydiarylaminocarboxylic acids.** W. W. GROVES. From I. G. FARBEIND. A.-G. (B.P. 352,644, 20.5.30. Addn. to B.P. 333,783; B., 1930, 1016).—A *m*-hydroxydiarylamine carrying Me *meta* or *para* to the OH group is carboxylated. Products from 5-methyl- (m.p. 195°), 4'-chloro-5-methyl- (m.p. 158°), 6-methyl- (m.p. 179°), 4'-chloro-6-methyl- (m.p. 200°), and 4':6-dimethyl- (m.p. 204°) derivatives of 3-hydroxydiphenylamine are described. C. HOLLINS.

**Hydrogenation of aromatic compounds [dihydro-eugenols].** BOOTS PURE DRUG CO., LTD., and J. MARSHALL (B.P. 352,663, 28.5.30).—Eugenol, isoeugenol, and corresponding *o*-compounds are treated with  $\text{H}_2$  in presence of Cu at 250–270° or of Ni at 150–170°, whereby only the side-chain double linking is reduced. 4-*n*-Propylguaiacol, m.p. 18–19°, and the 6-isomeride, m.p. 24°, are prepared in pure state. C. HOLLINS.

**Manufacture of a quinine double salt.** H. MEFFERT (B.P. 352,115, 15.4.30).—Quinine lactate and Ca lactate are cryst. together from mixed solutions, with or without addition of Na citrate. The prep. of quinine lactate from the sulphate is described. C. HOLLINS.

**Washing, cleansing, emulsifying, and wetting-out agents.** DEUTS. HYDRIERWERKE A.-G. (B.P. 356,694, 30.4.30. Ger., 30.4.29).—Piperidine, hydrogenated quinoline bases, or their homologues and/or substitution derivatives are combined with org. sulphonic acids, e.g., Turkey-red oil; the products are sol. in all org. solvents and suitable for use as boring and textile oils etc. [Stat. ref.] E. LEWKOWITSCH.

**Manufacture of  $\alpha$ -naphthol.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 352,568, 16.4.30).—1-Keto-1:2:3:4-tetrahydronaphthalene is dehydrogenated by heating with S at 190–200°, especially in presence of Se,  $\text{As}_2\text{O}_3$ , Sn, or ZnO with diphenylguanidine. C. HOLLINS.

**Manufacture of halogenated cyclic ketones of the acenaphthene series.** W. W. GROVES. From I. G. FARBEIND. A.-G. (B.P. 352,597, 30.4.30. Addn. to B.P. 342,373; B., 1931, 476).—Acenaphtha-5:6-indandione [5:6-malonylacenaphthene] and its derivatives, especially the monoimides, are halogenated, e.g., with  $\text{KClO}_3$  and HCl,  $\text{SO}_2\text{Cl}_2$ , or halogens. The simple monoimide gives on chlorination a trichloro-compound,  $\begin{array}{c} \text{CH}_2 \\ \diagup \quad \diagdown \\ \text{C}_{10}\text{H}_4 \\ \diagdown \quad \diagup \\ \text{CH}_2 \end{array} \begin{array}{l} \text{C:NCl} \\ \text{CO-CCl}_2 \end{array}$ , m.p. about 180°, which is hydrolysed by hot AcOH to the dichlorodiketone; the dibromo-bromoinide, m.p. 169° (decomp.), and a chlorinated product from the 3-chloro-derivative are described. C. HOLLINS.

**Manufacture of  $\omega$ -aminomethylbenzanthrone.** I. G. FARBEIND. A.-G. (B.P. 352,626, 12.5.30. Addn. to B.P. 249,883; B., 1927, 572).—Benzanthrone is condensed in  $\text{PhNO}_2$  with chloromethylphthalimide in presence of  $\text{ZnCl}_2$  at 130–150° and the product, m.p. 288°, is hydrolysed, e.g., with aq. NaOH, to give  $\omega$ -aminomethylbenzanthrone, m.p. 154–156°.

C. HOLLINS.

**Production of di- and isodi-benzanthrones.** SELDEN CO., ASSECS. OF L. C. DANIELS (B.P. 352,076, 4.4.30. U.S., 4.4.29).—Furfuraldehyde or furyl alcohol is used in place of dextrin etc. as the reducing agent in the alkaline fusion of benzanthrone. C. HOLLINS.

**Solutions for cooling systems etc.**—See I.  $\text{C}_2\text{H}_2$ . PhOH etc. from oils.—See II.

#### IV.—DYESTUFFS.

##### PATENTS.

**Manufacture of [polymethine] dyes. [Photographic sensitisers.]** F. M. HAMER, and ILFORD, LTD. (B.P. 351,555, 28.3.30).—A glutaric aldehyde, produced *in situ* from a pyridinium salt, is condensed with a heterocyclic  $\text{NH}_4$  (other than pyridinium) salt carrying a reactive  $\alpha$ - or  $\gamma$ -Me group to give dyes which are photo-sensitisers for infra-red rays; e.g., 1-*o*-dinitrophenylpyridinium chloride in EtOH is treated hot with NaOEt in EtOH and then at once with 2-methyl-benzthiazole ethiodide (2 mols.). C. HOLLINS.

**Manufacture of acid wool dyes [from naphthalic anhydride].** I. G. FARBEIND. A.-G. (B.P. 352,139, 29.4.30. Addn. to B.P. 299,721; B., 1929, 455).—Naphthalic or an aminonaphthalic anhydride is condensed with a hydroaromatic amine or with a hydrazine and sulphonated or converted into a sulphamic acid; or the sulphonic or sulphamic group may be present in the starting material. Examples are: sulphonated 4-amino-1:8-naphthalic anhydride with hydrazine (greenish-yellow), phenylhydrazine (yellow), cyclohexylamine (greenish-yellow), or *p*-cyclohexylaniline (yellow); 4-amino-1:8-naphthalic cyclohexylimide sulphonated with oleum at 100° (greenish-yellow). C. HOLLINS.

**Manufacture of halogen-containing vat dyes of the dibenzanthrone series.** I. G. FARBEIND. A.-G. (B.P. 351,948, 25.1.30. Ger., 27.4.29).—Chlorobromodibenzanthrones are obtained by treating halogenated dibenzanthrones (except I-derivatives) with halides of Fe, Al, Sb, Hg, with or without further halogenating agents, catalysts, solvents, diluents, or substances which lower the m.p. (Ca, Sr, and Ba halides). Examples are: dibromodibenzanthrone with  $\text{FeCl}_3$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 140–150°, or with  $\text{AlCl}_3$ , NaCl, and I at 180°, or with  $\text{HgCl}_2$  in  $\text{C}_6\text{H}_4\text{Cl}_2$ , or with  $\text{FeCl}_3$  in collidine at 120°, or with  $\text{FeCl}_3$  and  $\text{FeBr}_3$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 140–150°; monobromodibenzanthrone with  $\text{FeCl}_3$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 140–150°; tribromodibenzanthrone with  $\text{FeCl}_3$  and NaCl at 150°; tetrabromodibenzanthrone or dichlorotetrabromodibenzanthrone with  $\text{FeCl}_3$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 140–150°; pentabromodibenzanthrone with  $\text{SbCl}_5$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 80–100°; bromonitrodibenzanthrone with  $\text{FeCl}_3$  in  $\text{C}_6\text{H}_5\text{Cl}_3$  at 120–130°. The products are blue or navy-blue vat dyes fast to rain drops. C. HOLLINS.

**Manufacture of stable reduction compounds of vat dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 352,283, 1.8.30. Addn. to B.P. 334,878 and 334,920; B., 1930, 1144).—6:6'-Diethoxythioindigo is reduced as described in the prior patents.

C. HOLLINS.

**Manufacture of chromiferous [azo] dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,004, 31.1.30).—An aminosulphosalicylic acid (e.g., the 5:3- or 3:5-compound) is diazotised and coupled with resorcinol, and the product is prechromed to give a leather brown. [Stat. ref.]

C. HOLLINS.

**Manufacture of polyazo [direct] dyes.** J. R. GEIGY A.-G. (B.P. 352,368 and Addn. B.P. 352,878, [A] 29.11.30, [B] 1.12.30. Ger. [A, B], 29.11.29).—(A) An aminodisazo compound,  $A \rightarrow B \rightarrow C$ , in which  $A$  is an amino-carboxylic or -sulphonic acid of the  $C_6H_4$  or  $C_{10}H_8$  series and  $B$  and  $C$  are  $\alpha$ -naphthylamine or Cleve acid, is diazotised and coupled with a monoazo dye obtained by coupling  $J$ -acid in acid medium with a diazotised arylamine ( $D$ ). In the examples:  $A = 4$ -chlorometanilic acid,  $B = C =$  Cleve acid, and  $D =$  metanilic acid;  $A = 2:4$ -8-naphthylaminedisulphonic acid,  $B = \alpha$ -naphthylamine,  $C =$  Cleve acid, and  $D = 5$ -aminosalicylic acid. The shades are greenish-blue. (B) Greener shades are obtained by using 2-ethoxy-naphthylamine-6-sulphonic acid as third component; e.g., metanilic acid  $\rightarrow$  Cleve acid  $\rightarrow$  2-ethoxy- $\alpha$ -naphthylamine-6-sulphonic acid  $\rightarrow J$ -acid  $\leftarrow$  metanilic acid (blue-green).

C. HOLLINS.

**Photographic bleach-out layers.**—See XXI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Porosity: a primary property of textiles.** I. N. H. CHAMBERLAIN. II. H. J. WOODS (J. Soc. Dyers and Col., 1931, 47, 289–292).—Schofield's views (B., 1931, 152, 798) are criticised. This author's observations on the milling of wool fabrics do not establish any fundamental law of felting. The equations chosen to express some of his results are considered to be unsuitable and the conclusions drawn from the experimental data to need radical revision.

B. P. RIDGE.

**Porosity: a primary property of textiles.** J. SCHOFIELD (J. Soc. Dyers and Col., 1931, 47, 292–293).—A reply to the criticisms of Chamberlain and Woods (cf. preceding abstract).

B. P. RIDGE.

**Determination of solubility number: micro-method for measuring the extent to which a cellulosic material has been chemically modified or degraded.** C. R. NODDER (J. Text. Inst., 1931, 22, 416–424 T).—The material is first boiled for 6 hr. with a 2% solution of NaOH, and the solubility of the dry product on treatment with 10N-NaOH at 15° and subsequent dilution to 2N is determined by oxidation of 2 c.c. of the filtered solution with  $CrO_3$ . The solubility no. is the % of cellulose dissolved by this treatment under the stated conditions, and its measurement affords a reliable means of estimating the extent to which the material has been degraded by chemical attack. For the initial stages of such attack the cuprammonium viscosity method is the more sensitive,

but when the degree of attack is high the reverse obtains. The amount (0.1 g.) of material required is very small and the method is very suitable for use with linen goods.

B. P. RIDGE.

**Effect of sulphite-cooking on the weight of the wood-pulp fibres.** A. LAMPEN and I. KOSKINEN (Zellstoff u. Papier, 1931, 11, 573–575).—The wt. of the individual fibres decreases progressively with increasing degree of decomp. of the pulp, as indicated by decreasing Tingle and Bergmann Cl nos., but the decrease in wt. is more rapid than corresponds with the decrease in yield found in technical cooking. A method of estimating the wt. of  $10^6$  single fibres is described, and with hard pulps the discrepancy may be due to the difficulty of effecting complete separation of the fibres before they are counted.

B. P. RIDGE.

**Mechanical properties and testing of rayon yarns.** E. BERL and H. STAUDINGER (Chem.-Ztg., 1931, 55, 749–752).—In the measurement of breaking load and extensibility, the atm. temp. and humidity, length of specimen, and rate of fall of the lower grip on the testing machine should be standardised, whilst before conditioning the yarns in the required atm. they should be dried at 105° in order to avoid hysteresis effects. The slower the rate of breaking, the smaller is the breaking load up to a velocity of 25 cm./sec., beyond which the effect of time is small. Dry strength also decreases progressively with increasing humidity. For all varieties of yarn the wet strength is proportional to the dry strength, and since for measurement of the former the control of humidity and temp. and drying the yarn are unnecessary, this test is to be preferred for technical purposes. Before measuring wet strength, the yarns should be steeped for 20 min. in a 0.5% solution of Nekat AE.

B. P. RIDGE.

**Measurement of viscosity of solutions of cellulose in cuprammonium hydroxide solution.** A capillary tube viscosimeter. R. W. KINKEAD (J. Text. Inst., 1931, 22, 441–415 T).—The cellulose solutions are prepared separately in a bottle which is connected to a bulb containing a coarse, fritted glass filter and thence to a capillary viscosimeter and reservoir. The air in the system is replaced by  $H_2$  and the solution is filtered through the bulb before passing to the viscosimeter. In this way the insol. portion of substances such as linen may be retained for analysis, and clogging of the capillary is prevented. Any number of determinations of the time of flow can be made, and kinetic energy corrections are generally unnecessary.

B. P. RIDGE.

**Rate of deformation of cellulose nitrate-camphor films under static stresses.** J. G. McNALLY and S. E. SHEPPARD (J. Physical Chem., 1931, 35, 2498–2507).—Curves showing the rate of extension under const. stress plotted against time of cellulose nitrate films containing up to 50 wt.-% of camphor have the same shape for the different camphor contents, but the stress corresponding with similar rates of elongation varies with the composition of the films;  $\log R = kS$  approx., where  $R$  is the rate of extension for a stress  $S$ . An increase in camphor content of the film causes a progressive decrease in the stress at which extension

commences, up to a content of 35%; from 35 to 50% no shift in the curves takes place. Below 35% of camphor, celluloid consists of cellulose nitrate + a cellulose nitrate-camphor complex, and above, of camphor + the complex.

L. S. THEOBALD.

**Stability test for cellulose nitrate film.** J. P. LAWRIE (J.S.C.I., 1931, 50, 377—378 T).—A modified form of "German heat test" at 135° and the apparatus (B.E.S.A. No. 2 D. 8) subsequently used are described. Details and results of a series of physical, photographic, and chemical tests are given. It is concluded that ageing of the nitrate film base, as indicated by the stability test, does not materially affect the coated emulsion over a prolonged period of time. Results of the heat test on films of varying age, some over 20 years, are given and several modifications are suggested for the improvement of the test.

**Softeners for cellulose acetate.** H. MESSER (J.S.C.I., 1931, 50, 386—387 T).—Condensation products of low-boiling solvents and softeners (for cellulose acetate) with bigger appropriate mol., in order to give non-volatile  $H_2O$ -insol. liquid softeners, are described. In this connexion compounds made from glycerol derivatives,  $COMe_2$ , and esters, with dicarboxylic and hydroxy-carboxylic acids and  $NH_2Ph$  are discussed.

**Crude fibre.**—See XV. **Storage of nitrocellulose.**—See XXII.

#### PATENTS.

**Production of cellulose from fibrous vegetable material.** O. C., G. S., and O. H. STRECKER (B.P. 354,462, 7.6.30. Ger., 7.6.29).—An improved phenoxide process (B.P. 284,846; B., 1928, 364) is described. The hot liquor containing, *e.g.*, 5—6% of NaOH, is introduced in batches simultaneously at the top and bottom of the digester to destroy enzymes and aerobic ferments present in the wood. The temp. is then held at 110—125° for about 2 hr. without addition of alkali, after which heating is discontinued and about 25% of the liquor is removed and replaced by an equal vol. of aq. NaOH sufficiently conc. to bring the proportion of NaOH in the liquor to 6—6.5%. This alkali is added over a period of 0.5—1 hr. in such a way that contact of uncombined NaOH with the cellulose is avoided. The temp. is then raised to 170—175° and digestion continued for a further 3 hr. The liquor should at all times contain at least a 50% excess of phenolic substances. Hydrocarbons formed during digestion are distilled off during or after the cook to prevent staining of the cellulose. Surplus spent liquor is treated with  $CO_2$ , optionally after boiling with  $Ca(OH)_2$  to remove colloidal impurities, and the precipitated phenolic substances are used either for preparing fresh cooking liquor or for biological purposes, *e.g.*, as fertilisers.

D. J. NORMAN.

**Manufacture of cellulose derivatives.** L. LILLENFELD (B.P. 356,776, 14.3.30).—Bleached or unbleached cellulose or a conversion product of cellulose is treated with a halogenated olefine in the presence of alkali. Thus matured or unmatured alkali-cellulose (containing aq. NaOH) is autoclaved for 3—4 hr. at 90—100° with  $C_2HCl_3$ ,  $C_2H_2Cl_2$ , or  $C_2H_3Cl$ , optionally in the presence

of a catalyst, *e.g.*, a salt of Cu, Fe, Ni, etc. The resulting product, after purification by, *e.g.*, dissolution in 5—8% aq. NaOH and precipitation with acid, is obtained as a white powder insol. in  $H_2O$  and org. solvents, but sol. in 5—10% aq. NaOH to a clear viscous solution suitable for the manufacture of filaments, films, etc. or for use as a sizing or thickening agent for textiles.

D. J. NORMAN.

**Production of artificial materials [rayon] by wet spinning from solutions of organic derivatives [acetate] of cellulose.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 340,324, 5.11.28 and 11.4.29).—A 20—25% solution of cellulose acetate in  $COMe_2$  is extruded through spinnerets into a coagulating liquid containing a high proportion (25—65%) of a solvent for the cellulose acetate, *e.g.*, diacetone alcohol, Et tartrate or lactate, together with a volatile solvent, *e.g.*,  $COMe_2$ . Mixtures mentioned are 1:1 diacetone alcohol-EtOH, 30:15:55 diacetone alcohol- $COMe_2$ - $H_2O$ ; 60:40 diacetone alcohol- $H_2O$ , and 45:65  $H_2O$ -Et lactate. The plastic filaments may be stretched before drying and an oil incorporated with the cellulose acetate solution to improve the lustre of the rayon.

A. R. POWELL.

**Production of artificial materials by wet-spinning processes from solutions of organic derivatives of cellulose.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 340,325—6 and 340,436—7, [A] 18.1.29, [B] 11.4.29, [C, D] 5.11.28. Addns. [A—D] to B.P. 340,324; preceding).—Modifications of the parent patent consist in (A) effecting coagulation and/or subsequent treatment with a large proportion (60—100%) of a non-solvent plasticiser (triaryl phosphates), preferably diluted with a solvent (acetone) and/or a non-solvent. To render the filaments plastic and capable of coalescence so that they can be stretched, a solvent (diacetone alcohol, diethyl tartrate, ethyl lactate), which is less volatile than the non-solvent of the coagulating bath (water), together with a more volatile solvent (acetone), is (C) incorporated in the spinning solution, wholly or in part in place of solvents in the coagulating bath, or in (D) is used as a secondary bath after coagulation, or is present in the coagulating bath initially, or introduced therein by the spinning solution, the filaments being lubricated during coagulation or secondary treatment and excess of solvent removed by washing with water, salt, or weaker solvent solutions. In (B) the filaments produced by the various processes, after leaving coagulating or subsequent treatment baths above atmospheric temperature, are dried in a chamber by a countercurrent of air.

F. R. ENNOS.

**Manufacture of artificial threads and other products from cellulose compounds.** L. LILLENFELD (B.P. 356,286, 10.3.30. Addn. to B.P. 335,906; B., 1931, 153).—The reaction product of cellulose xanthate with one or more halogen derivatives of di- or poly-hydric alcohols described in the prior patent is isolated by precipitation with an acid, acid salt, or alcohol, or spontaneously. The product is washed if desired, and, optionally after reconversion into a sol. xanthate, is dissolved and spun as usual.

F. R. ENNOS.

**Manufacture of fine artificial fibres by the wet-spinning process.** I. G. FARBERIND. A.-G. (B.P. 356,427, 13.8.30. Ger., 14.8.29).—The thread is drawn from the spinning nozzle by two rollers the peripheral speed of which is such that the draught or degree of stretching between the nozzle and the first roller is  $< 1$ , whilst that between the first and second rollers is  $> 1$ , the total draught being  $>$  or  $< 1$ . F. R. ENNOS.

**Spinning of cuprammonium silk by the stretch-spinning process.** BRIT. BEMBERG, LTD. (B.P. 356,457, 28.8.30. Ger., 29.8.29).—The jet of precipitating liquid is deviated from the rectilinear path of the thread, before or at the outlet of the spinning apparatus but before the collecting or guiding device, by means of a siphon or suitably inclined rod or strip.

F. R. ENNOS.

**Spinning of artificial silk.** I. G. FARBERIND. A.-G. (B.P. 334,030, 29.7.29. Ger., 2.10.28).—Unripened alkali-cellulose (viscose) solutions with an alkali : cellulose ratio of  $> 1:1$  are spun into a bath containing coagulating salts and not more than 47% of  $H_2SO_4$ . The resulting filaments are stretched while in the coagulating bath.

A. R. POWELL.

**Spinning of artificial silk.** I. G. FARBERIND. A.-G. (B.P. 335,675, 30.7.29. Ger., 8.11.28. Addn. to B.P. 334,030; preceding).—Viscose made from unripened alkali-cellulose, in which the proportion of alkali to cellulose is greater than 1:1, is spun into a bath containing an ammonium salt (sulphate, acetate, etc.), with or without the addition of a small amount of acid and of another neutral salt (sodium sulphate, acetate, etc.); the formed threads are subsequently stretched and passed through a dilute acid bath.

F. R. ENNOS.

**Manufacture of hollow artificial silk.** I. G. FARBERIND. A.-G. (B.P. 356,327, 16.6.30. Ger., 14.6.29).—A solution of cellulose acetate in a solvent, *e.g.*,  $COMe_3$ , with at least 20% of a non-solvent (excluding  $EtOH$ ), *e.g.*,  $PhMe$ , is preheated before spinning into an atm. at a temp. below the b.p. of the non-solvent, where it is subjected to accelerated but localised evaporation by suitable means.

F. R. ENNOS.

**Manufacture of filaments, bristles, straw, etc. from organic esters of cellulose.** BRIT. CELANESE, LTD. (B.P. 356,170, 27.3.30. U.S., 27.3.29).—By using org. esters of cellulose containing  $< 2$  ester groups per  $C_6H_{10}O_5$ , *e.g.*, cellulose acetate containing 40–48% (particularly 45–48%) of  $AcOH$ , filaments are obtained which have a moisture regain of about 7%, a safe ironing point 25–30° above normal, and an increased affinity for dyes.

D. J. NORMAN.

**Manufacture of [lustrous] filaments etc. from cellulose derivatives [by wet spinning].** BRIT. CELANESE, LTD. (B.P. 356,343, 20.6.30. U.S., 25.6.29).—A solvent or latent solvent for the cellulose derivative at high temp., which is not readily sol. in the spinning bath, is added to the spinning solution and the filaments are subsequently heated to 90–125°.  $C_2H_4Cl_2$ ,  $CH_2Cl_2$ ,  $PhCOMe$ , triacetin, or  $Bu$  tartrate are suitable when spinning into  $H_2O$ , or diethylene glycol when into kerosene. Alternatively, ordinary spinning solutions may be used, the length of travel or time of contact of the

filaments with the coagulating liquor being so adjusted that the filaments retain a proportion of solvent.

D. J. NORMAN.

**Manufacture of [delustred] artificial threads and filaments and products therefrom.** COURTAULDS, LTD., and C. DIAMOND (B.P. 356,299, 10.6.30).—A solution, in a volatile org. solvent, of an org. derivative of cellulose and a compound of a hydroxy-amine with a fatty acid (hydroxyethyl oleate) is dry-spun and the product afterwards treated with hot soap solution.

F. R. ENNOS.

**Manufacture of (A) articles, (B) products, from cellulose esters or ethers.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 355,298 and 355,330, 21.5.30).—The solution obtained by heating an org. derivative of cellulose with a polyhydric alcohol, *e.g.*, ethylene glycol, is (A) extruded through a suitable nozzle and coagulated either by cooling or by passage through a liquid ( $H_2O$  or polyhydric alcohols), which is maintained above room temp. and contains a solvent or plasticiser for the cellulose derivative and/or salts or sugars; or (B) cooled, freed from part of the alcohol, and crushed to yield a moulding powder.

F. R. ENNOS.

**Production of artificial filaments, ribbons, and like materials.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 355,652, 11.4.30. Addn. to B.P. 327,740; B., 1930, 609).—The liquid ( $H_2O$ , paraffin, glycerin, olive oil, etc.) is applied to the materials during an early stage of drying and while they contain a relatively high proportion of solvent, to give them improved extensibility, tenacity, type and regularity of cross-section, levelness of dyeing, etc.

F. R. ENNOS.

**Production of foils, filaments, etc. from alkaline aqueous cellulose solutions.** WOLFF & Co. KOMM.-GES. AUF AKT., E. CZAPEK, and R. WEINGAND (B.P. 355,424, 25.6.30. Ger., 2.7.29).—Viscose solutions or cuprammonium solutions of cellulose may be spun at higher concentrations than are normally possible if they are heated to a temp. lower than the coagulation temp., *e.g.*, to about 50°, before entering the spinning nozzle or slot. By adjusting the temp. of the spinning solution it is possible to vary the thickness of the film.

D. J. NORMAN.

**Manufacture of ribbons, films, and tapes, or straw-like products of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 354,233, 5.5.30. U.S., 9.5.29. Addn. to B.P. 328,312).—The pliability of the products is improved by adding 2.5–10% of a high-boiling solvent to the solvent liquid used for moistening the filaments. Coalescence is then effected by application of both heat and pressure. Waterproof properties are imparted by passing the ribbons during or after coalescence between smooth rollers coated with, *e.g.*, carnauba wax. Various methods of producing ornamental effects are described.

D. J. NORMAN.

**Production of artificial silk.** GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 354,964, 14.7.30. Ger., 31.7.29).—The thread from washed and dried spinning cakes is directly wound off overhead on to cross-wound bobbins, the thread layers of the cakes having previously been loosened by washing (cf. B.P. 344,279; B., 1931, 437).

H. ROYAL-DAWSON.

**Apparatus for production of artificial filaments and the like.** BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 355,720, 24.2.30).—The spinning jet is provided with an annular depression on the side to which the spinning solution is supplied, and on the other side with a central circular depression into which a reinforcing member engages to support the centre of the jet and prevent its distortion. F. R. ENNOS.

**Manufacture of sheet materials having a basis of cellulose esters or ethers.** H. DREYFUS (B.P. 355,732, 21.5.30).—A homogeneous mixture of the cellulose derivative with a plasticiser is rendered fluid by heat in the substantial absence of volatile solvents, and is drawn off through an adjustable aperture in the heating tank between two similar water-cooled rolls rotated synchronously in opposite directions. The resulting sheets are conditioned by means of gases (moist air) and/or vapours (solvents or non-solvents) and cut to the desired shape. F. R. ENNOS.

**Production of kraft-simulating pulp.** G. A. RICHTER, ASSR. to BROWN CO. (U.S.P. 1,792,510, 17.2.31. Appl., 1.7.25).—"4-4"-pulp as obtained by digesting wood with a liquor containing 4% each of free and combined  $\text{SO}_2$  at 160° and 75–95 lb./sq. in. for 10–11 hr. is digested at 82–100° for 2–7 hr. with 3–12% of NaOH on the wt. of pulp. The resulting pulp has the colour and strength characteristics of kraft pulp.

D. J. NORMAN.

**Production of fibrous thermoplastic sheets.** H. WADE. From BAKELITE CORP. (B.P. 356,260, 5.6.30).—A reactive resin, *e.g.*, of the PhOH-aldehyde type is mixed with 5–45 wt.-% of a plasticiser, preferably a dialkyl ester of an org. acid, *e.g.*, Et or Bu oxalate, tartrate, phthalate, and then rendered substantially infusible and insol. by heat. This product is comminuted and incorporated with paper pulp in a beater. The resulting dried sheets are then superimposed and bonded together by heat and pressure. D. J. NORMAN.

**Manufacture of [duplex] waterproof foil.** NON-INFLAMMABLE FILM CO., LTD., and G. A. STALEY (B.P. 356,146, 28.5.30).—A layer containing nitrocellulose waterproofed by inclusion of a wax (Japan wax), a fat (lanoline), or paraffin wax, together with oil-sol. resins (phthalic anhydride-glycerol resin, PhOH-aldehyde condensation products, or natural resins) is superimposed by casting on a cellulose acetate film. F. R. ENNOS.

**Manufacture of [waterproof] paper.** A. NATHANSON (B.P. 356,277, 6.6.30. Ger., 7.6.29).—Anhydrides, acid chlorides, or other esterification-inducing derivatives of org. acids of high mol. wt., *e.g.*, stearic anhydride, are used as waterproofing agents. They may be applied as emulsions or as a 0.1–5% solution. The treated paper remains waterproof after extraction with volatile solvents. D. J. NORMAN.

**Rendering paper and cardboard impermeable.** PAPETERIES NAVARRE SOC. ANON. (B.P. 357,039, 7.1.31. Fr., 20.10.30).—The material is impregnated with an alkaline caseinate solution containing a high proportion, *e.g.*, 5%, of  $\text{CH}_2\text{O}$  and a volatile substance capable of retarding coagulation, *e.g.*,  $\text{NH}_3$ . After exposure to air to allow evaporation of the  $\text{NH}_3$  and coagulation of the caseinate the material is dried and heated to 120°. An

after-treatment with aq.  $\text{Al}(\text{OAc})_3$  (*d* 1.035) containing about 10% of  $\text{CH}_2\text{O}$  may be given. D. J. NORMAN.

**Preparation of coating material for paper.** C. ARNOLD. From I. F. LAUCKS, INC. (B.P. 356,287, 17.4.30).—Oleaginous seeds, *e.g.*, soya bean, cottonseed, castor seed, tung nut, etc., preferably oil-mill press-cake, are ground and extracted with, *e.g.*, 2 litres of 0.5%  $\text{Ca}(\text{OH})_2$  solution per 100 pts. of meal at 15° for about 12 hr. The separated liquor is then heated to 50° and its  $p_{\text{H}}$  is adjusted to 4 with dil.  $\text{H}_2\text{SO}_4$  to ppt. the protein. This is collected, washed, and dried at about 32°. Other alkalis, *e.g.*, NaOH or  $\text{Na}_2\text{SO}_3$ , may be used in the preliminary extraction, but a subsequent prolonged treatment with dil. aq.  $\text{Ca}(\text{OH})_2$  is necessary to modify the protein so that the dried product is readily dispersible in aq.  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7$ , etc. These proteins are particularly suitable for coating mixtures containing satin-white since they are unaffected by free CaO.

D. J. NORMAN.

**Manufacture of abrasive articles such as sand-paper or the like.** A. E. WHITE. From MINNESOTA MINING & MANUFACTURING CO. (B.P. 356,183, 1.3.30).—A mixture of abrasive particles with a liquid waterproofing adhesive, consisting of a drying oil (linseed or tung oil), rosin, mineral dryers, and thinners, is applied to a flexible backing and the whole dried at 55–65°. The abrasive surface is subsequently treated with a sizing compound, of similar composition to the adhesive but with a larger proportion of thinners, which is also applied to the back of the sheet, before, during, or after application of the abrasive mixture. [Stat. ref.] F. R. ENNOS.

**Manufacture of tracing cloth.** R. SCHWICKERT A.-G., and O. HUBER (B.P. 357,068, 2.3.31. Ger., 11.7.30).—Uniform transparency is obtained by using as the base material fabrics made from squirted or drawn filaments, *e.g.*, real or artificial silk. D. J. NORMAN.

**Kaolin [for paper].**—See VIII. **Paper strips for films.**—See XXI. **Nitrocellulose.**—See XXII. **Waste pulp liquors.**—See XXIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Dyeing of hosiery composed of mixed fibres.** H. MUDFORD (J. Soc. Dyers and Col., 1931, 47, 222–225).—Real silk-cotton hose are degummed by boiling for 1 hr. in a 0.8% soap solution, rinsed, and dyed simultaneously with direct and neutral dyeing acid colours; degumming and dyeing may also be effected in the same bath. The difficulty of the formation of "green streaks" on the fibre when most neutral-dyeing acid reds are employed is not encountered when Coomassie Red GS, Neutral Silk Red GDL, Cloth Red 3G extra, Fast Red G for silk, and Acid Anthracene Red 3BL are used. The difficulty of obtaining uniform delicate tints on viscose silk-mercerised cotton is reduced when dyeing is effected in hard water or in a dye liquor containing  $\text{Na}_2\text{SO}_4$  in excess; suitable dyes are Chlorazol Orange Brown XS, Fast Red FGS, and Black ES. The presence of a real silk thread necessitates a previous degumming in a 0.2% soap solution and the use in dyeing of neutral dyeing acid dyes, otherwise a mottled thread results. Full blacks are produced with Chlorazol Blacks ES and LFS



after-treated with  $\text{CH}_2\text{O}$ . The chief difficulty in dyeing viscose silk-unmercerised cotton is that in deep shades the viscose dyes deeper than the cotton; this tendency is minimised by dyeing at low temp. and using the min. amount of  $\text{Na}_2\text{SO}_4$  and suitable dyes (*e.g.*, Chlorazol Orange POS, Fast Red KS, and Black BKS for compound shades; Chlorazol Brown LFS and Icy Navy B for gunmetal shades, the last-named dye having a greater affinity for the cotton; Chlorazol Black SDS developed with *m*-phenylenediamine and shaded with Chlorazol Fast Green PLS for black). Icy Blue 2RS has a much greater affinity for cotton than viscose silk. In Celta (viscose) silk-mercerised cotton hose the Celta dyes to a much paler shade than the cotton with most direct dyes; Chlorazol Drab RHS, Brown GMS, and Black FFS must be used. Viscose silk-wool is scoured at 50° or below in a bath containing 2–3 pts. of soap and 1–2 pts. of aq.  $\text{NH}_3$  per 1000 pts. and dyed with direct and neutral-dyeing acid colours; direct colours are to be preferred since they give better penetration. Suitable dyes are Chlorazol Browns LFS and PBS, Orange Brown XS, Fast Red FGS, and Fast Black BKS; Chlorazol Blacks ES and LFS (for full blacks). Wool-real silk is degummed in a soap liquor at about 80°, 3% of Perminol N being added to reduce felting of the wool, and then dyed with direct colours. The dyeing of Visylka, Celfect, and wool-viscose silk-cellulose acetate silk hose is also outlined.

A. J. HALL.

**Study of kiering processes using a potentiometer.** P. P. VIKTOROV and B. GOLDBERG (Textilber., 1931, 12, 638–641).—Theoretically, in the kiering of raw cotton, the amounts of NaOH consumed in saponifying natural fats and waxes, in combining with the decomp. products of the pectin impurities, and in decomposing proteins are in the ratio 41 : 700 : 850, and in these reactions only 50% of the usual amount of NaOH present is utilised. Large-scale trials with a partly purified cotton showed that only 20% of the NaOH initially present was utilised in removing the impurities, whilst 60% was absorbed as in mercerising processes; the remainder appeared to be ineffective. In these determinations it was found advantageous to use potentiometric methods since the kier liquors were highly coloured. Purification of the cotton during kiering, as measured by the increasing reducing power of the kier liquor, is complete after 4 hr., but it is considered advisable to continue kiering for the usual 8 hr. The absorption of alkali from the kier liquor is almost complete within the first hr.

A. J. HALL.

**Effect of valency of electrolytes in direct dyeing.** P. P. VIKTOROV (Izvest. Tekstil. Prom., 1930, 9, No. 6—7, 73–76).—The effect of an electrolyte is special to each dye. By proper proportioning of electrolytes having multivalent cations they can be used advantageously.

CHEMICAL ABSTRACTS.

**Use of sodium silicate for the cottonisation and cleaning of fibres.** P. P. VIKTOROV (Izvest. Tekstil. Prom., 1930, 9, No. 6—7, 80–81).—The cottonisation of kender or flax fibres by means of a bath of NaOH and Na silicate is described. The following baths are specified: (1) NaOH 10 g., 38% Na silicate 10 g., per litre; (2) NaOH 30 g., 38% Na silicate 30 g.,  $\text{NaHSO}_3$  (*d* 1.33) 10 g.

CHEMICAL ABSTRACTS.

**Removal of stains from hosiery goods.** H. L. LONG (J. Soc. Dyers and Col., 1931, 47, 287–289).—Stains are classified into five groups: tannin basis (tea etc.), albumin basis (blood, egg, etc.), chemical (inks, Fe mould), oily (oils, paints), and mildew, whilst the reagent to be used depends on the nature of the stain and the material. Removal should first be attempted with  $\text{H}_2\text{O}$ , then with solvents, acids, and finally with bleaching agents. Tannin or albumin stains frequently yield to washing with  $\text{H}_2\text{O}$  below 100°, or with aq. NaCl. Chemical stains generally require bleaching, and if a metal is present acid treatment is necessary. For Fe stains  $(\text{CO}_2\text{H})_2$ ,  $\text{Na}_2\text{S}_2\text{O}_4$ , or, preferably, HF should be used. Cu stains should be treated with KCN prior to bleaching. Oil or grease stains are emulsified by scouring with soap and alkali or removed by a solvent. Oleic acid is superior to the former as an emulsifier. Mildew is removed with  $\text{H}_2\text{O}_2$  or  $\text{KMnO}_4$  followed by  $(\text{CO}_2\text{H})_2$ . B. P. RIDGE.

Textile soaps.—See XII.

## PATENTS.

**Dyeing of esters or ethers of cellulose or its transformation products.** I. G. FARBERIND. A.-G. (B.P. 352,099, 9.4.30. Addn. to B.P. 304,739; B., 1930, 506).—Acetate silk etc. is dyed in yellow shades with the sulphamic acid of a 4-amino-1:8-naphthalic imide or substituted imide, *e.g.*, the ethylimide, phenylimide, 4-*m*-xylylimide.

C. HOLLINS.

**Impregnated and coated fabric.** H. WADE. From BAKELITE CORP. (B.P. 356,818, 14.6.30).—In a flexible article embodying rubber-coated fabric, *e.g.*, a tyre or fire hose, the fabric is impregnated first with a flexible resinoid (*e.g.*, a resinoid of the phenol-oil-methylene type) or with a reactive phenol-methylene resin capable of conversion into the former; the resinoid-impregnated material is then coated with rubber. The strength and wear-resisting qualities of the fabric are markedly increased.

D. F. TWISS.

**Manufacture of fireproof porous material [fabrics and textiles].** H. HOPKINSON, Assr. to BRUNSENE CO. OF MASS. (U.S.P. 1,797,865, 24.3.31. Appl., 11.10.28).—Canvas etc. is impregnated successively with solutions containing a Pb salt [ $\text{Pb}(\text{OAc})_2$ ] and a sol. chloride ( $\text{AlCl}_3$ ); if desired, it may then be immersed in a solution containing a sol. borate, phosphate, carbonate, tungstate, or fluoride.

L. A. COLES.

**Making artificial silk yarn of diminished lustre.** N. V. HOLLANDSCHE KUNSTZIJDE IND. (B.P. 356,749, 6.6.30. Holl., 3.2.30).—Delustred viscose silk is spun from viscose solutions to which not more than 10% (calc. on the cellulose) of  $\text{H}_2\text{O}$ -insol. terpenes or terpene alcohols (preferably in the form of an aq. emulsion prepared by the aid of Turkey-red oil) has been added either during or directly after dissolving the cellulose xanthate (*cf.* B.P. 183,476; B., 1924, 51).

A. J. HALL.

**Treatment of textile materials.** BRIT. CELANESE, LTD., S. M. FULTON, and G. C. TYCE (B.P. 355,794, 24.5.30).—After the application of a treating liquid, *e.g.*, size, the yarn is wound in the form of a hank, and dried during the winding operation by a current of

heated air concentrated thereon by means of a surrounding casing.  
F. R. ENNOS.

**Rendering textiles water-repellant.** A. NATHAN-SOHN (B.P. 355,256, 16.4.30. Ger., 19.4.29).—Textiles such as cotton, artificial silk, and wool are partly esterified with a higher fatty acid, *e.g.*, stearic or palmitic acid, under mild conditions such that no caustic alkali and little or no condensing agent is required (cf. B.P. 313,616; B., 1930, 815).  
A. J. HALL.

**Coated textile fabric.** J. J. CLIFFORD, Assf. to STEADFAST RUBBER CO., INC. (U.S.P. 1,792,277, 10.2.31. Appl., 31.1.27).—A fibrous fabric coated with a rubber compound is rendered non-adherent, *e.g.*, by a suitable varnish, and is then embossed with the desired design with the aid of heat. An oil-free pasty mixture of pigment, nitrocellulose, and a solvent with affinity for both rubber and nitrocellulose is wiped into the depressions of the embossed surface, and the completed fabric is vulcanised.  
D. F. TWISS.

**[Device for uniformly] varnishing webs of cellulose hydrate.** KALLE & Co. A.-G. (B.P. 357,436, 8.12.30. Ger., 27.12.29).

**Alcohols.**—See III. Cellulose derivatives.—See V. Fumigant.—See XVI.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Effect of adsorbent powders on the solubility of phosphoric acid in ammonium citrate.** R. DUBRISAY (Ann. Falsif., 1931, 24, 400—402).—The effect of adding adsorbent powders to equilibrium mixtures of  $\text{CaHPO}_4$  or superphosphate and  $\text{NH}_4$  citrate has been studied. Paraffin black, and to a smaller extent talc or kaolin, adsorbs  $\text{NH}_3$  and, by lowering the  $[\text{H}^+]$ , increases the amount of sol.  $\text{P}_2\text{O}_5$ . Kieselguhr appears to be without action. When  $\text{HCl}$  is substituted for  $\text{NH}_4$  citrate the action of adsorbents is less marked, but, owing to the fixation of  $\text{HCl}$ , they increase the  $[\text{H}^+]$  and decrease the amount of  $\text{P}_2\text{O}_5$  dissolved.  
T. McLACHLAN.

**Solubility of phosphates in neutral ammonium citrate solution.** K. D. JACOB, K. C. BEESON, L. F. RADER, JUN., and W. H. ROSS (J. Assoc. Off. Agric. Chem., 1931, 14, 263—283).—When the wt. of sample used for the determination of available  $\text{P}_2\text{O}_5$  in  $\text{H}_2\text{O}$ -insol. phosphates by the official method ("Methods of Analysis," A.O.A.C., 1925, p. 4) was decreased by 0.5-g. steps from 2.0 g. to 0.5 g. there was a progressive decrease in the citrate-insol.  $\text{P}_2\text{O}_5$  in  $\text{CaHPO}_4$ ,  $\text{Ca}_3(\text{PO}_4)_2$ ,  $\text{Mg}_3(\text{PO}_4)_2$ , heavily ammoniated superphosphates, raw, steamed, and naphtha-extracted bone, basic slag, and calcined phosphate.  $\text{MgHPO}_4$  and  $\text{MgNH}_4\text{PO}_4$  were completely sol. when 2.0 g. were used,  $\text{CaHPO}_4$  and  $\text{Mg}_3(\text{PO}_4)_2$  when 1.0 g. was used, whilst  $\text{Ca}_2\text{P}_2\text{O}_7$  and  $\text{Mg}_2\text{P}_2\text{O}_7$  were only slightly sol. In the cases of "C.P." Fe and Al phosphates significant decreases in the insol. material (which varied from 1.0% to 55.5% of the total  $\text{H}_3\text{PO}_4$ ) were not obtained. Impure Fe and Al phosphates precipitated from crude  $\text{H}_3\text{PO}_4$  were almost completely sol. Acidulated phosphates untreated with aq.  $\text{NH}_3$ , and commercial phosphate rock showed no

significant decrease. The solubility of the  $\text{P}_2\text{O}_5$  in uncalcined bone approximated closely to that of the  $\text{P}_2\text{O}_5$  in  $\text{Ca}_3(\text{PO}_4)_2$ , being thrice as sol. as that in  $3\text{Ca}_3(\text{PO}_4)_2 \cdot \text{Ca}(\text{OH})_2$ . When 2.0 g. were used, 65—75% of the  $\text{P}_2\text{O}_5$  in  $\text{Ca}_3(\text{PO}_4)_2$  was insol., whereas when 0.5 g. was used only 18—36% was insol. 0.5—1.0 g. of high-grade basic slag treated with 100 c.c. of neutral  $\text{NH}_4$  citrate solution showed the same solubility as that found by the official method, using 2% citric acid solution.  
W. J. BOYD.

**Alkali content of chalk.** WERNER (Farben-Chem., 1931, 2, 402—403).—Extraction of numerous natural chalks with hot  $\text{H}_2\text{O}$  and titration of the filtrate with  $\text{HCl}$  invariably yields an alkali val. corresponding to a max. of 0.03 mg. of  $\text{KOH}$  per g. of chalk. As it is improbable that this alkalinity is due to the presence of free  $\text{CaO}$ , it is suggested that it really represents the solubility of  $\text{CaCO}_3$  in  $\text{H}_2\text{O}$ .  
A. R. POWELL.

**Electrometric analysis of solutions of hypochlorites.** M. ABRIBAT (Bull. Soc. chim., 1931, [iv], 49, 1119—1138).—A Pt electrode gives reproducible potentials when immersed in acidified hypochlorite solutions. Commercial samples can be titrated electrometrically with dil.  $\text{HCl}$ , and the free alkali, carbonate, hypochlorite, and free  $\text{HOCl}$  ascertained from sharp inflexions in the curve.  
C. W. DAVIES.

**Electrolytic production of potassium ferri-cyanide.** V. P. ILINSKI and N. P. LAPIN (J. Appl. Chem., Russia, 1930, 3, 981—998).—A solution of  $\text{K}_4\text{Fe}(\text{CN})_6$  saturated at 50° is electrolysed at 55° (anode c.d. 5, cathode 20—50 amp.), using an Fe cathode and Ni anode with (cathode c.d. 150—200 amp. without) asbestos diaphragm.  $\text{K}_2\text{SO}_4$ , if present, is removed. After crystallisation the process is repeated, the solution being evaporated to dryness after the fourth cycle. Solubility diagrams of  $\text{K}_4\text{Fe}(\text{CN})_6$ — $\text{K}_2\text{SO}_4$  and of  $\text{K}_4\text{Fe}(\text{CN})_6$ — $\text{K}_3\text{Fe}(\text{CN})_6$  mixtures are given.

### CHEMICAL ABSTRACTS.

**Production of copper sulphate from Ural copper ores and tailings.** I. G. STSCHERBAKOV and M. M. NARKEVICII (Tzvet. Met., 1930, 5, 1159—1167).—The yield with  $\text{NH}_3$  solution generally increases with the Cu content (0.2—4.5%); addition of  $(\text{NH}_4)_2\text{CO}_3$  or  $\text{NH}_4\text{Cl}$  is advantageous, but the concentration (1—6%) of the  $\text{NH}_3$  has little influence. CHEMICAL ABSTRACTS.

**Determination of lead and copper in Bordeaux-lead arsenate mixtures.** J. C. BUBB (J. Assoc. Off. Agric. Chem., 1931, 14, 260—262).—The sample (1.0 g.) is heated with 50 c.c. of 33%  $\text{AcOH}$  on the steam-bath for 5—10 min. 0.5 g. of  $\text{Cu}_3(\text{AsO}_4)_2$  is added and then conc.  $\text{HNO}_3$  dropwise with stirring until any blue coloration clears up and the  $\text{PbHAsO}_4$  is white. After being heated for a few min. the solution is cooled, the  $\text{HNO}_3$  present nearly neutralised with conc. aq.  $\text{NH}_3$ , and left at room temp. for 20 min. The mixture is filtered, the  $\text{PbHAsO}_4$  washed free from Cu with hot  $\text{H}_2\text{O}$  and dissolved in 25 c.c. of  $\text{HNO}_3$ , and the Pb determined by the official method ("Methods of Analysis," A.O.A.C., 1925, p. 58). The filtrate is neutralised by conc. aq.  $\text{NH}_3$  and the Cu determined iodimetrically (1 c.c.  $\equiv$  0.005 g. of Cu). The method is more rapid than the official method. W. J. BOYD.

**Storage of carbon monoxide. A correction.** O. H. WAGNER (Brennstoff Chem., 1931, 12, 352; cf. B., 1931, 438).—In a calculation in the previous paper too high a value was assumed for the bulk density of  $\text{Cu}_2\text{Cl}_2$ ; it follows that the vol. of CO which can be stored in a cylinder packed with  $\text{Cu}_2\text{Cl}_2$  is only 2.3 and not 4 times that which can be compressed into the empty cylinder under the same pressure (150 atm.). A. B. MANNING.

**Obtaining oxides of nitrogen from ammonia *via* nitrate.** D. A. EPSTEIN (Udobr. Urozhai, 1930, 2, 749—751).— $\text{NH}_3$  (20%) and air together were passed over soda-lime containing Co and Ni; the product contained 27%  $\text{NaNO}_3$ . CHEMICAL ABSTRACTS.

**Study of Travers' method for determination of fluorine with reference to insecticides.** C. M. SMITH, E. H. HAMILTON, and J. J. T. GRAHAM (J. Assoc. Off. Agric. Chem., 1931, 14, 253—260).—Travers' method (B., 1921, 873 A, 904 A) slightly modified gives low results in the presence of Al and Fe, and is useless in the presence of borax. Attempts to expel the B as  $\text{MeBO}_3$  by evaporation with MeOH and conc. HCl were only partly successful. NaF containing > 20% CaO gives seriously low results. A modified method is described which gives accurate results in presence of CaO. To 0.5 g. of sample 20—25 c.c. of water, 0.3 g. of finely-divided precipitated  $\text{SiO}_2$ , and a few drops of methyl-orange are added. The mixture is carefully acidified with conc. HCl, 2 c.c. in excess being added. It is boiled for 1 min., cooled, and 4 g. of solid KCl are dissolved in it. Next 25 c.c. of 95% EtOH are added, and after keeping for 1 hr. with frequent stirring it is filtered through a Gooch crucible containing a disc of filter paper covered by a pad of asbestos. The ppt. is washed with alcoholic KCl (made by dissolving 60 g. of KCl in 400 c.c. of  $\text{H}_2\text{O}$ , adding 400 c.c. of 95% EtOH, and neutralising to phenolphthalein) until the washings are neutral to phenolphthalein. The ppt. is then titrated with 0.2N-NaOH free from carbonate, the titration being completed with the fluoride solution actively boiling (1 c.c. of 0.2 N-NaOH  $\equiv$  0.005700 g. F). The presence of S, starch, flour, tobacco, talc, *p*-dichlorobenzene,  $\text{C}_{10}\text{H}_8$ , cresols, coal-tar neutral oils, Paris green, or  $\text{Ca}_3(\text{AsO}_4)_2$  does not interfere. The presence of pyrethrum or  $\text{Pb}_3(\text{AsO}_4)_2$  leads to high results, that of  $\text{AlCl}_3$ , K alum, kaolin, or diatomaceous earth to low results, and of  $\text{FeCl}_3$  to slightly low results. W. J. BOYD.

**Determination of iodine in iodised common salt.** A. GRONOVER and E. WÖHLICH (Z. Unters. Lebensm., 1931, 61, 306—314).—For a qual. test of the uniformity of distribution of I, a thin layer of the salt is spotted with a starch- $\text{NaNO}_2$  mixture. For the quant. determination a modification of Fellenberg's method (B., 1923, 1068 A; 1931, 391) is recommended. To 100 c.c. of filtered salt solution (100 g. in 500 c.c.) are added 1 c.c. of N-HCl and 1 c.c. of saturated Br water, the mixture being boiled gently for 10 min. with replacement of the evaporated  $\text{H}_2\text{O}$ . After cooling to  $10^\circ$ , 1.5 c.c. of 85%  $\text{H}_3\text{PO}_4$  and 0.2 g. of KI are added and the mixture is kept in the dark for 3 min., when it is titrated with 0.004N- $\text{Na}_2\text{S}_2\text{O}_3$ , 1 c.c. of starch being added immediately before the end-point. Fe and Mn salts cause an apparent increase in the I content, which may be neglected at the concentrations usually encountered, but

when appreciable amounts are present the effects may be eliminated by adding 1—2 drops of 10%  $(\text{CO}_2\text{H})_2$  solution. H. J. DOWDEN.

**Removing NO from coke-oven gas.**—See II. **Calcined  $\text{MgO}$ .**—See IX.  **$\text{SrSO}_4$  as rubber filler.**—See XIV. **Cr-tanning liquors.**—See XV. **Na aluminate in water treatment.**—See XXIII.

#### PATENTS.

**Preparation of phosphoric anhydride and phosphoric acid.** METALLGES. A.-G. (B.P. 357,026, 3.12.30. Ger., 6.1.30).— $\text{P}_2\text{O}_5$  is prepared by introducing powdered P through nozzles into a combustion space and causing the P to impinge on to a rapidly rotating distributing disc. Air enriched with  $\text{O}_2$  (to increase the concentration of  $\text{P}_2\text{O}_5$  in the waste gases) is simultaneously admitted as combustion gas. The  $\text{P}_2\text{O}_5$  may afterwards be converted in known manner with  $\text{H}_2\text{O}$  into  $\text{H}_3\text{PO}_4$ . F. YEATES.

**Production of concentrated phosphoric acid.** KUNSTDÜNGER-PATENT-VERWERTUNGS A.-G. (B.P. 357,041, 9.1.31. Swed., 29.1.30).—During the concentration of  $\text{H}_3\text{PO}_4$  produced from phosphate rock with the formation of  $\text{CaSO}_4$ , the residual unprecipitated  $\text{CaSO}_4$  which crystallises out during evaporation is continuously removed and returned to the leaching plant, thus preventing the formation of hard crusts on the heating surfaces of the apparatus used. F. YEATES.

**Production of soda and potassium hydroxide.** CHEMIEVERFAHREN GES.M.B.H. (B.P. 356,821, 4.6.30. Ger., 13.7.29. Cf. B.P. 343,691 and 354,451; B., 1931, 488, 924).—The combined liquors in the process described in B.P. 354,451 are treated for the removal of  $\text{NH}_3$  and, by cooling, of part of the  $\text{NH}_4\text{Cl}$  before use in the  $\text{NH}_3$ -soda process, and  $\text{SrO}$ , obtained by calcining the  $\text{SrCO}_3$ , is used for treating the  $\text{K}_2\text{SO}_4$ , so that KOH is obtained instead of  $\text{K}_2\text{CO}_3$ . L. A. COLES.

**Manufacture of ammonia by the contact process.** A. MENTZEL (B.P. 356,645, 16.2.31).—Pure  $\text{H}_2$  is produced electrolytically at such a high pressure that, after admixture with  $\text{N}_2$  at atm. pressure, the mixture will be at sufficient pressure to effect combination after passing through a series of contact stages. W. J. WRIGHT.

**Manufacture of ammonium sulphate.** H. J. HODSMAN and A. TAYLOR (B.P. 356,283, 30.5.30).—Aq.  $(\text{NH}_4)_2\text{SO}_3$  solution, produced by combining  $\text{NH}_3$  or  $\text{NH}_3$  liquor with  $\text{SO}_2$ , is passed down a tower in counter-current to hot gases containing free  $\text{O}_2$ . A heavy-metal compound, preferably a ferrous salt, may be added to the liquor as a catalyst, the liquor being kept at a suitable degree of alkalinity or acidity. Oxidation of the solution is completed electrolytically, electrolysis being arrested before any thiocyanates or thiosulphates become oxidised. W. J. WRIGHT.

**Manufacture of ammonium sulphate.** H. J. HODSMAN and A. TAYLOR (B.P. 356,757, 30.5.30).—Aq.  $(\text{NH}_4)_2\text{SO}_3$  solution is oxidised electrolytically in presence of a sol. chloride (preferably  $\text{NH}_4\text{Cl}$ ). The  $(\text{NH}_4)_2\text{SO}_3$  solution is prepared as in B.P. 356,283 (cf. preceding abstract), and may be partly oxidised before electrolysis by passing gases containing free  $\text{O}_2$ , such as products of combustion; catalysts ( $\text{FeSO}_4$ ) may be used. F. YEATES.

**Manufacture of ammonium sulphate.** F. A. F. PALLEMAERTS, and UNION CHIM. BELGE SOC. ANON. (B.P. 356,580, 27.11.30).— $\text{CaSO}_4$  and  $(\text{NH}_4)_2\text{CO}_3$  in the requisite proportions are mixed in a wet-grinding mill, the product being either transferred to reaction vessels for completion of the reaction and subsequently filtered, or filtered direct after leaving the mill. W. J. WRIGHT.

**Production of ammonium sulphate [and sulphur].** H. KOPPERS A.-G., Assecs. of F. KRUPP A.-G. (B.P. 356,739, 6.5.30. Ger., 21.5.29).— $(\text{NH}_4)_2\text{SO}_4$  and S are produced from suitable mixtures of  $\text{NH}_4$  salts ("neutral thionate salts" and "acid thionate salts") by heating aq. solutions of such salts in vessels susceptible to attack by  $\text{H}_2\text{SO}_4$  (e.g., Ni-Cr-steel vessels) and maintaining in the resultant products to the end of the operation a small quantity of the initial unconverted materials. (Cf. B.P. 309,565; B., 1930, 323.) F. YEATES.

**Production of mixed [fertiliser] salts containing ammoniacal and nitrate nitrogen jointly.** RUHR-CHEMIE A.-G. (B.P. 356,759, 3.3.30. Ger., 2.3.29).—Hydrated or anhyd. solutions of  $\text{NH}_4\text{NO}_3$  in  $\text{NH}_3$  (Divers' liquid) are treated with hydrated or anhyd. acids (carbonic acid being excluded) or acid salts until the  $\text{NH}_3$  present is neutralised. The process may be so controlled that a dry or almost dry finished product is released. F. YEATES.

**Removal of iron and iron compounds from substances containing iron.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 356,523, 6.10.30).—The Fe is extracted in the form of chloride by means of an org. solvent (e.g., ethers, aldehydes, ketones, or org. halogen compounds) in which the remainder of the substance is insol., conversion into chloride being effected, if necessary, by introducing gaseous HCl with the solvent or by treatment prior to extraction. W. J. WRIGHT.

**Evaporation of liquids.**—See I. Catalytic oxidations. Separation of CO from gases. S from gases. Gas-purification products.—See II. Complex salts.—See III. Steel resistant to chemicals.—See X. Fertiliser. Fungicidal solution.—See XVI. Waste pulp liquors.—See XXIII.

## VIII.—GLASS; CERAMICS.

**Properties of glass-tank refractories.** H. SCHILLING (Chem.-Ztg., 1931, 55, 691).—Such refractories should react neutral towards the glass and should be as dense and as free from pores as possible. This is obtained by using a material containing as much  $\text{SiO}_2$  as the glass, the remainder being all or nearly all  $\text{Al}_2\text{O}_3$ ; the material should be as fine-grained as possible and fired at a high temp. after shaping. A. R. POWELL.

**Scientific basis of glass melting.** W. E. S. TURNER (J. Soc. Glass Tech., 1930, 14, 368—394 r).—A comprehensive review of previous work dealing with (a) physical changes, thermal changes, m.p., mutual solubility of materials, volatilisation; (b) chemical changes, decomp. and dissociation processes, and interaction during melting. The author favours the isothermal heating of reaction mixtures in air rather than in a current of  $\text{CO}_2$ . A study of the rate of heating, as practised by the Tammann school, might be useful,

but left much to be desired in sensitiveness and needed a very finely-divided mixture, which then did not compare with the ordinary grade of glass-making material.

M. PARKIN.

**Effect on properties of soda-lime-silica glass of repeated melting in platinum.** The X-ray pattern. W. E. S. TURNER (J. Soc. Glass Tech., 1930, 14, 351—353 r).—X-Ray examination of four of the glasses of one of the series (III) dealt with in a previous paper by Turner and co-workers (B., 1930, 460) failed to show any difference of pattern, thus confirming, since the specimens covered the range of remelting treatment, the findings of Turner etc. that, provided no change in chemical composition occurs, remelting causes no change in physical properties.

M. PARKIN.

**Thermal endurance of glass.** W. J. A. WARREN (J. Soc. Glass Tech., 1930, 14, 313—329 r).—The expression proposed by Gould and Hampton (B., 1930, 989) is modified by the present author to  $B = \frac{0.7}{l}$ , where  $l$  is the thickness in the bottom of the beaker along the course of fracture, and  $\alpha$  is a const. (0.36—0.4 for beakers 1.4 mm. thick; 0.5 for those 1.1 thick), and  $B$  and  $\theta$  are as before.

M. PARKIN.

**Problems of safety glass manufacture.** T. PESCH (Chem.-Ztg., 1931, 55, 730—731).—The use of a synthetic resin for the intermediate layer gives brilliance, transparency, and good adhesion, but its resistance lies within narrow limits of temp. Celluloid is relatively unaffected by low temp., but becomes discoloured by heat or sun rays. A satisfactory material has been produced in Germany from cellulose acetate. The problems associated with its use, e.g., the selection of suitable softening agents and the avoidance of shrinkage, are discussed. A method of testing safety glass consists in placing a circular disc of the glass on a conical tinned container filled with  $\text{H}_2\text{O}$  and dropping a steel ball on the centre of the disc from a given height. A stereophotographic method has been developed for determining liability to shrinkage.

W. J. WRIGHT.

**Some effects of thermal shock in causing crazing of glazed ceramic ware.** H. G. SCHURECHT and D. H. FULLER (J. Amer. Ceram. Soc., 1931, 14, 565—571).—Thermal-shock crazing is caused in some cases by the development of craze-like cracks in the body itself, and bodies resistant to thermal shock are found to be less liable to craze when glazed. Glazed ware, in general, is more resistant to thermal shock when the glaze is under high compression rather than low compression or tension. High- $\text{SiO}_2$  bodies are more resistant to thermal shock than low- $\text{SiO}_2$  bodies, but there is no connexion between crazing and adsorption of the body or moisture crazing. The tendency to craze increases with the degree of lustre of the glaze. A suggested accelerated thermal-shock test is to cool repeatedly from  $120^\circ$  in air to  $\text{H}_2\text{O}$  at room temp.

J. A. SUGDEN.

**Use of lepidolite in zincless glazes.** H. A. DAVIS and R. L. LUEDERS (J. Amer. Ceram. Soc., 1931, 14, 562—564).—Addition of lepidolite improves the resistance to crazing and the surface texture without destroying the colour. Such zincless glazes may be produced well within the range of safety from moisture crazing.

J. A. SUGDEN.

**Coloured jewellery enamels for art-school use.** K. E. SMITH (J. Amer. Ceram. Soc., 1931, 14, 588—589).—Successful enamel compositions are given and the enamelling technique is briefly described.

J. A. SUGDEN.

**Origin of the colour produced on red bodies by zinc vapour.** A. MCK. GREAVES-WALKER (J. Amer. Ceram. Soc., 1931, 14, 578—582).—Petrographic examination showed that the green colour given to red burning bodies by Zn vapour is due, not to the presence of a green mineral, but to a yellow Zn glaze with a bluish (reduced) zone beneath it. If the bluish zone is re-oxidised during cooling, the green colour is lost. To obtain the best colours the coal used should not produce considerable fine ash, only pig or moss Zn should be used (scrap Zn gives yellows), and strongly reducing conditions should be maintained during cooling.

J. A. SUGDEN.

**Suggested method for determining plasticity of clays, and some applications.** E. J. C. BOWMAKER (J. Soc. Glass Tech., 1930, 14, 330—348 r).—A modified Pfefferkorn apparatus (Sprechsaal, 1924, 57, 297; 1925, 58, 183) is described, and by its use it is found that by plotting the amount of  $H_2O$  (g. per 100 g. of dry clay) against the deformed height of the cylinder, substantially straight-line graphs were obtained, from which it appeared that the plasticity  $P = R(R - r)$ , where  $R$  and  $r$  are the amounts of  $H_2O$  corresponding to zero and complete deformation, respectively. Plotting  $P$  against the % clay in the mixtures studied gave smooth curves from which it was possible to determine the point at which an increase of grog content caused an appreciable decrease in plasticity. So long as the proportion of clay is sufficient to bind the mixture, coarse grog influences plasticity less than fine. In comparing various clays, results from mixtures made with clays sieved to 100-mesh did not tally with the accepted views on plasticity, whereas those using the fraction lying between 20- and 40-mesh did.

M. PARKIN.

#### PATENTS.

**Lehrs for annealing glassware.** BRIT. HARTFORD-FAIRMONT SYND., LTD., Assees. of H. A. WADMAN (B.P. 356,920 and 357,075, [A, B] 25.8.30. U.S., 29.8.29).—In [A] a heat- or ventilation-control device sensitive to draughts of the order of 10 ft./min. is described. Though described for convenience as a thermostat, its action is independent of temp. It comprises a vertical flame which, in the complete absence of horizontal currents, will strike the centre point of a double thermostat; a draught either way will deflect the flame to one or the other arm of the thermostat and initiate appropriate control. In (B) a system of ventilation of a lehr is described.

B. M. VENABLES.

**Apparatus for making glass.** H. F. HITNER, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,799,371, 7.4.31. Appl., 23.5.27).—The ends of an elongated tank terminate in transverse bays, at the ends of which Fe-Cr plates serve as electrodes. A baffle of refractory material extends vertically through the glass in the tank near to the electrodes. Three-phase current is employed for melting and fining the batch.

C. A. KING.

**Apparatus for treating [firing] ceramic ware.** F. M. HARTFORD, Assr. to HARROP CERAMIC SERVICE Co. (U.S.P. 1,799,980, 7.4.31. Appl., 10.4.30).—The conveyor units in a tunnel kiln consist of flat plates of refractory material which are caused to slide along the floor. The floor has channels or flues so that the heating gases may be proportioned at will between direct heating of the articles to be fired and indirect heating by conduction through the conveyor plates on which the ware is stacked.

C. A. KING.

**Decoration of ceramic articles, e.g., glass, glazed porcelain, stoneware, and the like.** DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 308,226, 19.3.29. Ger., 19.3.28).—The articles are coated first with the colour or lustre in the usual way, then with solutions containing glycerol, chloral hydrate, or ethylene glycol, together with colloids, e.g., gum arabic, and, if desired, colouring materials. The top layer contracts on drying and heating and the lower layer is thereby caused to produce a network effect on the ware. A. R. POWELL.

**Production of coloured patterns on metallic articles.** DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 335,789, 4.11.29. Ger., 9.11.28. Addn. to B.P. 308,226; preceding).—The surface of the article is covered with a ceramic enamel or lustre and, when this has dried, a covering layer is applied by painting or spraying with a solution containing glycerol 14, gum arabic 12, sugar 5, alcohol 20, and water 49%. The articles are then heated slowly to the temperature at which the enamel fuses; during this heating the gum layer contracts and produces in the colour layer a network or veining effect which is fixed when the enamel melts.

A. R. POWELL.

**Altering the physical properties of finely-divided silicious mineral matter [kaolin].** R. ILLNER (B.P. 338,726, 18.11.29. Austr., 13.12.28).—Kaolin suspensions, e.g., from the elutriation of china clay, are coagulated with  $Ca(OH)_2$  or with  $Al_2(SO_4)_3$  and the coagulum is neutralised with  $H_2SO_4$ , bleached with  $Na_2S_2O_4$ , and made slightly acid with  $H_2SO_4$ , so that the particles of kaolin are coated with an adsorbed film of  $CaSO_4$  or  $Al_2O_3$ . Blue colouring matter, e.g., ultramarine or a dye, may be incorporated to produce a whiter product suitable for the paper or textile industries. A. R. POWELL.

**Improving the physical properties of kaolin.** R. ILLNER (B.P. 338,730, 18.11.29. Austr., 15.6.29. Addn. to B.P. 338,726; preceding).—A suspension of kaolin in dilute sulphuric acid is successively treated with sodium hyposulphite, blown with air, and nearly neutralised with calcium hydroxide; a catalyst, e.g., sodium nitrate, may be added before the blowing, and a vat dye, e.g., indanthrene-blue, before the blowing or after the addition of the calcium hydroxide.

L. A. COLES.

**Manufacture of insulating bodies for sparking plugs.** SIEMENS & HALSKE A.-G. (B.P. 356,983, 24.10.30. Ger., 13.3.30).—A mixture of finely-ground  $Al_2O_3$  with an acid, e.g., HCl, is moulded with the insertion of metal spindles in divided plaster moulds; when the articles have partly set, they are removed, dried, and fired to sintering point, i.e., at least 1600°.

L. A. COLES.

**Refractory [bricks etc.] and their manufacture.** S. S. COLE, Assr. to KOPPERS. Co. (U.S.P. 1,798,972, 31.3.31. Appl., 29.6.27).—Refractory bricks, *e.g.*, of  $\text{SiO}_2$  or magnesite, possess greater strength and lower porosity if a sol. Fe compound together with an org. substance which prevents precipitation of the Fe in alkaline solution be added to the batch. As example, a batch may consist of ganister 200 pts.,  $\text{FeCl}_3$  2 pts., lactic acid 1 pt.,  $\text{Ca}(\text{CNS})_2$  1 pt., CaO 4 pts., and  $\text{H}_2\text{O}$  24 pts. C. A. KING.

**Furnace linings.**—See I. Sandpaper.—See V.

## IX.—BUILDING MATERIALS.

**Volume changes in brick masonry materials.** L. A. PALMER (J. Amer. Ceram. Soc., 1931, 14, 541—564).—Vol. changes due to temp., the alternate wetting and drying, freezing and thawing of various mortars and bricks, together with the changes during the setting and hardening of the mortars, were measured. The differential vol. changes between the brick and mortar due to temp. (under average climatic conditions) are small compared with those due to moisture content. The least vol. change occurs in mortars containing only CaO and  $\text{SiO}_2$ , but is larger than that occurring in well-fired brick. As far as shrinkage and expansion are concerned it is not worth while to use a 1 : 4 in preference to a 1 : 3 mortar, nor to use a 1 CaO : 1 cement : 6  $\text{SiO}_2$  mortar. Considerable variation in vol. change was noted among the masonry cements and was greater than that among the Portland cements. The vol. changes during the setting of mortars, although greater than the subsequent changes, are less destructive to masonry. The addition of Ca stearate to mortars decreases the rate at which the vol. changes occur, but does not alter their magnitude. J. A. SUGDEN.

**Action of some aggregates on Portland cement.** P. P. BUDNIKOV and M. I. NEKRICH (Tech. Novosti, 1930, 3, No. 17—18, 41—45).—Gypsum, dolomite, and marl act as densening agents and as absorbents. The particles of admixtures are enveloped by the colloidal gel; they do not lower, but sometimes increase, the mechanical strength. CHEMICAL ABSTRACTS.

**Action of kerosene on Portland cement.** V. I. NAZAROV (Stroit. Mat., 1930, No. 9—10, 31—37).—The tensile strength is lowered. After 3 months kerosene penetrated 0.2—0.3 cm. into cement walls. CHEMICAL ABSTRACTS.

**Slag cement without clinker.** P. P. BUDNIKOV and V. M. LEZHOEV (Stroit. Mat., 1930, No. 9—10, 19—27).—When the clinker is replaced by gypsum fired at 700°, anhydrite cement, or Estrich gypsum, the setting and hardening of slag cement are increased, and the mechanical properties are good. CHEMICAL ABSTRACTS.

**Chemical examination and mechanical testing of caustic burnt magnesia, Sorel cement, and artificial stone.** C. BÜTTNER (Chem.-Ztg., 1931, 55, 689—690, 710—711).—In the analysis of calcined  $\text{MgO}$ , combined  $\text{H}_2\text{O}$  is determined by heating at 190° and "loss on ignition" by heating at 1300° to expel  $\text{CO}_2$  and  $\text{SO}_3$ , which may be collected in KOH and the  $\text{SO}_3$

determined as  $\text{BaSO}_4$ . Free CaO is determined by titration with HCl after extraction with 10% sucrose solution. An outline of methods for the analysis of  $\text{MgO}$ - $\text{MgCl}_2$  cements and of artificial stone made from these cements with a sawdust filling is given, together with some details of methods of testing the strength and setting time of such mixtures. A. R. POWELL.

**Identification of wood by chemical means.** I. H. E. DADSWELL (Counc. Sci. Ind. Res., Australia, Pamph. No. 20, 1931, 16 pp.).—The use of aq. or EtOH extracts in colour tests with certain reagents is of limited value on account of the wide variation in the amount of extractable material in different samples of one species. Ash, alkalinity, differences in cellulose content, and behaviour of EtOH extracts on dilution with  $\text{H}_2\text{O}$  have been used to separate karri, jarrah, tallow-wood, blackbutt, and white mahogany, and a simple test depending on the precipitation of differently coloured substances on heating an EtOH extract of the wood with 1% NaOH solution serves to distinguish between red box and red gum. The burning-splinter test is reliable for differentiating between karri and jarrah only when sound heartwood which has not been pretreated with inorg. preservatives is used. B. P. RIDGE.

**Chemistry of Australian timbers. I. Lignin determination.** W. E. COHEN and H. E. DADSWELL (Counc. Sci. Ind. Res., Australia, Pamph. No. 22, 1931, 27 pp.).—Eucalypts, hemlock, and spruce woods contain substances of an extraneous nature which are insol. in  $\text{C}_6\text{H}_6$ -EtOH and remain with lignin when it is isolated by the standard procedure. These substances are dissolved by dil. solutions of NaOH without attack of the wood structure or of the lignin of hemlock and spruce. Previous extraction of the wood powder with 0.125N-NaOH for 80 min. at 98—100° enables reasonable vals. for the lignin content of jarrah, red ironbark, and mountain ash to be obtained. B. P. RIDGE.

**Manufacture and testing of asphalt paving materials.** D. M. WILSON (S.C.I., Chem. Eng. Group, Oct., 1931, 14 pp.).—Such materials can be divided, according to the manner in which they are laid, into the following classes: compressed, slab, mastic, and steam-rolled asphalts. The method of manufacture of each is fully described and typical specifications for the ingredients are given. Methods of testing of raw materials for asphalt paving and of the finished material are also examined. Bitumen is usually tested for penetration, ductility, m.p. (ring-and-ball), loss on heating, and viscosity (Engler). The relative proportions of the different grain sizes of the mineral aggregate can be obtained by sieving, but, owing to the varied classification of sieves, the determination is made preferably with a "flourometer" (the Mayntz-Petersen instrument is described). The manufactured material should be checked for bitumen content. Methods are given for determining the penetration of the bitumen as it exists in road surfaces. With steam-rolled mixtures it is necessary to know if bitumen is present in slight excess of the amount required to fill the voids and coat the grain surfaces, and an improved method of making the pat-stain test is given. Under mechanical tests, details of the moulding of test pieces and methods of

determining the hardness no., deformation, and tensile strength are described. H. S. GARLICK.

**Principles of manufacture of mastic asphalt.** A. W. ATTWOOLL (Proc. Chem. Eng. Group, 1930, 12, 113—120).

**Applications of mastic asphalt in a chemical works.** D. McDONALD (Proc. Chem. Eng. Group, 1930, 12, 121—131).

**Determining bitumen in asphaltic materials.**—See II.

#### PATENTS.

**Plaster for walls and the like.** E. N. and K. N. CRAIG (B.P. 356,711, 4.6. and 30.8.30).—A liquid or semi-liquid magnesian cement, which may contain fillers, pigments, etc., is sprayed on to wall surfaces preferably previously rendered with cement and sand, or on to tiles, slabs, etc.; the cement when partly set may be air-blasted to produce a ripple effect, or finished with paint, varnish, etc. L. A. COLES.

**Manufacture of [heat-]insulating material.** A. J. RUSS, Assr. to ARMSTRONG CORK CO. (U.S.P. 1,799,255, 7.4.31. Appl., 15.4.27).—A facing layer approx.  $\frac{1}{4}$  in. thick, comprising, *e.g.*, 50% of Portland cement, 46.75% of screened river sand, and 3.25% of asbestos fibre, is laid dry in a mould and covered with an aq. paste containing a mixture of, *e.g.*, 52.6% of kieselguhr, 45.2% of  $\text{Ca(OH)}_2$ , and 2.2% of asbestos fibre; the blocks so formed are cured by treatment with steam. L. A. COLES.

**Tenacious and fireproof composition.** C. MARCZINCEK (U.S.P. 1,800,024, 7.4.31. Appl., 12.3.28).—A mixture of approx. 45% of volcanic cinders, 20% of cement, 5% of  $\text{Ca(OH)}_2$ , 5% of gypsum, 25% of finely-ground pumice, and sufficient  $\text{H}_2\text{O}$  to cause the mass to set, is used for making walls, floors, etc., or is worked into slabs or blocks. L. A. COLES.

**Manufacture of building blocks and the like.** OESTERR.-AMERIKANISCHE MAGNESIT A.-G., and K. ERDMANN (B.P. 356,001, 10.10.30).—A slightly compressed rope of, *e.g.*, wood-wool is passed through an emulsion bath, excess emulsion is squeezed out as the rope leaves the bath, and the rope is then divided, by "flinging forks" moving in an elliptical path, into portions which are thrown by the forks on to a conveyor for transference to the moulds. L. A. COLES.

**Production of composite slab for building purposes.** W. SIEBERT (B.P. 356,466, 1.9.30).—A grooved wooden centre portion is covered on both sides with an asbestos-cement composition, metal, or artificial stone. C. A. KING.

**Manufacture of building units.** H. C. BYLAND and F. L. ORMESHER, Assrs. to CEMROC, INC. (U.S.P. 1,796,559, 17.3.31. Appl., 15.9.28).—A plastic mixture comprising concrete,  $\text{NH}_4$  stearate,  $\text{CaCl}_2$ , pigments (if desired), and  $\text{H}_2\text{O}$  is subjected in a mould to a sudden blow of, *e.g.*, 1000 lb. and then to a sustained pressure of, *e.g.*, 75 tons, after which the block is removed from the mould and air-dried. L. A. COLES.

**Production of a material more particularly for building and street structural purposes.** ALUM-

INIUMERZ BERGBAU U. IND. A.-G. (B.P. 356,797, 10.6.30. Ger., 10.6.29).—Bituminous material free from inorg. substances is mixed or melted with finely-divided bauxite; the product may be used, *e.g.*, for making pavements or roofing felts, or for sealing joints in earthenware pipes. L. A. COLES.

**Manufacture of sheathing lumber from redwood bark.** E. R. DARLING, Assr. to PACIFIC LUMBER CO. (U.S.P. 1,797,901, 24.3.31. Appl., 22.6.29).—The comminuted bark is successively dry-sieved, ground with water, wet-sieved, and boiled under pressure with a solution of a salt forming insol. tannates [*e.g.*,  $\text{Al}_2(\text{SO}_4)_3$  solution]; the fibres are then washed, sized, and formed into boards. L. A. COLES.

**Uniting wood and other surfaces.** C. ARNOLD. From I. F. LAUCKS, INC. (B.P. 356,137—8, 25.3.30).—(A) The surface of plywood is coated with powdered soyabean flour or other oleaginous seed flour or vegetable, and in the presence of sufficient natural or added moisture two or more plies are pressed together with or without heating. (B) A dry,  $\text{H}_2\text{O}$ -dispersible protein, *e.g.*, blood or lactalbumin, may be used as an adhesive, and is coagulated with a dry protein or an agent such as paraformaldehyde. C. A. KING.

**Rotary kiln. [Concrete] mixer.**—See I. **Compositions for roads etc.**—See II. **Fungicidal solution [for wood].**—See XVI.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Manufacture of malleable cast iron.** S. S. NEKRYTI (Vestn. Metalloprom., 1930, 10, 78—96).—Tests on threaded unions show that addition of 30—35% of scrap Fe and 10—15% of filings to the melt affords white Fe suitable for casting and cementation. During cooling from the max. cementation temp. to 800—810° graphite nuclei are formed, and ferrite grains are subsequently deposited about them.

#### CHEMICAL ABSTRACTS.

**Action of molecular nitrogen on iron-carbon alloys.** H. H. GRAY and M. B. THOMPSON (J.S.C.I., 1931, 50, 353—357 r).—The action of mol.  $\text{N}_2$  on steel was investigated at temps. ranging from 100° to 1100°. Microscopical and analytical evidence is cited to show that up to 700° the chief effects are nitride formation and attack on pearlite. From 700° to 800° there is a zone of max. decarburisation. At 850° in medium-C steels there is formed a transparent cryst. layer containing N and having a duplex structure. Above 850° this transparent layer is formed more easily and the decarburisation is less marked. The C interferes with the formation of the transparent layer. Six photomicrographs are shown.

**Production of low-carbon semi-steels.** II. M. HORIKIRI (J. Iron Steel Inst. Japan, 1930, 16, 935—960, 1063—1086).—The C in semi-steels is absorbed by contact of Fe with C; hence the quantity of coke should be minimal and the oxides should be reduced with Si. Fe high in Si dissolves only a little C. There is no significant relation between the depth of the cupola hearth and the C absorbed. CHEMICAL ABSTRACTS.



**Determination of martensite and austenite in quenched and tempered steels by means of X-ray analysis.** S. SEKITO (Sci. Rep. Tôhoku, 1931, 20, 368—376).—The quantity of austenite in a quenched steel is determined by comparing the intensity of the 200  $K\alpha$  line of austenite with that of the 111  $K\alpha$  line of Au; similarly, martensite may be determined from the intensities of the 112  $K\alpha$  martensite line and the same Au line. Between 100° and 170° the tetragonal  $\alpha$ -martensite is completely converted into the body-centred  $\beta$ -phase. Residual austenite is converted into  $\beta$ -martensite only slowly below 200°, but rapidly at 220—240°;  $\beta$ -martensite decomposes into ferrite and cementite at 220—280°.

A. R. POWELL.

**Determination of retained austenite in quenched steels.** K. TAMARU and S. SEKITO (Sci. Rep. Tôhoku, 1931, 20, 377—394).—The proportion of austenite in steels quenched under varying conditions has been determined by X-ray analysis and by saturation magnetisation. For any given steel the austenite content increases with rise of quenching temp. to a max. at 950—1050°, then decreases, and is greater when oil is the quenching medium than when  $H_2O$  is used. This is attributed to the accelerating effect of the stresses set up by the more severe  $H_2O$  quench on the austenite-martensite transformation, and is still more marked if the steel is subsequently cooled in liquid air; in this treatment one third of the residual austenite of a 0.89% C steel quenched from 1000° is converted into martensite when oil is used and one fourth when  $H_2O$  is used as the quenching medium.

A. R. POWELL.

**Structure of nodular troostite.** N. T. BELAIEV (Iron and Steel Inst., Sept., 1931. Advance copy, 19 pp.).—Primary nodular troostite has a lamellar structure similar to that of lamellar pearlite, but the interlamellar distance,  $\Delta_0$ , is 100  $m\mu$  and thus can be resolved only under high magnifications and with lenses of high aperture. The hardness of troostite is about 450, and hence Green's formula,  $H \times \Delta_0 = 79.59$ , does not apply. Lamellar structures having a hardness between those of troostite and pearlite (200—300) correspond to sorbite, which also does not follow Green's rule, but has  $\Delta_0$  below the crit. val. of 260  $m\mu$  and is formed within crit. cooling rates of 5—15°/sec. These distinctions between pearlite, troostite, and sorbite are considered sufficient to justify these substances being recognised as distinct and different constituents of steel.

A. R. POWELL.

**Origin of the banded structure of a hot-worked hypo-eutectoid steel.** F. C. THOMPSON and R. WILLOWS (Iron and Steel Inst., Sept., 1931. Advance copy, 26 pp.).—From the results of earlier workers, amplified by their own tests, the authors conclude that the banded structure found in hot-worked mild steel is due to the presence of O in solid solution. The phenomenon is not produced by slag bands or P in electrolytic Fe carburised and heat-treated in vac., but oxidation of mild steel followed by carburisation often produces a laminated structure analogous to that found in ordinary rolled mild steel. Rapid cooling inhibits the banding effect, slow cooling accentuates it, and heating in CO removes it to a depth slightly exceeding the zone of

visible carburisation without changing the distribution of visible inclusions and without removal of P. The available evidence suggests that the original cause of the differential O concentration lies in the coring of the crystals during solidification, and is not due to mechanical work other than the distortion of the dendritic structure.

A. R. POWELL.

**Effect of surface conditions produced by heat-treatment on the fatigue-resistance of spring steels.** G. A. HANKINS and M. L. BECKER (Iron and Steel Inst., Sept., 1931. Advance copy, 42 pp.).—The most important factor in the production of a low fatigue-resistance of unpolished spring steels appears to be the presence of a thin, soft, outer layer due to decarburisation during heat-treatment. Removal of this layer by polishing restores the fatigue-resistance to its normal value. Heat-treatment in a normal furnace atm. or in vac. (0.01—1 mm.) results in a diminished fatigue-resistance, as also does treatment in fused baths containing alkali chlorides alone or with carbonates or graphite. Normal fatigue-resistance is obtained by heat-treatment in cyanide baths or, in the case of Si-Mn steel, in a neutral or CO atm. The fatigue-resistance of Cr-V steel is only 80% of the normal after heat-treatment in CO, but is normal after immersion in a cyanide bath. For large specimens the steel should be covered with powdered graphite and annealed at 950° for hardening.

A. R. POWELL.

**Diffusion of tin into iron, with special reference to the formation of columnar crystals.** C. O. BANNISTER and W. D. JONES (Iron and Steel Inst., Sept., 1931. Advance copy, 23 pp.).—From experiments on the rate of diffusion of Sn into Armeo Fe in an atm. of  $H_2$  it is concluded that at temps. below the Ac3 point and at higher temps. up to the extent necessary to inhibit the  $\gamma$ -phase (2% Sn) diffusion proceeds by interchange or interpenetration of atoms without reorientation of the lattice, that subsequent diffusion produces a new phase ( $\alpha$ -solid solution) by the formation of a new lattice, and that the nuclei of this new phase are immediately absorbed by the growing crystals, as fast as they are formed, by the advancing solute. This explains the formation of columnar crystals of  $\alpha$ -Fe only at temps. above the Ac3 point, the absence of recrystallisation on cooling, and the absence of the "diffusion line" when diffusion occurs above the A4 point. The mechanism of diffusion can be expressed by the equations  $(y - y_1)^2 = mt$  and  $V = n(a^2 - 1)$ , where  $y$  and  $y_1$  are the distances from the origin of two zones of const. concentration,  $t$  is the time,  $V$  the rate of diffusion,  $T$  the abs. temp., and  $n$  and  $a$  are experimentally determined consts.

A. R. POWELL.

**Constitution of the iron-tin alloys.** C. A. EDWARDS and A. PREECE (Iron and Steel Inst., Sept., 1931. Advance copy, 27 pp.).—The system has been examined by thermal analysis in a  $H_2$  atm. and by micrographical examination. At the Fe end the liquidus falls in an almost straight line from the m.p. of Fe to 1110° at 51% Sn, then remains horizontal to 80% Sn, and finally falls slowly at first and then very steeply to the m.p. of Sn. Between 50% and 80% Sn there is a gap of immiscibility in the liquid phase. The solubility of Sn in solid Fe increases with

rise of temp. from about 10% at 300° to 18% at 760°, then decreases up to the m.p. of Fe. The system contains three compounds only:  $\text{Fe}_2\text{Sn}$  which is stable between 760° and 900°, but reacts with Sn at 800° to form  $\text{FeSn}$ ; this is stable below 800°, but reacts with Sn below 496° to form  $\text{FeSn}_2$ . The last-named compound has been prepared as very pure crystals which decompose into  $\text{FeSn}$  and Sn when heated above 496°.  $\text{Fe}_3\text{Sn}$  does not exist, and the thermal transformations which occur at 760° and 800° are not due to polymorphic changes in  $\text{FeSn}_2$  (cf. A., 1926, 475), but result from the decomp. of  $\text{Fe}_2\text{Sn}$  and formation of  $\text{FeSn}$ , respectively. The solid solubility of Fe in Sn is <0.01% at room temp.

A. R. POWELL.

**Determination of vanadium in high-speed steel.** F. FÄRBER (Chem.-Ztg., 1931, 55, 691).—The steel is boiled with 5%  $\text{H}_2\text{SO}_4$  until the Fe has dissolved, leaving a residue containing all the V and W. This residue is dissolved in HCl with a little  $\text{HNO}_3$  and the solution evaporated to ppt.  $\text{WO}_3$ . The filtrate is evaporated with  $\text{HNO}_3$ ,  $\text{HPO}_4$ , and  $\text{H}_2\text{SO}_4$  until fumes of  $\text{SO}_3$  are copiously evolved. After cooling, the  $\text{V}_2\text{O}_5$  is reduced by double evaporation with HCl; the solution is then diluted and titrated with  $\text{KMnO}_4$ .

A. R. POWELL.

**Determination of manganese in cobalt steels.** F. SPINDECK (Chem.-Ztg., 1931, 55, 723).—The steel is dissolved in HCl and the Fe oxidised with  $\text{HNO}_3$  and precipitated with  $\text{ZnO}$ . An aliquot part of the solution is treated with Br and aq.  $\text{NH}_3$  in excess and boiled to ppt.  $\text{Mn}(\text{OH})_2$ . The ppt. is washed free from  $\text{Cl}^-$ , dissolved in  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}_2$ , and oxidised with  $\text{AgNO}_3$  and  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  to  $\text{HMnO}_4$ , which is titrated with  $\text{Na}_2\text{HASO}_3$ .

A. R. POWELL.

**Corrosion of cans.** T. N. MORRIS and J. M. BRYAN (Food Manuf., 1931, 6, 100—102).— $\text{O}_2$  increased the corrosion of Fe by 0.5% citric acid (+ Na citrate) solution at low more than at high acidities. Sn was not attacked by non-oxidising org. acids, but was readily attacked in presence of  $\text{O}_2$ . The behaviour of Sn plate was similar to that of Sn-Fe couples. Substances in unrefined beet sugar inhibit the corrosion of Fe; in beet sugar and agar-agar that of Fe and Sn; and in inverted cane sugar that of Sn.  $\text{SO}_2$  accelerated the corrosion of Fe at  $p_{\text{H}}$  2.4 and retarded it at  $p_{\text{H}}$  5.5.  $\text{SO}_2$  makes Fe permanently anodic to Sn, since a film of Sn sulphide is formed.

CHEMICAL ABSTRACTS.

**Brinell ball hardness tests.** M. ICHIHARA (Tech. Rep. Tôhoku, 1931, 10, 25—41).—The cross-sectional curves of the impressions formed by a spherical ball in the Brinell hardness test may be represented by the empirical formula  $y = K_a e^{-ax} - K_b e^{-bx}$ , which is applicable with an error of <0.002 mm. to all the common metals. The consts.  $K_a$ ,  $a$ ,  $K_b$ , and  $b$  vary with the degree of internal stress;  $K_a$ ,  $a$ , and  $b$  decrease with increasing deformation by cold-work, but  $K_b$  increases. For annealed Cu, Al, or bronze  $K_a < K_b$ , but for steel, cast Fe, Sn, and Zn, both hard-worked and annealed,  $K_a > K_b$  owing to the difficulty of completely removing the internal stresses in these metals by annealing. The "annealing degree" of a metal is given by the expression  $2x/d$ , the value of  $x$  being that at

which  $y$  has its max. value. Under ordinary annealing conditions the annealing degree of Cu is 3, of mild steel 0.32, and of high-C steel 0.26.

A. R. POWELL.

**Diameter measurement of certain Brinell indentations in cold-rolled metal.** H. O'NEILL (Inst. Metals, Sept., 1931. Advance copy, 4 pp.).—Measurements are found to differ by 3—4%, according to whether vertical or oblique lighting is used. The min. observation should be recorded, and the use of "dark-ground" objectives is to be preferred.

C. A. KING.

**Effect of cold-working on the magnetic susceptibility of metals.** K. HONDA and Y. SHIMIZU (Sci. Rep. Tôhoku, 1931, 20, 460—488).—The magnetic susceptibility of both diamagnetic and paramagnetic metals is reduced by cold-work to an extent which is linearly proportional to the resulting increase in  $d$ . Cu, which is normally a weak diamagnetic metal, becomes weakly paramagnetic with severe cold-work. Annealing at a temp. above that at which internal stress is relieved restores the susceptibility to its original value. All these facts may be explained by Honda's theory of magnetism.

A. R. POWELL.

**Rendering visible filed-out impressions in metals.** O. MEZGER, B. SCHÖNINGER, and E. ELBEN (Z. angew. Chem., 1931, 44, 651—652).—Filed-out impressions of numbers or designs in metal articles may be rendered visible again by suitable etching if the original design was produced by stamping with a steel die, as the greater deformation produced by the stamping causes these parts to be more readily etched than the remaining (softer) metal. For steels 1 : 1  $\text{HNO}_3$ -HCl, 1 : 9 fuming  $\text{HNO}_3$ -EtOH, or 10%  $\text{FeCl}_3$  solutions are the most suitable etching reagents. Cu, brass, and bronze are etched with 10%  $\text{FeCl}_3$  solution to which a little  $\text{KClO}_3$  solution may be added, and Al is etched with 10%  $\text{MnCl}$  solution in conc. HCl or first with 40% HF and then with conc.  $\text{HNO}_3$ . Ag and Ni alloys should be etched with conc.  $\text{HNO}_3$  or with  $\text{HNO}_3$  in EtOH followed by a short immersion in HCl- $\text{FeCl}_3$  solution.

A. R. POWELL.

**Crystallisation [of aluminium alloys] at pressures up to 20,000 atmospheres.** G. WELTER (Z. Metallk., 1931, 23, 255—259).—Castings of Cu- and Zn-Al alloys, and of silumin, cetel, and lautal, made under pressures of 12,000—20,000 atm., have a homogeneous, fine-grained structure free from segregations and with an extraordinarily regular distribution of the alloying constituents; the hardness, tensile strength, and ductility are all simultaneously increased. Thus for the German Zn-Al alloy, pressure increases the hardness from 49 to 85, the tensile strength from 19.5 to 26.5 kg./sq. mm., and the elongation from 13 to 20%; for silumin the corresponding increases are from 54 to 73, from 21 to 27.7 kg./sq. mm., and from 10 to 15%. The effect of high-pressure casting on silumin is to displace the eutectic composition still further towards the Si end and to raise the m.p. The Na modification treatment raises the eutectic composition from 11 to 13% Si and reduces the m.p. from 575° to 565°, whereas at 12,000 atm. pressure the eutectic composition is raised to 16% Si and the m.p. to above 600° without addition of Na and the grain size is more uniform.

Pressure also increases the solid solubility of Si in Al to 3% Si at 600°. A. R. POWELL.

**Annealing of aluminium and aluminium alloys.** H. NISHIMURA and G. SHINODA (J. Wednesday Soc., Japan, 1927, 5, 476—488).—In cold-worked Al and Al alloys elasticity was recovered at 90—180°. Annealing at 250° gives rise to recrystallisation.

CHEMICAL ABSTRACTS.

**Influence of iron on [the properties of] duralumin.** W. KROENIG (Z. Metallk., 1931, 23, 245—249).—With increasing Fe content the hardness and tensile strength of aged duralumin is reduced to an extent which is greater the higher is the quenching temp. Immediately after quenching, Fe-rich duralumin has a lower strength and hardness than a similar alloy with a low Fe content, but during ageing the strength increases to the same extent irrespective of the amount of Fe present, although the hardness rises less rapidly with a high than with a low Fe content. This behaviour is attributed to the action of the Fe in reducing the solid solubility of  $\text{CuAl}_2$  in Al at high temp. Fe up to 0.9% forms a eutectic in duralumin which appears to contain  $\text{CuAl}_2$  and this eutectiferous  $\text{CuAl}_2$  has only a small tendency to dissolve in solid Al. No evidence in support of the theory that Fe retards the separation of  $\text{CuAl}_2$  from Al could be obtained. A. R. POWELL.

**Influence of variations in heat treatment and ageing on duralumin.** A. VON ZEERLEDER (Inst. Metals, Sept., 1931. Advance copy, 13 pp.).—If Avional-D (3.85—3.94% Cu, 0.53% Mg, 0.47% Mn, 0.56% Si, 0.3% Fe), a material comparable with duralumin, is quenched in cold  $\text{H}_2\text{O}$ , considerable distortion ensues. Quenching in a bath at 50° eliminates distortion. Ageing at 50° produces no ill-effects. Ageing at 145° is accompanied by serious changes in electrochemical potential, resistivity, tensile properties, and corrodibility. The temp. changes which occur during the time interval between removal of the metal from the furnace and quenching are studied. E. H. BUCKNALL.

**Unsoundness in aluminium sand-castings. I. Pinholes: their causes and prevention. II. Effects of using metal previously subjected to corrosive conditions.** D. HANSON and I. G. SLATER (Inst. Metals, Sept., 1931. Advance copies, 29 pp. and 12 pp.).—I.  $\text{H}_2\text{O}$  vapour in the furnace atm. or bubbled into molten Al or Al alloys rapidly produces extensive pinholing and unsoundness; this may be removed by treatment with dry  $\text{N}_2$  or dry  $\text{Cl}_2$ , but preferably by a 1:1 mixture of these gases, at a temp. just above the m.p., but below 700°. In large melts made in a gas-fired crucible furnace the rate of absorption of gases at 760° (the usual pouring temp.) is usually greater than the rate at which they can be removed by the  $\text{N}_2$ — $\text{Cl}_2$  treatment. The  $\text{TiCl}_4$  treatment removes gases and refines the grain structure of "Y"-alloy, but is not so effective with the 12% Si alloy or with Cu—Al alloys. Castings of Al alloys containing Zn can be obtained free from pinholes by heating the alloy at 950° until Zn commences to vaporise. S, Se, and Te are insol. in Al and, in general, give rise to unsoundness in sand-castings, although in some cases Se and Te appear to be beneficial.

II. Castings made from Al or Al alloys which have been exposed to corrosive conditions are generally unsound and contain many pinholes; the extent of the unsoundness depends on the type of corrosion and the length of time the metal has been subjected to these conditions. It is suggested that the metal has adsorbed nascent H liberated by the electrolytic action inducing the corrosion; this H is liberated, on remelting and casting, in the mol. condition and thus produces pinholes. Treatment of the corroded metal with  $\text{N}_2$  at 500° or anodic pickling before melting fails to prevent formation of pinholes in the castings. A. R. POWELL.

**Properties of a copper-nickel-aluminium alloy.** I, II. T. SAITO (J. Wednesday Soc., 1927, 5, 433—442, 465—471).—The properties of the Cu—Ni (10%)—Al (2%) alloy were improved by heat treatment. Hardening by quenching and tempering is the result of the separation of the  $\beta$ - from the  $\alpha$ -phase. Corrosion tests were carried out. CHEMICAL ABSTRACTS.

**Drawing of non-ferrous wires.** E. L. FRANCIS and F. C. THOMPSON (Inst. Metals, Sept., 1931. Advance copy, 25 pp.).—The tension required is proportional to the max. stress of the material and independent of the speed over a range of 20—600 ft. per min. The pull depends on the reduction of area effected. The best lubricant was a Na soap with not less than 75% of fatty acid and free from  $\text{H}_2\text{O}$  or glycerin. Diamond dies require less power than those of WC, which in turn require only about  $\frac{1}{4}$  that of steel dies. C. A. KING.

**Experiments in wire-drawing. I. Behaviour of a composite rod. W. E. ALKINS and W. CARTWRIGHT. II. Relation between reduction of area by cold-drawing and tensile strength of H.-C. copper.** W. E. ALKINS (Inst. Metals, Sept., 1931. Advance copy, 11 pp. and 9 pp.).—Examination of composite round rods of high-conductivity (H.-C.) Cu built up by drawing a number of tubes over a solid core and then drawing through dies of different angle showed that all the layers undergo a proportionate reduction in area. Reduction was found to commence and to finish earlier nearer the centre of the rod. Through dies tapering at 5° the tensile strength/reduction of area curve of Cu is approx. rectilinear for reductions up to 15% and above this forms part of a rectangular hyperbola. The max. reduction possible at a single draft from annealed rod is about 56%, and successive drafts with 40% reduction can be effected. C. A. KING.

**Work-hardening capacity and elongation properties of copper.** H. O. NEILL and J. W. CUTHBERTSON (Inst. Metals, Sept., 1931. Advance copy, 17 pp.).—Specimens of H.-C. copper (> 99.92% Cu) have been reduced 0—60% at one pass by cold-rolling. After ageing for 6 yrs. at room temp., tensile and indentation tests have been made which have been followed by attempts to deduce values for the work-hardening capacity from the results of the tests. It is found that reliable criteria of this property are afforded by the "uniform elongation" val., the "extensibility," and the elongation on  $\leq 15^\circ/\text{A}$ . In the main the results indicate a great loss in cold-working capacity in the first 25% reduction by rolling. E. H. BUCKNALL.

**Physical testing of copper and copper-rich alloys in the form of thin strip.** M. COOK and E. C. LARKE (Inst. Metals, Sept., 1931. Advance copy, 17 pp.).—Tensile tests on strip material (H.-C. copper, 95:5 Cu:Zn, 70:30 Cu:Zn, 64:36 Cu:Zn, and 80:20 Cu:Ni) of thickness  $\leq 0.02$  in. gave consistent results, both in the annealed state and after various degrees of cold-rolling. Test results on material 0.01 in. thick were not reliable. Hardness vals. given by the Vickers diamond indentation test appeared more valuable than other hardness measurements. Cupping tests do not give sufficiently easily understandable results for use in specifications. The tabulated results of tensile and hardness tests indicate the average performance of good commercial materials. E. H. BUCKNALL.

**Flotation of copper oxide ores.** DAVIDOVICH and N. M. LUBMAN (Tzvet. Met., 1930, 5, 977–991).—Sulphurisation produces a thin film of sulphide which permits separation by flotation (yield 60–83% Cu).

CHEMICAL ABSTRACTS.

**Use of magnesite in the copper-melting furnaces of the Krasny Vyborjets plant.** D. D. VOIEIKOV (Tzvet. Met., 1930, 5, 1039–1048).—Despite sensitivity to variations in temp. and to discontinuity of expansion, successful use is recorded.

CHEMICAL ABSTRACTS.

**Hydrometallurgical treatment of Ural nickel ores.** G. G. URAZOV and M. M. ROMANOV (Tzvet. Met., 1930, 5, 1136–1144).—Ni may be extracted from washed ores containing 0.9–5.68% Ni by means of gaseous  $\text{SO}_2$  or  $\text{SO}_2$  solution. CHEMICAL ABSTRACTS.

**Refining of zinc obtained by distillation at Alagir.** N. P. ASREY, V. V. DOLIVO-DOBROVLSKI, and B. F. GRASHCHENKO (Tzvet. Met. Suppl., 1930, 5, No. 5, 1–40).—The best liquation temp. is  $450^\circ$ . The rate of separation of Pb increases with decrease in the Fe content. A second liquation affords no appreciable improvement. By liquation the Pb content can be reduced to 0.83% and the Fe to 0.023%. No solid solution of Fe in Zn was observed; the microstructure showed the formation of crystals of  $\text{FeZn}_7$  when the Fe content exceeded 0.06%. Redistillation may reduce the Fe content to 0.05%. Cd can be removed only by fractional distillation.

CHEMICAL ABSTRACTS.

**Effects of cold-rolling and of heat-treatment on some lead alloys.** H. WATERHOUSE and R. WILLOWS (Inst. Metals, Sept., 1931. Advance copy, 23 pp.).—Cold-rolling of Pb alloys with small quantities of Sn, Sb, or Cd increases the hardness of initially soft alloys and decreases that of initially hard alloys, so that alloys having a Brinell hardness of 5–18 in the cast condition have a hardness of 8–11 after rolling, and this may be further reduced to 6–8 by storage at the ordinary temp. The rate of self-annealing depends on the composition of the alloy and the temp., alloys containing Sn softening rapidly, whilst those containing Sb alone or Sb and Cd soften only slowly. The latter class of alloys age-harden after quenching from  $240^\circ$ , whereas those containing Sn or Sn and Cd age-harden only slightly and temporarily after this treatment. Cold-rolling hardens soft aged alloys and softens age-hardened alloys. Alloys most susceptible to age-hardening contain 2–3% Sb, 1%

Cd + 2% Sb, or 1.5% Cd + 0.5% Sb; they all age-harden after casting in chill moulds. As the mechanical properties of these alloys are equal to those of Pb-Sb alloys with 5–12% Sb, it is suggested that they may be more serviceable for accumulator grids than the alloys with higher Sb.

A. R. POWELL.

**Spectrographic assay of some alloys of lead.** D. M. SMITH (Inst. Metals, Sept., 1931. Advance copy, 15 pp.).—By direct comparison of the spark spectra of Pb alloys containing Sb, Cd, or Sn, alone or together, with the spark spectra obtained from standard alloys containing known amounts of these elements, results with an accuracy of  $\pm 10\%$  are obtained when the constituents are present in the range 0.1–1%. Application of the logarithmic sector method to alloys containing up to 3% Sn gives results within the same range of accuracy.

A. R. POWELL.

**Reduction of lead carbonate ores by means of gases.** V. A. VANYUKOV, N. N. MURACH, and P. K. PIGROV (Tzvet. Met., 1930, 5, 1145–1150).— $\text{H}_2$  reduces the enriched (60% Pb) ore more rapidly and at a lower temp. than CO, but fritting and slagging take place more rapidly with CO. CHEMICAL ABSTRACTS.

**Micro-determination of lead dust in factories.** K. P. KHOKHRYAKOV (Arb. Leningrad Inst. Gewerbehyg., 1930, 3, No. 4, 13–26).—Unless members of group III are present (error 20%) colorimetric determination, using the Arnold-Menzel-Trillat reagent, is recommended. Most accurate results are obtained by determining the Pb as  $\text{PbI}_2$ ,  $\text{PbCrO}_4$ , or  $\text{K}_2\text{CuPb}(\text{NO}_3)_6$ .

CHEMICAL ABSTRACTS.

**Determination of tin in antifriction alloys.** A. A. BOCHVAR and V. N. MASLENNIKOV (Tzvet. Met., 1930, 5, 500–503).—Reduction is rapidly effected with Cd; the end-point of the titration is sharp.

CHEMICAL ABSTRACTS.

**Determination of silver in gold bars.** F. MICHEL (Chem.-Ztg., 1931, 55, 731–732).—Results of 95 assays of alloys of electrolytic Au, Ag, and Cu in various proportions showed errors varying from  $-12.6$  to  $+9.6$  in the Ag content, low amounts being obtained with low, and the reverse with a high, Au content. The high Ag results were due to persistence of Pb, and were reduced, but still appreciably high, when the Pb was decreased, whilst altering the temp. of cupellation also reduced the errors in the Ag content. To compensate for these errors, it is necessary to make a preliminary assay and then conduct a test on an alloy of the proportions found, so as to obtain a correction factor.

W. J. WRIGHT.

**Conditions that cause changes in the composition of plating baths, and possible remedies.** F. C. MATHERS (Monthly Rev. Amer. Electroplaters' Soc., 1929, 16, No. 12, 9–24).—To avoid irregular corrosion, the anode should be very pure and thoroughly annealed; Zn and Cd should be amalgamated. Fe should be excluded. Excess of free acid, cyanide, etc. should be minimal. High c.d. is preferable, and stirring is advantageous when anode corrosion is poor.

CHEMICAL ABSTRACTS.

**Effect of presence of nitric acid in chromium-plating baths.** N. P. LAPIN and L. N. GOLTZ (J. Appl.

Chem., Russia, 1930, 3, 1151—1153).—A higher c.d. and more current are required, and a dull greyish deposit is obtained.

CHEMICAL ABSTRACTS.

**Chromium-plating of pure aluminium and its alloys.** K. ALTMANNBERGER (Chem.-Ztg., 1931, 55, 709—710).—The Al article is first cleaned by a cathodic pickle in dil. alkali to remove grease, and then immersed in a conc. solution of ZnO in NaOH, whereby it becomes coated with a smooth, bright, adherent film of Zn. After rinsing in cold H<sub>2</sub>O, the article is plated with Ni in a bath containing 100 g. of NiSO<sub>4</sub>·7H<sub>2</sub>O, 150 g. of Na citrate, 150 g. of Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O, 100 g. of H<sub>3</sub>BO<sub>3</sub>, and 250 g. of citric acid per litre. Finally the article is plated with Cr in any of the usual CrO<sub>3</sub> baths.

A. R. POWELL.

**Analysing chromium[plating] solutions.** J. L. NEWELL and W. H. KEEFE (Metal Ind., N.Y., 1931, 29, 206—207).—A complete scheme for the determination of CrO<sub>4</sub><sup>''</sup>, Cr<sup>'''</sup>, SO<sub>4</sub><sup>''</sup>, Cu<sup>''</sup>, Fe<sup>'''</sup>, Ni<sup>''</sup>, and Zn<sup>''</sup> in such solutions is given.

CHEMICAL ABSTRACTS.

**Zinc plating.** R. W. RICE (Monthly Rev. Amer. Electroplaters' Soc., 1930, 17, No. 1, 29—33, 35).—Acid and cyanide electro-galvanising baths are specified.

CHEMICAL ABSTRACTS.

**Protection of magnesium alloys against corrosion.** H. SUTTON and L. F. LE BROcq (Inst. Metals, Sept., 1931. Advance copy, 20 pp.).—Metal coatings applied by calorising, sherardising, metal-spraying, or electrodeposition of Zn or Cu fail to increase the resistance of Mg to sea-water. Immersion of the cleaned metal in solutions of chromates or dichromates effects substantial improvement, particularly if supplemented by a cellulose enamel. A preferred bath contains 1.5% of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, 1% of potash alum, and 0.5% of NaOH and is used at 95°. Lanoline was effective as a film on the metal, but does not allow of an enamel over-coating. Cleaning Mg by means of dil. HNO<sub>3</sub> results in considerable dimensional losses, and finely-machined fittings should be treated with hot NaOH.

C. A. KING.

**Rapid determination of arsenic in white metals.** K. L. ACKERMANN (Chem.-Ztg., 1931, 55, 702).—The alloy is dissolved in HNO<sub>3</sub>, the solution evaporated to dryness, the residue heated to decompose nitrates, and the resulting oxides are distilled with conc. HCl and Cu<sub>2</sub>Cl<sub>2</sub>. The As in the distillate is determined with I in the usual way.

A. R. POWELL.

**Relative safeties of mild and high-tensile alloyed steels under alternating and pulsating stresses.** B. P. HAIGH (Proc. Chem. Eng. Group, 1929, 11, 9—19).

**Caustic embrittlement.** W. S. COATES (Proc. Chem. Eng. Group, 1930, 12, 142—146).

**Treatment of flotation concentrates preparatory to zinc smelting.** S. ROBSON (Proc. Chem. Eng. Group, 1929, 11, 58—71).

**Welding in chemical works.** J. R. BOOER (Proc. Chem. Eng. Group, 1930, 12, 80—89).

**CuSO<sub>4</sub> from Cu ores etc.**—See VII. **Varnishes on phosphatised Fe.**—See XIII.

## PATENTS.

**Open-hearth furnace.** R. L. LEVENTRY (U.S.P. 1,798,611, 31.3.31. Appl., 8.4.21).—Air is drawn from any convenient point in the uptake from the regenerator by means of a high-pressure steam or air nozzle and is directed into the gas port. The fuel gas is delivered from the sides immediately in front of the air delivery, and so an intimate contact of the gases results in a short intensive flame.

C. A. KING.

**Open-hearth furnace.** S. H. MCKEE (U.S.P. 1,798,618, 31.3.31. Appl., 24.6.21).—For the better contact of air and fuel gas the forward ends of the air ports lying on either side of the gas port are divided by a partition. When supplying air to the furnace, a hinged deflector diverts the air to the sub-channel nearest to the gas port, and when the furnace is reversed the deflector lies in a recess in the furnace wall and allows the full area of the port for the passage of the flue gases.

C. A. KING.

**Down-blast oil-smelting furnace.** M. S. ROGERS, Assr. to F. E. KENNEDY (U.S.P. 1,799,643, 7.4.31. Appl., 6.2.29).—The inner walls of a stack furnace of increasing dimensions towards the bottom are provided with a continuous, well-defined, helical recess in which burners are arranged at different positions. The stack discharges into a converter chamber which connects with the main chimney.

C. A. KING.

**Reduction [of ores] and reducing furnaces.** S. L. MADORSKY, Assr. to GATHMYS RES. CORP. (U.S.P. 1,796,871, 17.3.31. Appl., 3.3.28).—Reducing gases are passed through a molten ore, e.g., Fe<sub>3</sub>O<sub>4</sub>, in a relatively deep hearth and then over molten ore in a shallow hearth partly to reduce it and maintain the temp. Ore is conveyed from the shallow to the deep hearth for final reduction by means of a connecting duct.

C. A. KING.

**Metal-recovering furnace.** T. D. STAY and H. O. BURROWS, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,797,276, 24.3.31. Appl., 25.6.26).—The hearth of a closed furnace chamber extends outside the furnace to form two exposed portions for the rapid submersion of a scrap-metal charge and for the removal of slag. Burners near to the top of the side wall direct the flame in an inclined direction on to the metal, thus causing agitation of the bath; the spent gases leave on the same side as, but at a lower level than, the burners. Mechanical means are provided for submerging the scrap metal.

C. A. KING.

**Metals-reduction bomb.** W. P. KIERNAN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,795,780, 10.3.31. Appl., 25.4.28).—For the reduction of difficultly-reducible metals, e.g., Th, by the Ca-CaCl<sub>2</sub> method, a cylindrical bomb of Ni-Cr-Fe alloy is used. A ground tapered stopper capable of being hermetically sealed permits the insertion of a "pure" Fe liner which contains the charge and proves the most resistant to the corrosive action of the reducing process.

C. A. KING.

**Manufacture of briquettes or non-moulded lumps of comminuted minerals [iron ore] for metallurgical purposes.** L. L. SIMPSON, Assr. to NEW ERA IRON & STEEL CORP. (U.S.P. 1,729,496, 24.9.29. Appl., 15.11.23).—Fine Fe ores are briquetted with a binder

comprising 2–3% of kieselguhr, 0.5–1% of NaOH, 0.5–1.25% of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ , and 9–12% of  $\text{H}_2\text{O}$ , based on the wt. of ore. A. R. POWELL.

**Manufacture of cast iron articles.** FORD MOTOR CO., LTD. (B.P. 356,795, 6.6.30. U.S., 21.6.29).—Valve tappets are made of Fe containing 3.4–3.7% C, 2.5–2.9% Si, <0.1% S, 0.4–0.48% P, and 0.6–0.8% Mn, cast in chill moulds, annealed at 925–955°, machined to shape, and hardened by oil-quenching from 845–955°. A. R. POWELL.

**Utilising scrap iron by welding whilst avoiding loss by burning.** T. GOLDSCHMIDT A.-G. (B.P. 356,652, 13.3.31. Ger., 26.3.30).—The scrap is formed into bundles which are heated to welding temp. in a bath of Na silicate slag and then welded by shingling, hammering, or rolling. A. R. POWELL.

**Articles made from steel alloys capable of resisting chemical attack.** F. KRUPP A.-G., and H. KOPPERS A.-G. (B.P. 356,264, 5.6.30. Ger., 13.6.29).—Articles resistant to  $\text{H}_2\text{SO}_4$ ,  $\text{SO}_2$ , and thiosulphate solutions are made from steel containing 17–40 (17–19)% Cr, 25–6 (8–10)% Ni, 0.02–0.5 (0.02–0.2)% C, and 0.2–3% Mo. The articles are rendered passive by annealing at 950–1200° and quenching in  $\text{H}_2\text{O}$ . A. R. POWELL.

**Silicon steel.** V. B. BROWNE (U.S.P. 1,784,811, 16.12.30. Appl., 13.2.30).—Scrap Fe is melted in an electric furnace and refined to remove completely the Si and most of the C; it is then covered with a fused slag containing 70% CaO and 30%  $\text{Al}_2\text{O}_3$  and treated with several additions of a mixture of Al powder and CaO to remove O and S. The requisite quantity of ferro-silicon is then added to give a steel with 3.5–4.5% Si, and after removing the slag the metal is cast. Sheet prepared from the castings is very ductile and has a high permeability. A. R. POWELL.

**[Nickel-iron] magnetic alloys.** CALLENDER'S CABLE & CONSTRUCTION CO., LTD., and S. BECKINSALE (B.P. 355,456, 14.7.30).—High-permeability magnetic alloys comprise 25–80% Ni, 15–60% Fe, 0.05–10% Ag, <10% Cu, and 0.1–10% Mn. For wire and strip the alloy contains 78% Ni, 21–1% Fe, 0.05% Cu, 0.7% Mn, and 0.1% Ag, and for dust cores either (a) 77% Ni, 20% Fe, 0.05% Cu, 0.75% Mn, 2% Ag, and 0.2% Si, or (b) 37.5% Ni, 57% Fe, 1% Cu, 0.5% Mn, 3% Ag, and 1% Si. A. R. POWELL.

**Concentration of [copper silicate] minerals by froth flotation.** L. W. FERRIS (U.S.P. 1,784,280, 9.12.30. Appl., 11.12.28).—The ore is heated to redness, cooled, ground, and subjected to flotation in the usual way. A. R. POWELL.

**Lead-calcium alloys for sheathing cables.** STANDARD TELEPHONES & CABLES, LTD., Assees. of G. M. BOUTON (B.P. 356,801, 12.6.30. U.S., 30.10.29).—Pb alloys with 0.02–0.1% Ca are extruded at above 225°, cooled rapidly, and aged to have a tensile strength below 4500 lb./sq. in. A. R. POWELL.

**Coating of metal bodies with tungsten bronzes and/or molybdenum bronzes.** EGYESÜLT IZZO-LAMPÁ ÉS VILLAMOSÁGI RÉSZVÉNYTÁRSASÁG (B.P. 355,334, 22.5.30. Hung., 23.12.29).—The metal article

is made the cathode in a hot conc. solution of an alkali or alkaline-earth metal salt of a meta- or poly-tungstic and/or molybdic acid, e.g.,  $\text{BaW}_4\text{O}_{13} \cdot 9\text{H}_2\text{O}$ . A. R. POWELL.

**Electrodeposition of chromium.** R. AUERBACH, ASSR. to CHROMEPLATE, INC. (U.S.P. 1,730,349, 8.10.29. Appl., 22.6.27. Ger., 30.6.26).—Cr-plating baths are treated with a small proportion of a salt of an acid having a dissociation const. less than the first dissociation const. of  $\text{H}_3\text{PO}_4$ , e.g.,  $\text{CrPO}_4$  or an oxalate or acetate. A. R. POWELL.

**Electroplating of aluminium and its alloys.** AUTOMATIC TELEPHONE MANUFG. CO., LTD., C. A. VELARDE, and E. SMITH (B.P. 356,443, 20.8.30).—The Al article is cleaned first in an alkaline bath, then in dil. acid, and finally plated with Ni in any suitable bath. The plated article is heated for 10–15 min. in a non-oxidising atm. at 475–525°. After cooling, the surface may be polished and plated with Cu or Cr. A. R. POWELL.

**Handling furnace residues.** Solutions for storage vessels etc.—See I. Furnace for coal analysis. Lubricant [for die-drawing]. Slushing grease.—See II. Smelting furnace. Crucibles for fused electrolytes.—See XI.

## XI.—ELECTROTECHNICS.

**Electric furnaces for heat treatment.** A. G. LOBLEY (Proc. Chem. Eng. Group, 1930, 12, 90–95).

**Kiering processes.**—See VI. Analysis of hypochlorites.  $\text{K}_3\text{Fe}(\text{CN})_6$ .—See VII. Plating baths. Zn. Cr on Al.—See X.

## PATENTS.

**Electric furnace for smelting of metals.** E. F. RUSS (U.S.P. 1,793,137, 17.2.31. Appl., 26.3.30. Ger., 12.3.29).—An electric furnace for smelting metals in grooves filled with liquid metal and ending above in a hearth space, co-operating with a conducting bridging web having tubular projections on the side turned away from the groove mouth and provided with slits at its lower end, is claimed. J. S. G. THOMAS.

**[Electric] furnace.** J. F. BAKER, ASSR. to WESTINGHOUSE ELECTRIC & MANUFG. CO. (U.S.P. 1,795,921, 10.3.31. Appl., 23.6.26).—A motor-driven conveyor arranged within an annular chamber carries a number of pivotally-mounted hearths, and tilting means are arranged to tilt each hearth when it arrives opposite the outlet and to operate only when the driving motor is inactive. J. S. G. THOMAS.

**Induction furnace.** P. H. BRACE, ASSR. to WESTINGHOUSE ELECTRIC & MANUFG. CO. (U.S.P. 1,795,926, 10.3.31. Appl., 27.7.26).—The furnace comprises a crucible arranged within an induction coil in the direct path of the magnetic axis of the coil, a cylindrical core member composed of metallic, magnetic, radially-spaced laminations arranged outside the coil, and means for arch-binding the laminations. J. S. G. THOMAS.

**Crucibles for electrolysis of fused electrolytes.** H. BARON. From SIEMENS & HALSKE A.-G. (B.P. 357,032, 23.12.30).—Radial, internal wall channels and intercommunicating transverse channels are arranged

in the wall of a conducting crucible, *e.g.*, of graphite, which is closed by a hollow metal plate through which air or other cooling fluid can be circulated.

J. S. G. THOMAS.

**Manufacture of oxide cathodes for electric-discharge tubes.** EGYESÜLT IZZOLÁMPA ÉS VILLAGMOSÁGI RÉSZVÉNYTÁRSASÁG (B.P. 356,715, 6.6.30. Hung., 23.12.29).—A core, *e.g.*, of W, Mo, or Ni, is coated with W- and/or Mo-bronze, *e.g.*, by electrolysis of  $\text{BaW}_3\text{O}_9$ , prior to deposition of alkaline-earth metal vapour on the core.

J. S. G. THOMAS.

**Luminous electric-discharge tubes.** CLAUDE NEON LIGHTS, INC., ASSCES. OF L. L. BECK (B.P. 356,745, 5.6.30. U.S., 30.11.29).—A gas-discharge tube having a thermionic cathode, *e.g.*, a hollow Cu, Fe, or Ni cylinder or cone coated with Ba suboxide, supplied with current corresponding to a discharge c.d. of 0.03—1.25 amp. per sq. cm. of tube cross-section, and a gas filling composed of He and not more than 8% of Ne, together with not more than 0.5% of A, Kr, or Xe, is claimed.

J. S. G. THOMAS.

**[Electrodes for gas-filled] luminous tube.** J. E. GROSS (U.S.P. 1,797,683, 24.3.31. Appl., 28.6.29).—A powdered mixture composed of a metallic nitrate, *e.g.*,  $\text{Ti}(\text{NO}_3)_4$ , and graphite is covered with a sol. silicate, *e.g.*, Na silicate, and heated electrically on a support to form a solid mass.

J. S. G. THOMAS.

**[Low-pressure] electron-emission device.** S. RUBEN, ASSR. TO RUBEN PATENTS CO. (U.S.P. 1,799,645, 7.4.31. Appl., 27.11.26).—The cathode consists of a fused and cast mixture of  $\text{Fe}_3\text{O}_4$ ,  $\text{Cr}_2\text{O}_3$ , and  $\text{SrO}$ .

J. S. G. THOMAS.

**Electric-discharge devices.** SCHERING-KAHLBAUM A.-G. From C. SPAETH (B.P. 356,689, 3.3.30. U.S., 2.3.29).—A gas-filled discharge device comprising regulatable electrical means for evaporating into the gas, *e.g.*, Ne, and maintaining a supply of a monat. material, *e.g.*, Hg, from a side chamber away from the path of the main discharge, whereby the light emitted is maintained of any desired const. colour, is claimed.

J. S. G. THOMAS.

**Manufacture of electric incandescence lamps etc.** L. D. LOCKWOOD, ASSR. TO WESTINGHOUSE LAMP CO. (U.S.P. 1,795,785, 10.3.31. Appl., 4.2.29).—Oxidation of enclosed metal parts of the lamp is prevented by introducing into the envelope during the baking process a reducing gas, *e.g.*, a mixture of  $\text{N}_2$ ,  $\text{H}_2$ , and CO prepared from water-gas, which is reactive below 450°.

J. S. G. THOMAS.

**[Electrode for] photoelectric cells.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 356,895, 5.8.30. Holl., 26.2.30. Addn. to B.P. 319,650; B., 1930, 775).—An alkali metal, *e.g.*, Cs, deposited upon a unimol. layer of a compound, preferably an alkali-metal oxide, *e.g.*,  $\text{Cs}_2\text{O}$ , is claimed.

J. S. G. THOMAS.

**[Internal coating for] photoelectric cells.** STANDARD TELEPHONES & CABLES, LTD. From MATÉRIEL TÉLÉPHONIQUE (B.P. 356,802, 12.6.30).—Alkali metal, *e.g.*, K, introduced into the interior of the wall of the bulb by electrolysis of a molten alkali-metal salt contained within the bulb immersed in a bath of similar molten salt, is liberated from the wall on to the internal

surface of the bulb by reversing the direction of electrolysis. Preferably, the internal surface of the wall is coated with Mg by evaporating Mg in an atm. of  $\text{H}_2$  within the bulb, before the direction of electrolysis is reversed.

J. S. G. THOMAS.

**[Gas filling for] photoelectric cells.** GRAMOPHONE CO., LTD., and W. F. TEDHAM (B.P. 356,747, 5.6.30).—The filling consists of Xe and/or Kr, at a pressure of 80  $\mu$  or less of Hg.

J. S. G. THOMAS.

**Effecting chemical reactions between gases.** F. LECHLER, ASSR. TO INTERNAT. PRECIPITATION CO. (U.S.P. 1,796,110, 10.3.31. Appl., 13.9.27. Ger., 24.11.26).—Apparatus for passing gases through a silent electric discharge and in contact with a catalyst, and for maintaining such a relation between the direction of the discharge and the position of the catalyst that the electric wind due to the discharge tends to move the gases into contact with the catalyst, is claimed.

J. S. G. THOMAS.

**Electrical precipitators [for separation of suspended particles from gases].** H. A. WINTERMUTE and C. W. J. HEDBERG, ASSRS. TO RESEARCH CORP. (U.S.P. 1,798,511 and 1,798,964, 31.3.31. Appl., [A] 4.5.26, [B] 1.5.29).—(A) The apparatus comprises a collector electrode assembly of the graded-resistance type composed of parallel plates with embedded rods the ends of which project beyond the vertical ends of the plates, and an end wall at each side of the group of parallel plates and co-operating therewith to define a number of gas passages. (B) Gases are subjected to the action of the electric discharge, first in the absence of electrode-flushing liquid and then with the collecting electrodes flushed with  $\text{H}_2\text{O}$ . A form of collecting electrode and supports therefor are claimed.

J. S. G. THOMAS.

**Protection of electrical apparatus.** M. BUCHHOLZ (U.S.P. 1,796,746, 17.3.31. Appl., 29.10.27. Ger., 2.11.26).—Gases or vapours produced by decomp. of insulating material diffuse through a porous wall into a chamber separated from the apparatus to be protected, and create therein a change of physical conditions, *e.g.*, of temp. or pressure, whereby an electrical circuit including an alarm or cut-out is closed.

J. S. G. THOMAS.

**[Cathode-ray] electric-discharge tubes.** TELEFUNKEN GES. F. DRAHTL. TELEGRAPHIE M.B.H. (B.P. 356,978, 22.10.30. Ger., 9.11.29).

**High vacua in valves.**—See I.  $\text{C}_2\text{H}_2$ . Boilers for tar etc.—See II.  $\text{NH}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ .—See VII. Apparatus for making glass. Insulators for sparking plugs.—See VIII. Magnetic alloys. Alloys for cables. Coating metals with bronzes. Cr- and Al-plate.—See X. Irradiation of milk. Preservation of substances.—See XI. Synthesis of vitamins.—See XX.

## XII.—FATS; OILS; WAXES.

**Cacao butter.** VIII. Use of interferometry in fat analysis. H. P. KAUFMANN (Chem. Umschau, 1931, 38, 265—267; cf. B., 1931, 934).—Interferometric measurements emphasise small differences in  $n_D$ . Pressed cacao butters ( $n_D^{20}$  1.4570—1.4575) showed differences of —50 to +46 scale divisions from an arbitrary zero val.



("Stollwerk" expressed fat). Extracted fats (cf. B., 1931, 123) gave readings +430 to +512 and mixtures of extracted and expressed fats (having normal chemical characteristics) +80 to +410. Although useful, the test cannot therefore detect with certainty small additions of extracted fat to pressed fats with vals. near the upper limits. E. LEWKOWITSCH.

**Crismer's index and the critical temperature of solubility of fats in alcohol of varying concentration.** E. DE'CONNO and L. FINELLI (Annali Chim. Appl., 1931, 21, 365—373).—The Crismer index must be determined with EtOH the sp. gr. of which has been determined by means of the crit. temp. of solubility, using a mixture of mineral oil and vaseline. The correction to be applied for obtaining the correct Crismer index with EtOH of varying concentration is not a const., but is a variable which is not proportional to the % of  $H_2O$ . The validity of the correction proposed by Crismer for the acidity of the fat is confirmed. O. F. LUBATTI.

**Factors influencing the choice of textile soaps.** W. GARNER (Ind. Chem., 1931, 7, 409—413).—The technical properties of soaps, sulpho-compounds, etc. are discussed. E. LEWKOWITSCH.

**Analysis of refined [olive] oils. II. Second-treatment oils.** G. MAROGNA (Ann. R. Staz. Chim. Agrar. Sperim., 1931, Pubbl. No. 277, 329—348).—Fifteen "sansa" or second-treatment olive oils have been analysed. In general, their sp. gr. exceeds that of expressed oil; the refractometer reading is often above 63 or, for superior qualities, 64 and the Tortelli thermo-sulphuric val. is frequently above 50. The Blarez test for arachis oil mostly results in a cryst. and often partly gelatinous ppt., which adheres to the glass vessel. With the Bellier test for arachis oil a pulverulent cryst. ppt. is formed at temps. above 18° and a persistent opalescence above 40°. The I and sap. vals. are near the lower limiting values for the expressed oils and unsaponifiable matter is in larger proportion. Tests of general character, based on treatment of the oils with strong acids, give the same indications as mixtures of olive and seed oils. The Fachini-Morawski test is of value for the identification of extracted oils, either alone or mixed in large proportion with expressed oils, but becomes less definite for small proportions of the extracted oils. Under Wood's light most second-treatment oils show bluish colour and fluorescence, but refined expressed oils cannot be distinguished from refined second-treatment oils in this way. T. H. PORR.

**Cooking of tung oil.** E. FONROBERT and C. BOLLER (Farben-Ztg., 1931, 36, 2196—2197, 2239—2240; 37, 15—17).—In an investigation of the suitability of tung oils for use in varnish, the parabolic relation between temp. and time of gelation showed that below 200° and above 280° gelation is excessively slow and rapid, respectively. Samples of tung oil were therefore heated to gelation point under standard conditions at 10° intervals between these two temp., portions being withdrawn from the bulk at appropriate intervals during the heating at each temp. and tested for sp. gr.,  $n_D$ , and viscosity. Tabulated results are given and graphed against time. The "Albertol number," a new

criterion devised for tung oil stand oil, is the wt.-% of Albertol 111L that has to be added to the oil (to which 0.1% of Co as resinate has been added) in order to give a non-frosting film when stoved in the oven described in British Standard Specification 256. This test, when applied to the above oils, showed that the most desirable oil from the point of view of varnish making is one obtained by brief heating at 280°. S. S. WOOLF.

**Composition of Philippine kapok-seed oil.** A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1931, 46, 131—137).—Pods of *Ceiba pentandra*, Gaertner, consist of 17% of a superior floss and 32% of seeds, yielding about 25% of oil on pressing. The clear oil obtained (after treatment with kieselguhr, Suchar, and talc powder) had  $d_4^{20}$  0.9109,  $n_D^{20}$  1.4678, I val. (Hanus) 95.6, sap. val. 192.1, unsaponifiable matter 0.78% (I val. 82.4), acid val. 7.39, saturated acids (Pb salt-Et<sub>2</sub>O method) 18.64% (corr.), unsaturated acids 75.71% (corr.) (I val. 123.9). The composition of the oil is given as: acids %: myristic 0.46, palmitic 15.17, stearic 2.23, arachidic 0.79, oleic 47.68, linoleic 28.03. The Halphen reaction is positive.

E. LEWKOWITSCH.

**Changes in dispersions of sulphonated oils on heating.** W. SCHINDLER and E. RÖMER (Collegium, 1931, 349—358).—5% emulsions of a sulphonated fish oil remained stable even after heating for 1 hr. at 100°, but 12% emulsions were completely broken down. Emulsions of 2 pts. of sulphonated oil and 1 pt. of mineral oil were stable to heat at all concentrations. Stability to heat was increased by adding  $NH_3$  to the oil or the  $H_2O$ , but the dispersed oil was not so fine when  $NH_3$  and oil were emulsified as with the oil alone. The greatest degree of dispersion was produced by oil in dil. aq.  $NH_3$ , but not by mixtures of mineral and sulphonated oils. Dispersions of a highly sulphonated castor oil were only slightly affected by heat, but dispersions of this with mineral oil were stable to heat treatment only at considerable dilution. A lightly sulphonated oil (neatsfoot) was very sensitive to heat; a 5% emulsion separated at 60°. In some cases the degree of dispersion and heat-stability were diminished by adding  $NH_3$ . A greater improvement was produced by mixing the  $NH_3$  with the oil than by dispersing the oil in dil. aq.  $NH_3$ . D. WOODROFFE.

**Coconut oil industry.** A. E. WILLIAMS (Engineering, 1931, 132, 481—484).

**Petroleum soap.** Naphthenic acid soaps.—See II. CO from paint.—See XIII. Fat of frozen bacon. Rancidity changes in fats. Determining fat in ice cream.—See XIX.

#### PATENTS.

**Apparatus for treating soya beans.** T. SATOW (U.S.P. 1,799,256, 7.4.31. Appl., 23.1.26).—In order to preserve the proteins in the cake, the (cracked) seed is dried by hot air until the  $H_2O$  content is reduced to 12.5—13% before extracting with light petroleum at not above 45° in a steam-heated rotary drum (details given). The solvent is removed from the extracted meal by hot dry air, the temp. being raised to 115—120°.

E. LEWKOWITSCH.

**Oleic acid [*n*-butyl] ester.** W. J. BANNISTER, ASSR. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,796,231, 10.3.31. Appl., 16.5.30).—Oleic acid is esterified with *n*-BuOH in the presence of a catalyst, preferably  $\text{H}_3\text{PO}_4$ . The product is purified by washing, distillation, etc. *n*-Bu oleate has boiling range (10–18 mm. Hg) 235–245°, f.p. –12° (opaque) or –26.4° (solid),  $d_{20}^{20}$  0.864,  $I_{\text{val}}$  58.3,  $s_{\text{ap. val}}$  134.0. E. LEWKOWITSCH.

**Improved soap.** D. DE NAGY (B.P. 356,847, 27.6.30).—The mash prepared by treating potatoes with dil.  $\text{H}_2\text{SO}_4$ , pressing, and neutralising with dil. alkali is boiled with (15%) alkaline lye until it becomes saponaceous; a small amount (e.g., 8%) of palm oil and more lye containing 5% of  $\text{NEt}_4\text{OH}$  are added and saponification is completed by further boiling. Casein and aq.  $\text{NH}_3$  may be added. E. LEWKOWITSCH.

**Lubricants. Slushing grease.**—See II. Washing etc. agents.—See III. Material for coating paper.—See V. Castor oil condensation product.—See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**"Chalking" of paint [films].** C. P. VAN HOEK (Farben-Ztg., 1931, 36, 737–738).—Wagner's views on the cause of chalking are criticised. The assumption of a mechanical effect due to pigment-rich surface layers is considered untenable, the existence of such homogeneous strata (postulated also by Wolff and Blom) being queried. The non-chalking of aluminium paint is instanced in this connexion. The author attributes chalking entirely to weathering of the binding vehicle, and denies that chalking can be regarded in any case as a source of durability, on the grounds that a chalking surface is always water-absorbent etc. He further disagrees with Wagner on the relation of "wetting of pigments" to chalking, and instances the chalking of oleophilic white lead and the non-chalking of the more oleophobic zinc oxide as arguments against the view that a well-wetted pigment will not yield a chalking surface layer. The effect of ultra-violet light is also discussed (cf. B., 1931, 259). S. S. WOOLF.

**Determination of carbon monoxide produced from paint in confined spaces.** F. H. NEWINGTON (J.S.C.I., 1931, 50, 371–375 r).—Following a fatal accident in a "bulge" on board one of H.M. ships, an investigation was made into the possibility of the presence of CO in the atm. of confined spaces generally on board ships. An apparatus was devised for the determination of small amounts of CO by the  $\text{I}_2\text{O}_5$  method which could be used *in situ*. Appreciable amounts, reaching a concentration of 0.2%, were found, particularly in freshly painted spaces, and subsequent investigation showed that the CO was derived from the linseed oil of the paint during drying. The atm. in a sealed empty drum which had contained boiled linseed oil was found to contain over 0.3% CO and to be almost entirely deficient in  $\text{O}_2$ .

**Aluminium stearate in paint technology.** H. WOLFF and J. RABINOWITZ (Farbe u. Lack, 1931, 36, 428–429).—A general account is given of the functions of Al stearate (a) as a pigment-suspending agent, e.g., for retarding the setting of  $\text{Pb}_3\text{O}_4$ , (b) in reducing the oil absorption of pigments, leading to improved rust-resist-

ance of paints made therefrom (particularly if the stearate be mixed with a little white spirit before incorporation with the pigment), and (c) as a "matting" agent in varnish, in which connexion, although an improvement in the  $\text{H}_2\text{O}$ -resistance of an ester gum varnish was noted, statements that Al stearate improves varnishes containing acid resins, e.g., rosin, were not confirmed. S. S. WOOLF.

**Weatherproof paints.** W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1931, 36, 2084–2085).—The use of paints based on  $\text{Na}_2\text{SiO}_3$  is adversely criticised. The disadvantages of such paints include poor resistance to the acids in the air of industrial regions, presence of  $\text{CaCO}_3$  as usual pigment for cheapness, restriction of range of coloured pigments that may be used owing to the alkalinity of the vehicle, tendency to bloom, etc. S. S. WOOLF.

**Evaluation of "chalking" of paint [films].** R. KEMPF (Farben-Ztg., 1931, 36, 2158–2159).—A reply to Wolff's recent criticism (B., 1931, 895) of the evaluation of chalking data by integration of results. Whilst the limitations of the procedure are admitted, it is pointed out that although chalking is a continuous process the main effects are seasonal and the time intervals used in Kempf's current experiments are short enough to enable any significant changes to be recorded. The relations between loss of wt. and degree of chalking are also briefly discussed. S. S. WOOLF.

**Comparative investigation of driers.** ANON. (Farben-Ztg., 1931, 36, 2200–2201).—A summary is given of a publication by H. WOLFF on the behaviour of driers, with special reference to the different forms in which they are encountered, e.g., resinates, linoleates, "soligenates," the last-named showing advantages particularly over resinates. The drying action on linseed oil under various conditions of humidity and temp., the alteration of drying power with storage of the "boiled oil," the effect on stand oil, the influence of light irradiation, the effects of driers on swelling and the mechanical properties of the films, etc. receive consideration. S. S. WOOLF.

**Ink tests.** O. MEZGER, H. RALL, and W. HEES (Z. angew. Chem., 1931, 44, 645–651).—For the detection of Cr in ink-writing the latter is destroyed with NaOCl solution containing  $\text{BaCl}_2$ , whereby  $\text{BaCrO}_4$  is deposited on the paper in place of the ink marks; treatment of the paper with a solution of diphenylcarbazine and HCl in EtOH restores the writing in a violet tint. Cl' is detected in the ink by treating the writing with  $\text{HNO}_3$  containing  $\text{AgNO}_3$  and  $\text{NaNO}_2$  or  $\text{KMnO}_4$  to destroy the colouring matter; after washing with dil.  $\text{HNO}_3$ , the paper is immersed in alkaline  $\text{CH}_2\text{O}$  or in alkaline  $\text{Na}_2\text{S}_2\text{O}_4$ , when the  $\text{AgCl}$  first formed is reduced to black Ag. A similar method is used for detecting  $\text{SO}_4^{2-}$  by converting it into  $\text{PbSO}_4$  and then into PbS. The Fe compounds may be removed from ink-writing by treatment with HCl and  $\text{BaCl}_2$ ; the  $\text{BaSO}_4$  formed by the  $\text{SO}_4^{2-}$  which is always present in the ink or paper adsorbs the dye, whilst the Fe is removed by the acid and the nature of the dye can then be detected by appropriate tests. By making the chloride test on old writing it is possible to obtain a good idea of the age of the writing,

as the chloride slowly diffuses with age away from the writing until eventually it penetrates right through the paper. Sulphates behave in a similar manner.

A. R. POWELL.

#### Incorporation of dry pigments into the medium.

A. W. C. HARRISON (J. Oil Col. Chem. Assoc., 1931, 14, 162—171).—Media are classified and the characteristics of each group outlined. The importance of using linseed oil with a degree of free acidity to suit the type of pigment paste being prepared is emphasised. Composite media should be mixed and stored for a considerable period before use, and should be so compounded as to give exactly the desired qualities with pigments usually incorporated in it. Methods of mixing are discussed and the principal properties of dry constituents enumerated.

F. C. HARWOOD.

#### Lacquers for aircraft and their testing.

O. MERZ (Farben-Ztg., 1931, 36, 2156—2158, 2197—2199).—The requirements of such lacquers and the respective merits of cellulose acetate and nitrate are summarised, mention also being made of the use of cellulose ethers for this work. Details of the cellulose esters, plasticisers, solvents, diluents, and pigments used and typical formulations are given. The physico-mechanical testing of these lacquers is described in full, testing of films on glass, general suitability for "doping" including wt. applied, tensile strength, elasticity, burning tests, durability, all being considered. Details of application of the above lacquers and the use of oil varnishes as "dopes" are also included.

S. S. WOOLF.

#### Acid-resistance of varnishes on phosphatised iron.

F. KOLKE (Farben-Ztg., 1931, 36, 2235—2238).—Normal and phosphatised Fe plates coated with each of 6 lacquers (3 PhOH-CH<sub>2</sub>O synthetic resin varnishes, shellac, bitumen, and cellulose ester—all except the last-named being stoved) were immersed in 3% H<sub>2</sub>SO<sub>4</sub>, HCl, AcOH, and H<sub>3</sub>PO<sub>4</sub>, and observed after 1, 2, 3, 5, and 7 days. The tabulated results and photographs of the panels after 7 days are given. With the exception of AcOH, the acid-resistance of the phosphatised panels is not higher, but generally lower, than that of the unphosphatised panels. As phosphate treatment (*e.g.*, "Parkerising," "Bonderising," "Atrament" process [I.G.]) improves durability as regards normal weathering, resistance to H<sub>2</sub>O and sea-water, etc., this result is considered to be due to the sp. action of the acids on the phosphate coatings.

S. S. WOOLF.

**Cooking of tung oil.**—See XII. **Pine-needle oil.**—See XX. **Storage of nitrocellulose.**—See XXII.

#### PATENTS.

**Preparation of solutions of cellulose esters containing insoluble pigments.** G. B. ELLIS. From Soc. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 356,740, 9.5.30).—Finely-ground pigments suspended in a "homogenising liquor," *e.g.*, a solvent mixture containing, if desired, a small amount of cellulose acetate, are forced under pressure and at high speeds, *e.g.*, 50—300 m./sec., through one or more narrow orifices, further quantities of cellulose acetate etc. being subsequently added for conversion of the intermediates into varnishes, films, or filaments.

S. S. WOOLF.

**Manufacture of destructively distilled castor oil condensation product.** E. C. HOLTON, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,799,420, 7.4.31. Appl., 14.12.28).—Castor oil is destructively distilled (under reduced pressure if desired) down to 60—80% by wt. of the charge, esterified with excess of glycerin, and condensed with phthalic anhydride to yield a clear homogeneous product suitable for use in varnishes etc.

E. LEWKOWITSCH.

#### Refining of natural [wood] and artificial resin.

W. SCHULTZE, Assr. to A. SCHULTZE & Co. OELFABR., and H. SCHUBERT, TEXTILWERKE (U.S.P. 1,793,967, 24.2.31. Appl., 7.11.27. Ger., 23.9.27).—Pale resin is obtained by distilling (dead) wood rosin in a high vac. (8 mm. pressure or less); 80—85% of the distillate (yield 80%) is obtained at 230—255°.

E. LEWKOWITSCH.

#### Production of resin-acid derivatives.

CHEM. FABR. DR. K. ALBERT G.M.B.H. (B.P. 356,742, 4.6.30. Ger., 19.8.29).—Abietic (resin) acid or one of its pyro- or halogenated or oxidised derivatives is converted into the anhydride (by treatment with Ac<sub>2</sub>O) and this is treated with alcohols, phenols, amides, amines, or esters to yield abietic esters, amides, etc. The derivative formed is separated from the equiv. abietic acid regenerated by distillation or washing processes etc.

E. LEWKOWITSCH.

#### Derivatives of polyhydric alcohol-polybasic acid condensation products.

IMPERIAL CHEM. INDUSTRIES, LTD., and W. BAIRD (B.P. 356,738, 6.5.30).—Resins of the glycerol-phthalic anhydride type (prepared at temp. >150° with or without a fatty ingredient, and having an acid val. >30) are treated with aq. NH<sub>3</sub> or its substitution derivatives, *e.g.*, triethanolamine, piperidine; the products give clear aq. solutions which dry to adherent films on stoving at 100°.

E. LEWKOWITSCH.

**Laminated products.** BAKELITE CORP., Assees. of R. P. COURTNEY (B.P. 356,769, 7.6.30. U.S., 8.6.29).—A resinoid composition sheet is bonded to a base material, *e.g.*, a metal, by a rubber cement obtained, *e.g.*, by heating rubber with 4—5% of conc. H<sub>2</sub>SO<sub>4</sub> or an equiv. amount of a sulphonic acid derivative for 4—10 hr. at 120—140°, or by treating rubber with a PhOH-oil condensation product partly advanced to the infusible condition.

S. S. WOOLF.

**Water paints [for cement, stucco, etc.].** A. E. WHITE. From J. V. REARDON (B.P. 357,119, 14.3.30).—See U.S.P. 1,749,923; B., 1930, 726.

**[Apparatus for] manufacture of moulded articles from synthetic resin and like materials.** M. M. EATON and J. C. VREDENBURG (B.P. 357,194, 20.3.30).

**Thermoplastic sheets. Sandpaper.**—See V. **Impregnated fabric.**—See VI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Strontium sulphate as a rubber filler.** J. R. SCOTT (India-rubber J., 1931, 82, 442—444).—Powdered natural SrSO<sub>4</sub> (celestine) gives vulcanised rubber mixings of higher tensile strength, greater extensibility, and better resilience and permanent set than powdered barytes and approximating to those yielded by precipitated BaSO<sub>4</sub>. The ageing qualities of the rubber

containing  $\text{SrSO}_4$  are better than those of the rubber with either form of  $\text{BaSO}_4$ . The  $n$  of  $\text{SrSO}_4$  is nearer than that of barytes to that of rubber and its pigmenting val. is less.

D. F. TWISS.

**Reclaimed rubber.** A. T. McPHERSON (U.S. Bur. Standards, Circ. 393, 1931, 22 pp.).—A review.

## PATENTS.

**Manufacture of rubber.** GOODYEAR TIRE & RUBBER Co. (B.P. 356,933, 3.9.30. U.S., 5.10.29).—Rubber with good ageing qualities is obtained by vulcanisation in the presence of a substance of the formula  $\text{S}_2\langle\frac{\text{R}}{\text{R}'}\rangle\text{NII}$  in which R and R' are  $\text{C}_6\text{H}_4$  or  $\text{C}_{10}\text{H}_6$  nuclei, *e.g.*, dithiophenyl- $\beta$ -naphthylamine, m.p.  $140^\circ$  (decomp.).

D. F. TWISS.

**Improvement of age-resisting properties of rubber and rubber-like substances.** IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, and F. J. SIDDLE (B.P. 356,923, 27.8.30).—The age-resisting properties are improved and vulcanisation is facilitated by incorporating an  $\text{NH}_2$  derivative of a diaryl oxide, *e.g.*, 4:4'-diaminodiphenyl ether, with or without the additional presence of other substances possessing anti-ageing properties.

D. F. TWISS.

**[Softening] treatment of rubber stock.** H. L. MORAN (U.S.P. 1,790,875, 3.2.31. Appl., 16.12.27).—The stock is softened preparatory to subsequent operations by subjecting it for a definite period to the proper degree of heat in a special tank of hot water through which it passes on a conveyor moving at the appropriate speed.

D. F. TWISS.

**Manufacture of rubber products.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 354,841, 17.5.30).— $\text{ZnO}$  produced from precipitated Zn compounds at a temp. below incandescence and containing a small proportion of  $\text{Al}_2\text{O}_3$ , *e.g.*, 1–6%, imparts high mechanical resistance in vulcanised rubber, particularly towards tearing.

D. F. TWISS.

**Treatment of rubber [for age-resistance].** W. S. CALCOTT and W. A. DOUGLASS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,790,794, 3.2.31. Appl., 17.9.29).—By the interaction of a primary arylamine,  $\text{CH}_2\text{O}$ , and  $\text{NaHSO}_3$  and treating the resulting product with a phenolic compound (cf. G.P. 109,498), a mixture is obtained of which the main constituent is a hydroxybenzylarylamine of the general formula  $\text{R}\cdot\text{NH}\cdot\text{CH}_2\cdot\text{R}'\cdot\text{OH}$ , where R is an aryl group which may contain an alkyl substituent, and R' is an aryl group which may contain an alkyl or alkoxy-substituent. Examples of the prime products, which are effective antioxidants, are *p*-hydroxy-*m*-methoxybenzylaniline and *p*-hydroxybenzyl- $\beta$ -naphthylamine.

D. F. TWISS.

**Age-resisting vulcanised rubber.** W. SCOTT Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,792,042, 10.2.31. Appl., 23.9.29).—The S derivatives of the reaction product of a ketone and a naphthylamine are used as antioxidants for rubber. *E.g.*, the acetone- $\alpha$ -naphthyl obtained from  $\text{COMe}_2$  and  $\alpha$ -naphthylamine in the presence of I is heated with an equimol. proportion of S at  $150^\circ$  and a little I; the antioxidant product is a hard black resin.

D. F. TWISS.

**Rubber composition and preservation of rubber.** M. C. REED, Assr. to B. F. GOODRICH Co. (U.S.P. 1,793,635, 24.2.31. Appl., 5.4.30).—The *as*-diarylhydrazines, *e.g.*, *as*-diphenylhydrazine, are applied as antioxidants for rubber.

D. F. TWISS.

**Vulcanisation of rubber.** L. B. SEBRELL and D. N. SHAW, Assrs. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,792,770, 17.2.31. Appl., 18.11.24).—Vulcanisation is accelerated by amidoximes and their aldehyde derivatives; examples cited are *o*-tolylamidoxime,  $\text{Me}\cdot\text{C}_6\text{H}_4\cdot\text{C}(\text{NH}_2)_2\cdot\text{NOH}$ , and the ethylidene derivative, m.p.  $125^\circ$ , of *p*-tolylamidoxime.

D. F. TWISS.

**Vulcanisation of rubber.** A. A. SOMERVILLE, Assr. to R. T. VANDERBILT Co. (U.S.P. 1,791,876, 10.2.31. Appl., 30.12.27).—Scorching or premature vulcanisation of rubber mixtures containing ultra-accelerators is prevented by the presence of a small proportion (*e.g.*, 1% on the rubber) of a suitable peroxide such as  $\text{PbO}_2$  or  $\text{BaO}_2$ .

D. F. TWISS.

**Rubber vulcanisation accelerator.** W. SCOTT, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,792,041, 10.2.31. Appl., 22.5.25).—Vulcanisation is accelerated by the reaction product of  $\text{CH}_2\text{O}$  and crotonaldehydedianiline.

D. F. TWISS.

**Vulcanisation accelerators [for rubber] and application thereof.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 353,580, 17.5.30).—Diarylenethiazyl monosulphides obtained by action of 2-chloroarylenethiazoles on the alkali salts of 2-mercaptoarylenethiazoles are valuable vulcanisation accelerators and give particularly good results when used in conjunction with a basic org. accelerator such as diphenylguanidine.

D. F. TWISS.

**Recovery of waste materials [containing rubber].** E. WOOD (B.P. 355,871, 23.6.30).—Rubberised materials such as motor tyres are disintegrated dry, *e.g.*, in a mill provided with revolving hammers; the cotton and rubber scrap are then separated from each other by screening or by means of a centrifugal machine.

D. F. TWISS.

**Coated fabric.**—See VI. Laminated products.—See XIII.

## XV.—LEATHER; GLUE.

**Evaluation of bating materials.** A. KÜNTZEL and B. POTOTSCHNIG (Collegium, 1931, 475–495).—Nine commercial bating materials were compared by seven different methods of testing, *viz.*, Fuld-Gros, Boidin, Löhlein-Volhard, Schneider-Ulček, gelatin m.-p. determination, Willstätter-Waldschmidt-Leitz, and the EtOH titration of the collagen decomp. products. The results obtained agreed only where the same substrate was used. Collagen and gelatin acted as similar substrates, but were not completely replaceable by casein or milk. A method based on a collagen or gelatin substrate should be used. The casein-substrate methods are simpler and more exact than the others and the Löhlein-Volhard method (Oppenheimer, "Fermente," 4, 119) is better than the Fuld-Gros or Schneider-Ulček methods.

D. WOODROFFE.

**Fat-liquoring [of leather].** W. SCHINDLER and K. KLANFER (Collegium, 1931, 339–349; cf. B., 1928,

763).—Only part of the fat absorbed by different pieces of chrome leather fat-liquored with sulphonated castor, cod, and neatsfoot oils, respectively, was extracted with  $\text{CCl}_4$ . The residue varied directly as  $(\beta + \delta)$  in the different sulphonated oils. Most of the fat was extracted with  $\text{CCl}_4$  from leathers which had been fat-liquored with soap fat-liquors. The unextracted fat was recoverable in every case by decomposing the leather with conc.  $\text{NaOH}$ . Misleading results are obtained if the fat contents of chrome leathers are judged by the amount extracted with  $\text{CCl}_4$ . A portion of the combined  $\text{SO}_4$  in chrome leather, which had previously been neutralised with  $\text{NaHCO}_3$ , was always removed during fat-liquoring, but some of the combined  $\text{SO}_4$  in the sulphonated oil used combined with the leather and could be only partly removed by treatment with a boiling conc. solution of  $\text{NaHCO}_3$ . At higher  $p_{\text{H}}$  the  $\text{SO}_4$  lost by the leather greatly exceeded that gained from the fat-liquor. Relatively more  $\text{SO}_4$  was removed from the leather by sulphonated oils than would be anticipated from their composition. A commercial sulphonated oil, which was very acid when stable, removed a large amount of  $\text{SO}_4$  at  $p_{\text{H}}$  4.2. There was no evidence of any hydrolysis of the sulphonated fat esters.

D. WOODROFFE.

**Analytical chemistry of chrome [tanning] liquors.** F. L. HAHN (Collegium, 1931, 429—438).—A special pipette for determining their sp. gr. is described.  $\text{Br-NaOH}$  is recommended for oxidising the chrome liquor in the Cr determination. The excess of  $\text{NaOBr}$  is decomposed with thiocyanate. The colorimetric determination of Cr in the oxidised solutions is discussed. For this determination, a portion of the chrome liquor is treated with  $\text{HNO}_3$  and  $\text{AgNO}_3$ ,  $\text{K}_2\text{S}_2\text{O}_8$  is added, the mixture heated on the water-bath, cooled, made up to 100 c.c.,  $\text{NH}_3$  or  $\text{HNO}_3$  added to produce a pure yellow colour, and the Cr determined colorimetrically. The  $\text{SO}_4$  in  $\text{Cr}(\text{SO}_4)_3$  liquors is not completely titratable in the usual acid determination, so that the figure is merely a control.

D. WOODROFFE.

**Chrome tanning. X. Glucose-chrome liquors.** E. STIASNY and M. ZIEGLER. **XI.** E. STIASNY and E. GERGELY. **XII. Composition of chromium tanning extracts.** E. STIASNY, E. GERGELY, and A. DEMBO (Collegium, 1931, 438—444, 444—453, 458—474; cf. B., 1930, 158).—X. More glucose is required when  $\text{H}_2\text{SO}_4$  is run into  $\text{Na}_2\text{Cr}_2\text{O}_7$ -glucose mixture than when glucose solution is added to  $\text{Na}_2\text{Cr}_2\text{O}_7$ - $\text{H}_2\text{SO}_4$ , and still more if  $\text{Na}_2\text{Cr}_2\text{O}_7$  solution is run into glucose- $\text{H}_2\text{SO}_4$  in order to reduce  $\text{Cr}^{\text{VI}}$  to  $\text{Cr}^{\text{III}}$  in each case. Only volatile acids are formed when glucose solution is added to  $\text{Na}_2\text{Cr}_2\text{O}_7$ - $\text{H}_2\text{SO}_4$  solution, the yield increasing as the amount of added glucose is increased. A proportion of oxalic acid is formed when  $\text{H}_2\text{SO}_4$  is run into a  $\text{Na}_2\text{Cr}_2\text{O}_7$ -glucose solution or when  $\text{Na}_2\text{Cr}_2\text{O}_7$  solution is added to glucose- $\text{H}_2\text{SO}_4$ . More volatile acids are formed by adding the  $\text{H}_2\text{SO}_4$  to the other ingredients than by the other methods of reduction. A greater amount of glucose containing dextrin is required to effect the reduction than of dextrin-free glucose.

**XI.** The nature of the compounds formed when solutions of  $\text{Na}_2\text{Cr}_2\text{O}_7$  are reduced with  $\text{SO}_2$  varies with the concentration of the solution.  $\text{Cr}_2(\text{S}_2\text{O}_6)_3$  was the chief

product in dil. solution, whereas in highly conc. solutions of  $\text{Na}_2\text{Cr}_2\text{O}_7$ , the product consisted almost entirely of "olated" dihydroxo-trisulphato-chromate, which was converted on heating or ageing the dil. solution into Cr sulphates with uncharged or cationic Cr complexes. The tanning properties of such liquors depend largely on their method of manufacture and the previous history of the liquor.

**XII.** Anionic Cr complexes are invariably formed when the Cr-tanning liquors are conc. and solid products are prepared therefrom. Their properties depend on the no. of OH and  $\text{SO}_4$  groups in the complex as well as on the nature of the cation. The  $\text{SO}_4$  is masked when a solution of  $\text{K}_2\text{SO}_4$  and chrome alum is conc. and allowed to crystallise, its precipitation figure with  $\text{NH}_3$  then being  $\infty$ ; the solubility of the product in  $\text{H}_2\text{O}$  and  $p_{\text{H}}$  of its solution diminished as the solution was aged before evaporation. The composition and properties of the resultant Cr-tanning liquors were affected by the method used for dissolving the solid extracts, ageing, dilution, and by the duration of the heating if they were dissolved hot. These factors affected the migration of the  $\text{SO}_4$  radicals from the Cr complexes. Better tannage and penetration were obtained by use of cold-prepared basic Cr-alum liquor than with a solution of the derived solid product.

D. WOODROFFE.

**Effect of synthetic tannins on the detection of sulphite-cellulose waste-liquor in vegetable tanning extracts by the [Procter-Hirst] aniline-hydrochloric acid, cinchonine, and fluorescence tests.** Committee Report. O. GERNGROSS and H. HERFELD (Collegium, 1931, 524—538).—1% of sulphite-cellulose waste-liquor can be detected in a vegetable tannin extract by the Procter-Hirst reaction, 3% by the cinchonine test, but not less than 10% by the fluorescence test. Positive reactions are given by all three tests in presence of synthetic tans. or if the extract has been sulphited in presence of sawdust, but not by sulphiting in presence of phlobaphens or resins. A min. of 5% of sulphite-cellulose waste can be detected by the Procter-Hirst reaction if the extract has been made from damaged or decayed material.

D. WOODROFFE.

**Free [mineral] acids in the analysis of vegetable [tanned] leathers. II, III.** V. KUBELKA and K. ZIEGLER (Collegium, 1931, 544—550, 550—557; cf. B., 1931, 819).—II. Slightly more acid was extracted by the continuous extraction of 20 g. of leather with 1 litre of  $\text{H}_2\text{O}$  at 42° than by Innes' method, but the "difference figure" was not materially changed. Not only this figure, but also the actual  $p_{\text{H}}$  of the original solution should be considered, since strong acids can be present only if the difference figure exceeds 0.7 and the  $p_{\text{H}}$  of the original solution is  $< 3$ . When leathers were treated with known amounts of different acids, a portion of the acid was always neutralised by the leather. The aq. extracts of all leathers to which  $> 0.75\%$  of  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$  had been added were turbid and the turbidity increased as the amount of added acid was increased, probably due to decomp. of the leather substance. This turbidity of the aq. extract may be used as a criterion of the presence of an injurious amount of strong acid.

III. Inconcordant results are obtained by the Atkin-Thompson method and the three readings are often not on a straight line when the graph becomes an approximation. This method is of equal qual. val. with the Innes method. The Atkin-Thompson crit. figure 2.5 for the  $p_H$  at zero dilution is shown to be too low. Innes' method is more reliable. D. WOODROFFE.

**Decay of bookbinding leather.** R. W. FREY and I. D. CLARKE (J. Amer. Leather Chem. Assoc., 1931, 26, 461—482).—Bookbinding leathers were weakened by exposure to air and light, and pyrocatechol tannages were weakened more than pyrogallol tannages. The S content and Procter-Searle figures of the leathers were increased by such exposure. Least deterioration was shown by three leathers tanned with pyrogallol tannins. Bookbinding leathers should be rendered less porous to air and less absorptive to S compounds in the air by treatment with dressings. D. WOODROFFE.

**Effect of [sulphuric] acid on vegetable-tanned leather. Committee report.** T. BLACKADDER (J. Amer. Leather Chem. Assoc., 1931, 26, 482—490).—Vegetable-tanned leathers are hydrolysed by the  $H_2SO_4$  in them, thus increasing their content of  $H_2O$ -sol. N. Greater deterioration is found in leathers containing  $H_2SO_4$  at higher R.H. (85%). A discussion of the report is included. D. WOODROFFE.

**Combination of collagen with dyes.** G. A. BRAVO and F. BALDRACCO (Annali Chim. Appl., 1931, 21, 355—365).—Known amounts of 12 direct dyes of tested purity were dissolved in  $H_2O$  and measured portions (100—200 c.c.) were transferred to glass-stoppered 500-c.c. flasks. Hide powder (collagen), from three different sources, in amounts between 0.25 and 4.0 g. was added; after shaking, the powder was allowed to be acted on for 48 hr. The filtered liquid was then examined with a Pulfrich photometer to determine the residual concentration of the dye. The amount taken up by the collagen was determined by difference from the photometric examination of the original solution. It was confirmed that together with adsorption a true chemical reaction also occurs. From this reaction the equiv. wt. of collagen was found to be 740. O. F. LUBATTI.

#### PATENT.

**Chromiferous dye.**—See IV.

### XVI.—AGRICULTURE.

**Mechanism of the buffer action of soils.** P. B. MYERS and G. M. GILLIGAN (Delaware Agric. Exp. Sta. Tech. Bull., 1930, No. 11, 37 pp.).—Removal of bases from soil by electrodialysis results in a loss of buffer capacity, the  $[H^+]$  increasing steadily as the bases are removed. The  $[H^+]$  of the soil colloid is an index of the degree of saturation with bases. Acids added to soil react with the salts of colloidal acids. The free colloidal acids formed are sufficiently weak to permit only a slight change in reaction of the system. Acid added in excess of that required to react with the cations of the colloidal salts is very incompletely ionised through the agency of the common ion of the cryst. salts formed. The resulting change in  $p_H$  of the system is small.

A. G. POLLARD.

**Occurrence of nitrites in soils.** G. S. FRAPS and A. J. STERGES (Texas Agric. Exp. Sta. Bull., 1930, No. 412, 15 pp.).—Nitrites occur in soil cultures and field soils in amounts greater than is generally assumed, and may persist for several weeks even where no recent addition of N material has occurred. The presence of  $CaCO_3$ ,  $MgCO_3$ , and a  $H_2O$  content of approx. 50% of the max. capacity favour nitrite formation in soils.

A. G. POLLARD.

**Sulphur as a soil amendment.** G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1930, No. 414, 56 pp.).—The S contents of numerous soils and crops are recorded. Application of S to soil increased the intake of S by plants and in some cases of P, but not of K or N. Oxidation of S in soils had no effect on the availability of P or K, but increased the permeability to  $H_2O$ , particularly on black alkali soils.

A. G. POLLARD.

**Behaviour of colloidal humic-mineral complexes in an electric field.** C. ANTONIANI (Atti R. Accad. Lincei, 1931, [vi], 13, 524—526).—Soil treated with 5% HCl and extracted with 10% NaOH solution yielded a complex which, after purification with 5% KCl solution to remove clay, sedimentation, and electrodialysis, contained: org. matter 82.75,  $Fe_2O_3$  5.05,  $Al_2O_3$  4.35,  $SiO_2$  4.65, and  $P_2O_5$  2.90%. After being flocculated at the isoelectric point ( $p_H$  7.4), the complex was brought back to the disperse state by addition of NaOH and placed in an electric field (0.36 volt/cm.), anodic migration then occurring. Gradual increase of the  $[OH^-]$  resulted in increase of this migration to a max., followed by decrease to zero and by change to cathodic migration. Hence in alkaline dispersion the humic colloid, which is the essential protector of the complex, exerts its protecting action only over a definite range of  $[OH^-]$ . T. H. POPE.

**Mutual coagulation of colloids in the soil.** V. N. SIMAROV (Pochvovedenie, 1929, 24, Nos. 1—2, 22—77).—Mutual coagulation takes place between 0.577 and 1.227 mols.  $MnO_2$  per mol.  $Fe(OH)_3$ , between 2.757 and 3.452 mols.  $SiO_2$  per mol.  $Fe(OH)_3$ , or between 3.753 and 8.045 g. of Na-saturated soil per g. of  $Fe(OH)_3$ . There are two zones at which a stable mixture of the two colloids exists,  $Fe(OH)_3$  or the soil suspension being in excess. Mutual coagulation takes place between 0.085 and 0.447 mol.  $MnO_2$  per mol.  $Al_2O_3$ . The coagulation vals. of the sols are calc. in terms of another or of a combination. CHEMICAL ABSTRACTS.

**Rapid determination of organic carbon in soils.** E. BOTTINI (Annali Chim. Appl., 1931, 21, 374—387).—0.5 g. of dry soil is refluxed with 100 c.c. of 10% aq. KOH. After cooling, to 50 c.c. of the filtrate are added 50 c.c. of 0.1N- $KMnO_4$ , 50 c.c. of  $H_2O$ , and 4 c.c. of 25%  $H_2SO_4$ . The liquid is boiled for  $\frac{1}{2}$  hr., excess (50 c.c.) of 0.1N-oxalic acid is added, and, after boiling again, the excess of acid is back-titrated with 0.1N- $KMnO_4$ . The no. of c.c. of  $KMnO_4$  required multiplied by 2, called  $x$ , represents the amount of  $KMnO_4$  used up by the org. matter dissolved by KOH. The residue insol. in KOH remaining on the filter is transferred to a flask and treated with the same amount of reagents and in the same manner as the filtrate. The no. of c.c. of 0.1N- $KMnO_4$  used up is indicated by  $y$ . Then % org.

$C = 0.03 \times 2(x+y)$ . The approx. content of org. matter in the soil is obtained by multiplying the % C by the factor 2.3.

O. F. LUBATTI.

**Determination of carbon dioxide in soil carbonates. A modification of the official method.** W. M. SHAW (J. Assoc. Off. Agric. Chem., 1931, 14, 283—292).—In the official method ("Methods of Analysis," A.O.A.C., 1925, p. 24) aspiration for 30 min. at 4 litres per hr. is insufficient, but at 6 litres is sufficient to remove all the  $\text{CO}_2$  from the reaction flask, in the case of precipitated  $\text{CaCO}_3$ . Dolomite ground to 60—100-mesh requires aspiration for 2 hr. The superiority of Ascarite as absorbent for  $\text{CO}_2$  at the higher rate of flow is confirmed. The vitiating effect of org. matter on the determination when the decomp. is carried out at room temp. is completely removed by the addition of  $\text{FeCl}_2$  or  $\text{SnCl}_2$ . At  $100^\circ$  the effect of the org. matter is not entirely eliminated,  $\text{SnCl}_2$  being more effective than  $\text{FeCl}_2$ . The method described permits the rapid determination of carbonate- $\text{CO}_2$  in soils where an error of 0.01% may be tolerated, the sample being boiled with  $\text{SnCl}_2$ -HCl solution and the  $\text{CO}_2$  absorbed by Ascarite. W. J. BOYD.

**Neubauer's method as applied to the determination of the availability of phosphate materials.** S. F. THORNTON (J. Assoc. Off. Agric. Chem., 1931, 14, 292—295).—The availability of 13 phosphatic materials in both acid soil and neutral sand has been studied by an adaptation of the Neubauer method (B., 1929, 571). There is poor correlation with the results obtained by the Official neutral citrate method ("Methods of Analysis," A.O.A.C., 1925, p. 24), but pot tests show that the Neubauer method gives valuable indications as to the true availability of phosphates. W. J. BOYD.

**Neubauer's method for determining potash and assimilable phosphoric anhydride in soils.** M. SARTORI (Ann. R. Staz. Chim. Agrar. Sperim., 1931, Pubbl. No. 270, 249—256).—The soils examined were: red puzziolana, granular tufa, highly humic, CaO-rich, and CaO-free. In comparison with the results of cultural experiments on wheat and maize, the biological method gave satisfactory indications of the  $\text{P}_2\text{O}_5$  content for the first 3 soils and the citric acid method for the humic soil; the boiling conc.  $\text{HNO}_3$  method yielded discordant results for all the soils. Neubauer's and the citric acid methods for  $\text{K}_2\text{O}$  agreed with the cultural tests for all except the red puzziolana, with which the citric acid method was the best; for the humic, CaO-rich, and CaO-free soils the boiling conc.  $\text{HNO}_3$  method gave results differing from those of the biological or citric acid method. T. H. POPE.

**Mobility of soil-phosphoric acid; a consideration of Eschenhagen's work.** H. NEUBAUER (Z. Pflanz. Düng., 1931, 10B, 409—410; cf. B., 1931, 856).—The necessity of an analysis of seed used in control experiments in the seedling method for determining root-sol. P in soils is emphasised. A. G. POLLARD.

**Simple electroanalysis cell for routine determination of exchangeable bases in soils.** M. L. M. SALGADO and G. W. CHAPMAN (Soil Sci., 1931, 32, 199—215).—Convenient apparatus is described. Results are in close general agreement with those obtained by leaching with  $\text{N-NH}_4\text{OAc}$ . A. G. POLLARD.

**Determination of the degree of saturation of soils with bases.** R. H. WALKER, B. J. FIRKINS, and P. E. BROWN (Iowa Agric. Exp. Sta. Res. Bull., 1931, No. 139, 157—176).—Comparison is made of recognised methods for determining the replaceable bases in soils. Electrodialysis methods give irregular results where <100-g. samples are examined. No correlation exists between the replaceable  $\text{H}^+$  of soils (Hissink) and their  $\text{pH}$ . Parker's methods for determining the base-exchange capacity and replaceable  $\text{H}^+$  of soils gave more satisfactory results than any other methods examined.

A. G. POLLARD.

**Effect of drying and of ultra-violet light on soils.** A. E. MORTENSON and F. L. DULEY (Soil Sci., 1931, 32, 195—198).—The initial increase in the  $\text{NH}_3$  content of soils caused by drying or ultra-violet treatment is greater where these treatments are most severe. Subsequently the  $\text{NH}_3$  content falls to a very low value. Corresponding changes in the  $\text{NO}_3^-$  content are of the reverse order, the finally increased values being enhanced by the action of ultra-violet light. The  $\text{H}_2\text{O}$ -sol. Ca content of soil is raised by drying, the effect increasing with the intensity of the drying process. The values are still further increased by ultra-violet treatment, which is more active on undried than on dried soils. The rate of settling of aq. soil suspensions was increased by drying, and to a still greater extent by ultra-violet light treatment.

A. G. POLLARD.

**Utilisable water capacity and the mobility of water in soils.** F. SEKERA (Z. Pflanz. Düng., 1931, 22A, 87—111).—A method for determining the  $\text{H}_2\text{O}$  capacity ( $W$ ) of soil is described. The value  $W$  denotes  $\text{H}_2\text{O}$  held in pore spaces of <2 mm. equiv. diam. Larger pore spaces remain air-filled. The "static water" in soils ( $W$  — hygroscopic  $\text{H}_2\text{O}$ ) is that available to micro-organisms.  $\text{H}_2\text{O}$  available to plants averages 72—75% of the static  $\text{H}_2\text{O}$ .

A. G. POLLARD.

**Alcohol method for determining moisture content of soils.** G. J. BOUYOUCOS (Soil Sci., 1931, 32, 173—179. Cf. B., 1927, 887; 1930, 474).—The satisfactory nature of the method is confirmed. A shaking machine is described for the rapid and complete dispersion of the soil, this being essential for accurate working.

A. G. POLLARD.

**Moisture equivalent as a measure of the field capacity of soils.** F. J. VEIHMEYER and A. H. HENDRICKSON (Soil Sci., 1931, 32, 181—193).—The moisture equiv. of soils (Briggs and Shantz) is a satisfactory measure of field capacity ( $\text{H}_2\text{O}$  retained by a field soil after drainage of gravitational  $\text{H}_2\text{O}$ ) in the case of fine-textured soils. In sandy soils (moisture equiv. <12—14%) the field capacity is relatively the higher value. The permanent wilting % in soil is not a linear function of the moisture equiv. Applications to irrigation practice of moisture equiv. vals. are recorded and discussed.

A. G. POLLARD.

**Experimental investigations with Mitscherlich's physiologico-mathematical method on the action of fertilisers.** S. D. DI DELUPIS (Ann. R. Staz. Chim. Agrar. Sperim., 1931, Pubbl. No. 278, 69 pp.).—Experiments with wheat grown on various soils, under various climatic conditions and during the four seasons 1926—



1930, gave results corresponding closely with the values, 0.122 and 0.6, given by Mitscherlich for the coeffs. of the fertilising actions of N and  $P_2O_5$ , respectively. The few discordant data obtained are capable of ready explanation. The influence of various factors on the availability of N and  $P_2O_5$  is discussed. T. H. POPE.

**Decomposition of soil organic matter and its bearing on the question of manuring.** A. S. CORBET (J. Rubber Res. Inst. Malaya, 1931, 3, 5—27).—The rate of  $CO_2$  production for soils and pure cultures of micro-organisms is examined and expressed mathematically. Application of artificial fertilisers up to 3 cwt. per acre had no appreciable effect on the microbiological activity of soil. A. G. POLLARD.

**Chemical and microbiological study of Lufkin fine sandy loam in relation to productiveness.** E. B. REYNOLDS (Texas Agric. Exp. Sta. Bull., 1931, No. 421, 30 pp.).—The nitrifying capacity of this soil was a better index of its productivity than any other factor examined and was positively correlated with the total N and total and available  $H_3PO_4$ . Continuous growing of cotton or maize tended to reduce the nitrifying power. Applications of nitrogenous materials, cottonseed meal, superphosphate, or ground rock phosphate increased the  $NO_3^-$  produced and then nitrifying power of the soil. Under laboratory conditions liming increased the nitrifying power, but the increased nitrifying power was not more significantly correlated with cotton yields than the nitrifying power of the unlimed soil. A. G. POLLARD.

**Growth, behaviour, and maintenance of organic foods in Bahia grass.** W. A. LEUKEL and J. M. COLEMAN (Florida Agric. Exp. Sta. Tech. Bull., 1930, No. 219, 56 pp.).—The top growth of Bahia grass removed by frequent cutting had a more uniform and higher N content and a narrow ratio of total hydrolysed carbohydrate : total N throughout the season than when the plant was grown to maturity. A. G. POLLARD.

**Sources of nitrogen for potato fertilisers in Aroostook county.** B. E. BROWN, F. V. OWEN, and E. R. TOBEY (Maine Agric. Exp. Sta. Bull., 1930, No. 354, 38 pp.).—In a 16-year average  $(NH_4)_2SO_4$  produced higher yields of potatoes than did  $NaNO_3$ , but differences are largely due to climatic factors. The increased yield obtained by applying part of the N in an org. form is not great. Leuna saltpetre,  $NH_4NO_3$ ,  $NH_4Cl$ ,  $NH_4$  phosphates,  $Ca(NO_3)_2$ , and urea compare favourably with  $NaNO_3$  as sole sources of N.  $CaCN_2$  is satisfactory if used in moderate quantities. The reaction and  $H_2O$ -retaining capacity of soils are important factors in determining the efficiency of the various N fertilisers. A. G. POLLARD.

**Fertilising with leucite.** G. TOMMASI and S. D. DI DELUPIS (Ann. R. Staz. Chim. Agrar. Sperim., 1931, Pubbl. No. 279, 29 pp.).—Experiments on wheat, maize, beans, potatoes, etc., grown over a period of several years on soils which Neubauer's method showed to be lacking in potash, indicate that the beneficial effect of leucite is lower during the first year, but equal to or higher than that of KCl during the second year. Leguminous crops profit most from the application of leucite. For wheat, the leucite is best applied to the preceding

crop, especially if this is leguminous, as this appears to accelerate the solubilisation and, hence, the utilisation of leucitic potash. T. H. POPE.

**Exhaustion of soil phosphate.** M. POPP and J. CONTZEN (Z. Pflanz. Düng., 1931, 22A, 1—20).—In pot experiments with soils receiving N and K, the utilisation of P by crops decreased steadily in successive seasons. Grass, but not lucerne, utilised difficultly-sol. P, thus increasing the relative utilisation vals. in a succession of crops. Manuring with P had no effect on the % utilisation by crops of K and N, but manuring with K and N increased the utilisation of P. In unmanured soils the K taken up by plants in 11 years was greater than that initially sol. in 10% HCl. The K of fertilisers applied to soils was more completely utilised where heavier dressings of N fertilisers were used. No evidence of the exhaustion of soil P occurred in 11 years' cropping. The use of rye for determining the assimilable P in soils by the seedling method is unsuitable for grassland tests and rye grass should be substituted. A. G. POLLARD.

**Influence of lime on recovery of total nitrogen in field crops.** J. G. LIPMAN, A. W. BLAIR, and A. L. PRINCE (Soil Sci., 1931, 32, 217—233).—In a 5-year rotation the recovery of N in crops was of the order: forage crops (mostly legumes) > maize > vegetables or potatoes. In general, liming increased both crop yield and the total N recovered, the effects of Ca- and Mg-limestones being practically the same. With legumes CaO increased the % N of the crop. A. G. POLLARD.

**Use of sodium chlorate as a weed-killer.** M. HESSENLAND and F. FROMM (Chem.-Ztg., 1931, 55, 589—590).—In using  $NaClO_3$  solutions as weed-killer it is recommended that the liquid be coloured with an acid wool dye so that an estimate can be made of the amount of solution which is absorbed by the linen overalls of the workers. This is of importance because of the increased fire hazard. Experiments show that linen overalls are more satisfactory than cotton or fire-proofed material, as the fire hazard is increased appreciably only after four soakings with the  $NaClO_3$  solution (1—2%). A. R. POWELL.

**Relationships between soil maps and Niklas' cropping charts in Bavaria.** E. OSTENDORFF (Z. Pflanz. Düng., 1931, 10B, 410—417).—The average yields of various crops are discussed in relation to soil type. A. G. POLLARD.

**Fertilising tomatoes, sweet corn, and musk melons in a 3-year rotation.** J. W. LLOYD (Illinois Agric. Exp. Sta. Bull., 1931, No. 364, 18 pp.).—In manurial trials the use of P (notably as steamed bone-flour) was of prime importance for tomatoes. K decreased the yield of melons but was without effect on the other crops.  $NaNO_3$  and dried blood increased tomato yields, but did not affect the other crops.  $(NH_4)_2SO_4$  decreased yields in all 3 cases. A. G. POLLARD.

**Effect of calcium and phosphorus contents of soils in W. Washington on the calcium and phosphorus components of oats and red and white clover.** H. F. HOLTZ (Washington Agric. Exp. Sta. Bull., 1930, No. 243, 45 pp.).—The Ca content of oats

is closely proportional to that in residual upper glacial depression deposits and org. soils, but in no other soil types. The P content of oats varies directly with that of the soil in all types except the residual and lower glacial, but is less closely related to the available than to the total P content. The P content of red clover is closely proportional to the available P content of org. soils. The Ca and P contents of oats is less in eastern than in western areas, but that of red clover is practically the same in both areas. Many oat samples and some of red clover had sufficiently low Ca and P contents to create the risk of deficiency decreases in live stock.

A. G. POLLARD.

**Control of bunt in wheat.** F. D. HEALD and E. F. GAINES (Washington Agric. Expt. Sta. Bull., 1930, No. 241, 30 pp.).—The efficiency of seed treatments for spring wheats was in the order  $\text{CH}_2\text{O} > \text{CuSO}_4 > \text{CuCO}_3$ . Fumigation was unsatisfactory owing to inefficient distribution. For winter wheats the order of efficiency was  $\text{CuSO}_4 > \text{CuCO}_3 = \text{org. Hg. compounds} > \text{CH}_2\text{O}$ . Moderate to high soil-moisture content favoured field infection with bunt. This may be minimised by early sowing.

A. G. POLLARD.

**Control of soft rot (water blister) of pineapples caused by *Theilaviopsis paradoxa*.** B. T. DICKSON, H. R. ANGELL, and J. H. SIMMONDS (J. Coun. Sci. Ind. Res., Australia, 1931, 4, 152—161).—Stem infection of pineapples with soft rot is largely prevented by treatment of the cut stems with borax,  $\text{H}_3\text{BO}_3$ , or  $\text{CaOCl}_2$ , within 5 hr. of cutting. Salicylic and benzoic acids gave complete control; these acids dissolve in the stem juices and penetrate in minute amounts into the core, but not the flesh, of the fruit. PhOH is conveniently applied by brushing the stem with an EtOH solution of not  $> 10\%$  concentration, or by rubbing-in a mixture of 4 pts. of kaolin and 1 pt. of acid.

A. G. POLLARD.

**Factors affecting the mineral content of [South Australian] pastures.** A. E. V. RICHARDSON, H. C. TRUMBLE, and R. E. SHAPTER (Coun. Sci. Ind. Res., Australia, Bull., 1931, No. 49, 47 pp.).—In pot cultures of barley, *Lolium*, and *Trifolium*, N assimilation and the absorption of mineral nutrients were most active at the tillering stage. Photosynthesis reached a max. just before flowering, and transpiration losses were greatest during the final growth stages. The % of N, P, and K, and, to a smaller extent, of Ca and Mg, fell continuously from tillering to maturity. The total absorption of N, P, and K per unit of  $\text{H}_2\text{O}$  used declined from the initial growth stage to maturity. The major differences in the composition of individual species grown on different soils are attributable to differences in the available supply of nutrients in the soils. The P content of herbage is increased by applications of sol. P fertilisers only on P-deficient soils and other mineral constituents were not appreciably affected. The effect of superphosphate on growth, P intake, and  $\text{H}_2\text{O}$  requirement was greatest in the early growth stages and least at maturity, and also it was greater in a relatively dry than in a wet soil. On a soil of low  $\text{H}_2\text{O}$  content the dry-matter production of *Lolium subulatum* was low, but its protein, Ca, and Mg contents at maturity were higher than on wet soils.

A. G. POLLARD.

**Chemical composition of pasture grasses under different systems of management.** T. W. EVANS (Welsh J. Agric., 1931, 7, 255—267).—Analyses of grasses cut or grazed at 4-, 14-, and 30-day intervals are recorded. The protein content of herbage increased and the proportion of fibre decreased with the intensity of grazing. The CaO content reaches a max. in July, whilst that of  $\text{H}_3\text{PO}_4$  rises continuously throughout the season. The nutritive ratio tended to become narrow with increasing intensity of grazing, but the differences were not great. Under all conditions the nutritive ratio was narrower at the end than at the beginning of the season.

A. G. POLLARD.

**Increasing soil acidity as a means of controlling black root-rot of tobacco.** W. L. DORAN (Massachusetts Agric. Exp. Sta. Bull., 1931, No. 276, 118—146).—Application of  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ,  $\text{Al}_2(\text{SO}_4)_3$ , or S to soil reduced the injury to tobacco by *Thielavia basicola*.  $\text{H}_3\text{PO}_4$  was without effect.

A. G. POLLARD.

**New protection against stored grain insects.** W. P. FLINT and C. O. MOHR (Illinois Agric. Exp. Sta. Bull., 1930, No. 359, 376—388).—Insect injury to maize is preventing by dipping in lubricating oil emulsions made with K oleate. Germination was not affected and treated grain was fed to cattle without injury.

A. G. POLLARD.

**Oil sprays for dormant use.** A. SPULER, F. L. OVERLEY, and E. L. GREEN (Washington Agric. Exp. Sta. Bull., 1931, No. 247, 27 pp.).—Lubricating oil fractions (Saybolt 100—120 sec.) are suitable for dormant spraying. Refinement beyond 50% unsaponifiable val. is unnecessary. The effectiveness of emulsions is not markedly affected by the emulsifier used. The size of the dispersed oil droplets in the emulsion and its stability control the amount of oil retained by the leaves of the plant. Quick-breaking emulsions deposit more oil and cause correspondingly greater injury to plants. Miscible oil emulsions up to 4% concentration do not usually injure trees even when applied at the crit. period of bud development.

A. G. POLLARD.

**Termite damage and recommendations for its prevention and control.** S. F. LIGHT, M. RANDALL, and F. G. WHITE (California Agric. Exp. Sta. Circ., 1930, No. 318, 64 pp.).—Methods of dry-powder injection, fumigation, and ground treatment are described.

A. G. POLLARD.

**Sodium bisulphate as a disinfectant against *Salmonella pullorum* in poultry-yard soils.** P. W. ALLEN and M. JACOB (Tennessee Agric. Exp. Sta. Bull., 1930, No. 143, 14).—Protection of poultry from infection by *S. pullorum* from contaminated soil is obtained by the application of 1 gal. of 5%  $\text{NaHSO}_4$  solution per sq. ft. of soil. The consequent growth of grass on the soil is not affected.

A. G. POLLARD.

**Citric-solubility of phosphates. Determination of Pb and Cu in Bordeaux mixtures.**—See VII.

## PATENTS.

**Composition serving as a fertiliser and agricultural pest destroyer.** E. DEDOLPH (B.P. 356,195, 31.5.30. Can., 1.6.29).—A finely-ground mixture of

limestone with 1—50% S and 5% of a mixture of some or all of the following minerals is claimed: Mn ores, Fe or Cu sulphides or oxides,  $\text{CuFeS}_2$ ,  $\text{ZnS}$ ,  $\text{ZnO}$ ,  $\text{FeAsS}$ ,  $\text{MgCO}_3$ , and KI.

A. R. POWELL.

**Manufacture of a fungicidal and insecticidal composition [solution of arsenious acid].** L. P. CURTIN, Assr. to WESTERN UNION TELEGRAPH CO. (U.S.P. 1,785,074, 16.12.30. Appl., 25.1.27).— $\text{As}_2\text{O}_3$  is boiled with a 0.025—1% solution of  $\text{Na}_2\text{CO}_3$  or with a 0.05% solution of  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ , whereby a 2.5—12.5% solution of  $\text{As}_2\text{O}_3$  is obtained which is useful as a weed-killer or for the impregnation of wood.

A. R. POWELL.

**Insecticide and fumigant.** R. C. ROARK and R. T. COTTON, Assr. to U.S.A. (U.S.P. 1,791,429, 3.2.31. Appl., 23.2.29).—Ethylene oxide in a concentration of 2 lb./cu. ft. of space is an effective fumigant for wheat, fabrics, etc., and is relatively non-toxic to man. (Cf. B., 1928, 732.)

E. LEWKOWITSCH.

[Residues from] cellulose from vegetable fibres.—See V. HCN. Mixed fertiliser salts.—See VII. Treated garbage.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Decolorising "greens" from refined loaf sugar with norit.** I. F. ZELIKMAN and A. I. SICHKARENKO (Nauk. Zapiski Tzuk. Prom., 1930, 10, 403—417).—Treatment with norit (regeneration described) gave sugar of quality lower than that of refined sugar of middle quality. Regenerated norit gave poor results. Decolorisation of refinery greens with bone-black is preferred.

CHEMICAL ABSTRACTS.

**Determination of sugars in marzipan containing "Convertit" and "Invertin."** F. HÄRTEL (Z. Unters. Lebensm., 1931, 62, 253—255).—These substances are added to marzipan to keep it moist and soft, and consist principally of invertase, which produces gradual inversion of the sucrose. Merck's "Invertin" (solids 31.5 g., ash 0.72 g./100 c.c.) contains no diastase or zymase, and 3 drops will invert 50 c.c. of 20% sucrose in 2 hr. at 55°. Since addition of  $\text{H}_2\text{O}$  to the marzipan initiates rapid enzyme action, solutions in hot  $\text{H}_2\text{O}$ , which destroys the enzyme, should be used for polarimetric sugar determinations, or a reading should be taken immediately and again after the inversion period. Since lactose is not attacked by these preparations they may be used for determinations of sucrose in its presence (e.g., in condensed milk or milk chocolate). J. GRANT.

**General rules for the evaluation of commercial sugar products.** F. HÄRTEL (Z. Unters. Lebensm., 1931, 62, 242—252).—In view of the lack of such rules, proposals concerning the prep., the addition of mineral fillings, artificial colouring, sweetening, and flavouring agents, and the purity of the other constituents are scussed in detail for numerous types of confectionery.

J. GRANT.

**Physical chemistry of starch and bread-baking.** VII. X-Ray spectra of the two constituents of starch: amylopectin and amylose. J. R. KATZ and T. B. VON ITALLIE (Z. physikal. Chem., 1931, 155, 199—207; cf. B., 1930, 1126).—Amylopectin and amylose prepared from starch paste both give in the

fresh state the same X-ray diagram, viz., a V-spectrum which is definitely a crystal spectrum. Retrogradation results in the appearance of a B-spectrum and the complete or partial disappearance of the V-spectrum.

R. CUTHILL.

Corrosion of cans.—See X.

## XVIII.—FERMENTATION INDUSTRIES.

**Production and nitrogenous nutrition of bakers' yeast.** H. CLAASSEN (Chem.-Ztg., 1931, 55, 742—744).—Attention is drawn to Henneberg's experiments (B., 1910, 1029, 1125), which showed that, if the amount of N in the fermentation liquid was increased by 45—60% by the addition of  $\text{NH}_4$  salts, the N content of the re-produced yeast was increased by 6—35%. The yeast had a greater fermentative power, and its action in the baking oven was improved. The addition of  $\text{NH}_4$  salts to worts, the N of which was solely in the org. form, did not decrease, but actually increased, yeast reproduction. Wohl and Scherdel (B., 1921, 191 A) submitted only one new fact: that org. N could be replaced by  $(\text{NH}_4)_2\text{HPO}_4$  without decreased yeast production, provided not more than 50% of the total N was replaced.

C. RANKEN.

**Composition of Italian beer.** G. FIANO (Annali Chim. Appl., 1931, 21, 388—396).—Italian beers are of the Pilsen, Munich, and Vienna type. Analytical data are given.

O. F. LUBATTI.

**Detection of benzoic acid as methyl ester.** L. PICK (Z. Unters. Lebensm., 1931, 61, 358).—Conversion into the Et ester by Rohrig's method often fails to detect  $\text{BzOH}$  in wine and meat. Reliable results are obtained if the acid is transformed into the Me ester by stirring an  $\text{Et}_2\text{O}$  extract in the case of wine, or a purified aq. extract in the case of meat, with a few drops of  $\text{Me}_2\text{SO}_4$ .

H. J. DOWDEN.

### PATENT.

**Manufacture of proteinases free from peptidases.** KALLE & Co. A.-G. (B.P. 356,638, 2.2.31. Ger., 31.1.30).—Peptidases are destroyed by keeping for several hr. the solution of mixed proteinases and peptidases the  $p_{\text{H}}$  of which has been adjusted to 3—4 by added acid. The optimum  $p_{\text{H}}$  of the proteinases is thereafter obtained by adding alkali to the solution, and the product is worked up into a dry preparation.

C. RANKEN.

## XIX.—FOODS.

**Slimy gluten of wheat, caused by an insect.** E. BERLINER (Mühlenlab., 1931, No. 1, 1; No. 4, 25—26).—Wheat with slimy gluten and of such poor baking qualities that edible bread cannot be produced therefrom has been observed during the last few years in consignments from Eastern Europe, Italy, and Spain. It is produced by the attack of *Aelia rostrata* and *Eurygaster integriceps* while the grain is milky. Grains so attacked have a low protein content and show punctures made by the insect on the surface. A small proportion of slimy gluten has a noticeable effect on the quality of the flour.

W. J. BOYD.

**Determination of total phosphorus, lipid phosphorus, and phytosterols in ground wheat products.** A. LEULIER and H. CREVAT (J. Pharm. Chim.,

1931, [viii], 14, 214—217).—The refuse grain is much richer in P, phosphatides, and sterols than the flour (75% of the whole grain), which contains only about 33% of the total P and about 55% of the total sterols. About 70% of the total lipid P remains in the flour.

E. H. SHARPLES.

**Action of benzoyl peroxide as a bleaching agent for flour in the manufacture of rye bread.** H. DILLER (Z. Unters. Lebensm., 1931, 62, 255—265).— $Bz_2O_2$  lowers the (gasoline) colour value of rye flour by 25—50%, but also decreases the fermentation power, yeast activity, loaf vol., pore size, elasticity val., and digestibility; the  $H_2O$  content and acidity are raised and the fermentation period is increased by 20%. Its use is therefore disadvantageous both to baker and consumer.

J. GRANT.

**Determination of water in maize.** U. FABRIS (Z. Unters. Lebensm., 1931, 61, 354—357).—Drying at 100—110° for 24 hr. or at 100° for 5 hr. gives almost identical results. The Brown-Duval method of heating with mineral oil at 180° for 20—25 min. and measuring the  $H_2O$  distilled gives low results. More accurate vals. are given by distilling on an oil-bath with turpentine oil (100 g. of maize with 150—200 c.c. of oil) and measuring the  $H_2O$  after 150 c.c. have been collected. By using  $H_2O$ -saturated turpentine and making a correction of +0.2 c.c., vals. are obtained which agree almost exactly with those given by vac. drying.

H. J. DOWDEN.

**Effect of various methods of preparation on the digestibility and feeding value of cereals.** N. HANSSON (Bied. Zentr., 1931, B, 3, 243—274).—In feeding trials with pigs the digestibility of maize and other cereals was increased (up to 18%) by grinding. Values for "flaked" maize were similar. The digestibility and feeding val. of whole or ground cereals was not increased by soaking in  $H_2O$  and was only slightly increased by cooking. The feeding of maize (up to 75% of the concentrates) to pigs did not affect the firmness of the lard produced.

A. G. POLLARD.

**Digestibility by sheep of the constituents of the nitrogen-free extract of feeds.** G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1930, No. 418, 15 pp.).—Feeding-stuffs known to be of high feeding val. are characterised by high contents of starch or of starch and sugar combined, or, if starch is low, of high protein val. The N-free extract of low-value feeding-stuffs contains high proportions of pentosans and of residual N-free extract. The N-free extracts of some feeding-stuffs contain notable amounts of non-carbohydrates. The digestibility of pentosans averages 50—60% in a no. of feeding-stuffs. The pentosans in crude fibre are digested to a less extent than the total pentosans or the pentosans in the N-free extract. The residual N-free extract is usually digested to a greater extent than are the pentosans.

A. G. POLLARD.

**Comparative value of kale and maize silage for milk production.** I. R. JONES and P. M. BRANDT (Oregon Agric. Exp. Sta. Bull., 1930, No. 272, 34).—The substitution of kale for maize silage in the rations for milch cows affected the yield of milk and of butter fat to a greater extent than was indicated by a comparison of

the digestible crude proteins and total digestible nutrient contents of the two materials.

A. G. POLLARD.

**Silage (a) from sunflowers, (b) with the addition of urea.** P. BRIGL and C. WINDHEUSER (Bied. Zentr., 1931, B 3, 220—242).—Sunflowers for silage should be cut when the seed reaches the milk-ripe condition. The product has a higher crude fibre content than that made from maize. Addition of urea (1—2%) to a maize-roots mixture produced a good silage free from butyric acid. Detailed analyses are recorded.

A. G. POLLARD.

**Utilising the soya-bean crop in livestock feeding.** W. B. NEVENS (Illinois Agric. Exp. Sta. Circ., 1931, No. 369, 44 pp.).—Essential points in the compounding of rations containing soya bean for cattle, sheep, horses, pigs, and poultry are recorded.

A. G. POLLARD.

**Soya beans as a substitute for tankage in fattening spring pigs on legume pasture.** C. M. VESTAL (Indiana Agric. Exp. Sta. Bull., 1930, No. 341, 14 pp.).—Soya beans may be substituted for tankage provided mineral supplements are used.

A. G. POLLARD.

**Variability, accuracy, and adaptability of some common methods of determining the keeping of milk.** IV. Correlation studies. V. Broader aspects of variability. A. H. ROBERTSON and J. M. FRAYER (Vermont Agric. Exp. Sta. Bull., 1930, No. 317, 154 pp.; No. 318, 54 pp. Cf. B., 1931, 991).—IV. Comparative tests of a very large number of samples by the methylene-blue and various bacteriological methods are recorded and discussed.

V. Limits of error are discussed.

A. G. POLLARD.

**Refractometry of the lead serum [in analysis] of milk.** S. ROTHENFUSSER (Z. Unters. Lebensm., 1931, 62, 202—209).—The serum is prepared by shaking 50 c.c. of milk with 2.5 c.c. of  $Pb(OAc)_2$  and filtering; this provides a rapid method for refractometric analysis which may be used in the cold and with acid milk, and a serum from which excess of reagent may easily be removed. Compared with the  $CaCl_2$  serum, the acidity, solids, and sp. gr. are less, the ash and N higher, whilst  $n$  may be slightly less or, for acid milks, slightly higher ( $\pm 0.08$ ). Tables give the fall in  $n$  for sera from milk containing up to 100%  $H_2O$  (2.5 for addition of 10%  $H_2O$ ). Additions of  $H_2O$  may also be followed from the fall in Cl content of the serum, but the P content is too small to be of use. Determinations have been made of the change in  $n$  of  $H_2O$  after shaking with solutions of fat in  $C_2H_5Cl_3$  and other solvents.

J. GRANT.

**Relationship between the sugar and chlorine content of milk and its refractometer figure.** T. SUNDBERG (Svensk Kem. Tidskr., 1931, 43, 198—202).—The relationship between the lactose and Cl content of milk (expressed as g./100 c.c. of milk) can be represented by the equation  $l = 7.06 - 18Cl$ , whence  $l = 7.06$  if  $Cl = 0$ , and  $Cl = 0.392$  if  $l = 0$ . If the refractometer vals. of solutions corresponding to the above concentrations be plotted, a curve is obtained which runs parallel to that actually connecting the refractometer val. of milk with its lactose content; the difference in position of the two curves is due to the presence of other substances in the milk. The tables previously

given by Ackermann and Schulze for determining lactose in milk from the refractometer reading are unsatisfactory in the case of unadulterated milk high in Cl. Determinations of lactose and Cl should be carried out, not with raw milk, but with the serum obtained by treatment of the milk with  $\text{Ca}(\text{OAc})_2$  and  $\text{AcOH}$ . H. F. HARWOOD.

**Copper serum [for the refractometric analysis] of milk.** A. BECKEL (Z. Unters. Lebensm., 1931, 62, 170—198).—The serum produced by addition to 30 c.c. of milk of 1.5 c.c. of 17.5%  $\text{CuSO}_4$  in the cold is more sensitive to changes in  $n^{20}$  with %  $\text{H}_2\text{O}$  than the  $\text{CaCl}_2$  serum, added  $\text{H}_2\text{O}$  ( $W$ ) being given by  $100(n_1 - n_2)/(n_2 - \alpha)$ ; where  $\alpha = 12$  (for  $n_2 > 39$ ) to 16 (for  $n_2 < 20$ ), and  $n_1$  and  $n_2$  are the vals. for the original (stall) sample and watered milk, respectively. Heated milk gives lower  $n$  vals., depending on the temp., and acid milk higher vals. Cl' ( $C$  mg./100 c.c.) may be determined (after Drost) by titration of 11 c.c. of milk in the presence of 10 c.c. of  $\text{AgNO}_3$ ,  $\text{HNO}_3$ , and  $\text{Fe}$  alum with  $\text{NH}_4\text{CNS}$ , the concentration of which is adjusted so that 1 c.c.  $\equiv 10$  mg. Cl/100 c.c. of milk. The refractometer Cl val. ( $A$ ) is then given by  $n + 0.06 \times (C - 100)$ . Since  $100d = 54.5 + 1.5(A - 40)$ , where  $d$  is the f.-p. depression,  $W$  may also be found from  $100 \times (A_1 - A_2)/(A_2 - \beta)$ , where  $A_1$  and  $A_2$  refer to the original and watered milks, respectively, and  $\beta$  is 6 (for  $A_2 > 37$ ) and 10 (for  $A_2 < 24$ ). The accuracy is 1% for 0–50% additions of  $\text{H}_2\text{O}$ .  $A$  is min. in late Aug. and max. in Dec. Milk may also be characterised by (a)  $Q = 100 \times (n - \gamma)/C$ , where  $\gamma$  is 15.4 (for  $A > 41$  or 25–30) to 15.0 (for  $A = 35$ –39); (b)  $S = 0.1C/(\text{g. lactose}/100 \text{ c.c.})$ . Tables and curves are given relating  $d$  with  $A$ , and (a) with (b), and for the consts. In spite of fluctuations in  $n$  and  $C$ , the frequency-distribution const. of  $A$  (6%) is as high as that of  $d$ . J. GRANT.

**Preservation of milk for examination.** RÜDIGER (Z. Unters. Lebensm., 1931, 62, 198–202).—A number of org. and inorg. preservatives were tested and 0.07% paraformaldehyde was found to be the most generally suitable. After 3 days at 16–24°, the microscopical characteristics,  $n$ , and Cl content were unchanged, and tests for uterine diseases were still effective. Catalase, however, was killed, and the acidity was increased slightly, so that its original value could not be determined.  $\text{NaOBz}$  (1–1.5%) will preserve milk for 48 hr. without affecting catalase activity. J. GRANT.

**Creaming of milk pasteurised at high temperatures.** J. C. MARQUARDT and A. C. DAHLBERG (New York State Agr. Expt. Sta., Tech. Bull., 1931, No. 180, 26 pp.).—Milk heated to temps. between 65.6° and 72.8° and held for varying periods (<10 sec. to >10 min.) at such temps. has been studied in relation to the effect on the creaming properties. This relationship is expressed by the equation  $Y = 20.0468 - 0.1289X$ , where  $Y$  is the time in min. of exposure to the temp.  $X$ . Absolute uniformity could not be obtained in the results, since different milks showed different "heat induction" periods, i.e., the periods of time during which milk could be heated before creaming properties were affected. E. B. HUGHES.

**Non-acid Babcock method for determining fat in ice cream.** O. R. OVERMAN and O. F. GARRET

(Illinois Agric. Exp. Sta. Bull., 1930, No. 360, 393–406).—The sample is mixed with a solution of  $\text{BuOH}$ ,  $\text{EtOH}$ , and aq.  $\text{NH}_3$  and subsequently treated with a solution of  $\text{Na}_3\text{PO}_4$  and  $\text{NaOAc}$ . The customary Babcock apparatus and technique are utilised.

A. G. POLLARD.

**Packaged ice cream.** K. E. WRIGHT (Massachusetts Agric. Exp. Sta. Bull., 1930, No. 269, 196–206).—The texture of ice cream was controlled mainly by the temp. of the product at the beginning of the hardening process. The improvement produced by additions of gelatin was the result of the subsequent ageing of the gelatin.

A. G. POLLARD.

**Vitamin-A content of milk chocolate.** A. SCHEUNERT and J. RESCHKE (Z. Unters. Lebensm., 1931, 61, 337–340).—Nutritional experiments with rats proved that both Nestlé's and Peter's milk chocolate contain appreciable amounts of vitamin-A. H. J. DOWDEN.

**Action of "Absorbo" filters on coffee infusions.** F. E. NOTTBOHM and F. MAYER (Chem.-Ztg., 1931, 55, 721–723).—Filtration of coffee infusions through normal and special "Absorbo" filters removes at the most 10% of the total caffeine content, but certain bitter constituents which detract from the flavour and aroma are removed almost completely, as well as part of the substances which produce the characteristic coffee flavour.

A. R. POWELL.

**Determination of citric acid [in coffee].** P. A. CLIFFORD (J. Assoc. Off. Agric. Chem., 1931, 14, 298).—When the method of Hartmann and Hillig (B., 1930, 392) was applied for this purpose, the pentabromoacetone was found to be contaminated by  $\text{EtOH-Et}_2\text{O}$ -sol. material. A method is described whereby the pentabromoacetone is volatilised from the impurity in a current of warm air, and its wt. determined by difference. The sublimate may be collected and its m.p. found. Pentabromoacetone has m.p. 73–74°. W. J. BOYD.

**Microscopical pollen analysis of honey. IV. Foreign honey.** C. GRIEBEL (Z. Unters. Lebensm., 1931, 61, 241–306; cf. B., 1930, 740).—To assist in the detection of imported honey, the botanical characteristics, colour, shape, and size of a number of foreign pollen grains are described and illustrated by photomicrographs. An index to more than 400 varieties is given.

H. J. DOWDEN.

**Bitter honey from Gallura [Sardinia].** A. SANNA (Annali Chim. Appl., 1931, 21, 397–402).—This honey has an aromatic flavour, the taste is decidedly but pleasantly bitter, and the colour lemon-yellow. The taste is attributed to the presence of arbutin, which the bees obtain from the leaves and flowers of *Arbutus unedo*, L.

O. F. LUBATTI.

**Composition and digestibility of turnips.** F. HONCAMP and W. SCHRAMM (Bied. Zentr., 1931, B, 3, 208–219).—Newer varieties of turnips have no greater differences in their contents of dry matter and crude nutrients than those produced by soil and climatic conditions, and the digestibility of the nutrients is not essentially different. The digestible protein content is higher than the usually accepted val. (averaging 0.4–0.5%). Turnips having high dry matter contents as a result of cultural practices show an increased

starch equiv. Losses of nutrient val. during winter storage are considerable. A. G. POLLARD.

**Feeding value of sugar-beet tops.** F. HONCAMP and W. SCHRAMM (Bied. Zentr., 1931, B, 3, 174—207).—A satisfactory conc. foodstuff may be obtained from washed beet leaves by ensilage or artificial drying. Washing the leaves prior to ensilage produced a better material than washing the ensiled leaves. Detailed analyses and digestibility trials with sheep are recorded.

A. G. POLLARD.

**Judging the quality of raspberry juices.** H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1931, 61, 341—345).—A "strength" test has been developed which consists in progressive dilution until the taste and odour become just imperceptible. The vals. for a number of samples of different origin varied between 1:667 and 1:250. Colour measurements on the Ostwald colorimeter fell within fixed limits, and the common adulterants (cherry and currant juices) were found to be of identical colour but different depth. Artificial essences are distinguishable on dilution by their ethereal perfume. Storage for a year does not appreciably affect the "strength" test vals., although it produces changes in general quality and in colour.

H. J. DOWDEN.

**Refractometric studies on fruit juices.** H. ECKART (Z. Unters. Lebensm., 1931, 61, 346—353; cf. B., 1926, 460).—The vals. of  $n_D$  for the juice of a no. of fruits have been determined, especially raspberries of different qualities. The average val. for the juice of fresh ripe raspberries (fit for dessert) is 1.3462, for second-quality fruit (for preserves) 1.3422, and for low-quality fruit 1.3367, the decrease being approx. proportional to the time which has elapsed since the gathering. Adulteration of the fruit juice with  $H_2O$  has a similar influence, the addition of 50% reducing  $n_D$  from 1.3473 to 1.339. The construction and application of a hand refractometer are described.

H. J. DOWDEN.

**Pickling green olives.** W. V. CRUESS (California Agric. Exp. Sta. Bull., 1930, No. 498, 42 pp.).—Best results were obtained by the use of a lye containing 1.7—2.0% of NaOH for 5—6 hr., followed by washing for 20—48 hr. Fermentation was facilitated by the addition of a starter of active green-olive brine, of small amounts of vinegar, or of lactic acid. Addition of dextrose aided fermentation and increased acidity.

A. G. POLLARD.

**Rancidity changes and the flavour of fats.** C. R. BARNICOAT (J.S.C.I., 1931, 50, 361—365 r).—The effect of free fatty acids and of atm. oxidation of the unsaturated constituents on the flavour of fats was investigated. Addition of the free acids in proportion up to 10 or 15% was found to have no harmful effect on the flavour of beef kidney or brisket fat, mutton kidney fat, or lard. The active O and Kreis vals. at which rancidity became perceptible varied greatly (a) for a given fat exposed to different conditions of temp. and light, and (b) for fats of different composition when subjected to identical conditions. Exposure to direct sunlight and the relative absence of acids less saturated than oleic both favour the early production of rancidity. It is probable that the substances responsible for the odour and flavour of oxidised fats at ordinary temp.

are products of the oxidation of oleic acid rather than of linoleic or linolenic acids.

**Reddening of boned codfish.** J. HANZAWA and S. TAKEDA (Arch. Mikrobiol., 1931, 2, 1—22).—In salted cod which had reddened during storage indole and skatole, but not ptomaines or  $H_2S$ , were observed. In so-called sound fish  $H_2S$  was present and a  $H_2S$ -producing bacillus could be isolated. The red pigment is produced by *Torula Welmeri*.

A. G. POLLARD.

**Preservative experiments with fish.** A. BEHRE and G. ULEX (Z. Unters. Lebensm., 1931, 62, 58—80).—The preservatives tested (which included BzOH, its esters, and its substituted derivatives, hexamethylenetetramine, and  $H_2O_2$ ) doubled the keeping properties of jellied and Bismarck herrings and of sliced tinned salmon and its substitutes (dyed sea salmon); crab preps. could be kept for 3 months. The effect was most marked during the winter months. Jellied herrings and crab preps. were kept for 3 months in the presence of 0.05% of hexamethylenetetramine; 0.15% of  $H_2O_2$ , which is toxic to anaerobes, was found most suitable for jelly preps. which are susceptible to "jelly-illness." For tinned salmon and its substitutes *p*- is preferred to *o*-hydroxybenzoic acid, though the latter is a better preservative but objectionable on medical grounds. Fried herrings required no preservative, the temp. of the oil being of greater importance. Addition of gherkins, onions, etc. usually lowered the keeping properties.

J. GRANT.

**Determination of water-soluble mineral matter in foodstuffs by dialysis.** F. W. SIEBER and H. DIETMANN (Z. Unters. Lebensm., 1931, 62, 266—271).—10 g. of sample (ground with sand if necessary) are dialysed overnight into 200 c.c. of  $H_2O$ , the process is repeated for 3 hr. with fresh  $H_2O$ , and the total dialysate diluted to 500 c.c. Aliquot portions are titrated for the Cl<sup>-</sup> determination (Mohr), whilst for the  $KNO_3$  determination they are reduced with Zn, Fe, and NaOH, and the resulting  $NH_3$  is removed by distillation and titrated with 0.1N-HCl (methyl-orange). The method gave results which were higher by 0.1% or less than those obtained by an ashing process for chlorides in milk, salted egg-yolk, and cheese, and for  $NaNO_3$  in meat, but gave low results (0.5%) for chlorides in salted meat. Cheese gives low results if not de-fatted.

J. GRANT.

**Rapid determination of crude fibre in feeding-stuffs.** K. SCHARRER and K. KÜRSCHNER (Bied. Zentr., 1931, B, 3, 302—310).—The sample (1—3 g.) is heated under reflux for 30 min. with 75 c.c. of 70% AcOH 5 c.c. of conc.  $HNO_3$ , and 2 g. of  $CCl_3 \cdot CO_2H$ . The residue is collected on a Gooch crucible, washed with EtOH and  $Et_2O$ , dried at 100—110°, and weighed.

A. G. POLLARD.

**Corrosion of cans.**—See X. Cacao butter.—See XII. Marzipan. Starch and bread-baking.—See XVII. Bakers' yeast. Detection of BzOH.—See XVIII. Rhubarb.—See XX.

#### PATENTS.

**Treatment of cereals and other starch-containing food substances.** T. S. KELSEY (B.P. 356,794, 6.6.30).—The starch cells are softened by steam and disrupted by passage through cooled steel rollers. After

further disintegration, moisture is removed by a cyclone separator. E. B. HUGHES.

**Baking [especially of sweetened bakery goods].** A. W. WAHL, Assr. to E. W. STEWART & Co. (U.S.P. 1,795,980, 10.3.31. Appl., 15.2.30).—Pectin, either dry or in solution, is incorporated in the mix before baking in the proportion of 1 pt. of pectin to 20 pts. of flour. E. B. HUGHES.

**Preparation of bread and the like for wrapping.** AUTOMATIC BREAD BAKING Co., LTD., F. HAWKINS, and W. MULLER (B.P. 356,791, 5.6.30. Austral., 6.6.29).—The hot bread is placed in a vac. chamber in order to remove excess of moisture before wrapping. E. B. HUGHES.

**Treatment of residue remaining after extracting soluble substances from malt grain.** M. M. KOLLER (U.S.P. 1,799,142, 31.3.31. Appl., 12.10.27).—The pressed liquor from spent grain is subjected to electrolysis, which effects complete precipitation of the suspended solids free from fermentation and in usable form as live-stock food. E. B. HUGHES.

**Pasteurisation of milk.** AKTIEB. SEPARATOR (B.P. 357,064, 24.2.31. Swed., 1.3.30).—Milk is pasteurised at 80° by means of steam at reduced pressure. The steam is at a temp. only a few degrees higher than the highest temp. of the milk. Suitable apparatus is claimed. E. B. HUGHES.

**Production of vitamin-containing pasteurised milk of low bacterial content.** R. R. GRAVE (U.S.P. 1,798,413, 31.3.31. Appl., 14.10.27).—Cow are milked mechanically, and the milk is held at bod temp. for  $\frac{1}{2}$ —1 hr. to allow bacterial action, then pasteurised, and bottled under reduced pressure. The whole process takes place in one plant, access of air being prevented. E. B. HUGHES.

**Treatment of liquids [e.g., milk] by irradiation.** V. C. FROM, C. D. ROWLEY, and A. W. LARSKY (B.P. 346,682, 15.10.29).—Milk is treated simultaneously at just below the pasteurisation temp., preferably 49°, but in any case below 60°, with radiation from ultra-violet and from an infra-red lamp immersed in the milk at opposite sides of the container, whereby the bacteria content is reduced without adverse effect on taste or other properties of the milk. A. R. POWELL.

**Treatment of substances with a view to their purification, preservation, or sterilisation.** V. FROM and C. D. ROWLEY (B.P. 356,783, 10.5., 3 and 11.10.30, and 19.1.31).—Apparatus for subjecting perishable organic solids or liquids (e.g., milk, liquid egg) to intense sound waves of frequency near or above the highest audible frequency is claimed. J. S. G. THOMAS.

**Preservation of fruits, vegetables, grain, and other organic substances.** S. A. KAPADIA (I.P. 356,844, 24.6.30).—An apparatus is described for preserving fruits etc. by maintaining them in a refrigerating chamber containing gases produced by a coke liquid-fuel-fired furnace or the like. The gases should be free from CO. E. B. HUGHES.

**Preservation of fresh fruit and vegetables by gas inhibition and refrigeration.** C. BROOKS (U.S.P. 1,798,781, 31.3.31. Appl., 15.12.30).—Spoilage occurring during the first 24 hr. after warm fruits or vegetables

are loaded into a refrigerator car may be avoided by placing solid CO<sub>2</sub> on top of the shipment. The CO<sub>2</sub> content of the air thereby rises 25—30% in  $\frac{1}{2}$  hr. and the temp. falls; at the end of 24 hr. the CO<sub>2</sub> content is 10%, and in the meantime the standard means of refrigeration has the processes of spoilage under control. E. B. HUGHES.

**Treatment of fruit for prevention of decay.** H. R. FULTON and J. J. BOWMAN (U.S.P. 1,797,572—3, 24.3.31. Appl., 19.11.27).—Fresh fruit, particularly citrus fruit, is treated for 1—10 min. at 15.5—54.4° with a 1—5% solution of (A) Na<sub>2</sub>Al<sub>2</sub>O<sub>4</sub>, (B) MnSO<sub>4</sub>, to prevent stem-end rot, blue-mould rot, etc. E. B. HUGHES.

**Manufacture of pectin solution from apple pomace.** E. R. DARLING and H. F. MACMILLIN, Assrs. to HYDRAULIC PRESS MANUFACTURING Co. (U.S.P. 1,799,140, 31.3.31. Appl., 6.9.27).—Crushed apples are treated with an enzyme to convert starch into sugar, after which palatable juice is expressed first at ordinary pressures and then under high (500 lb.) pressure; the broken mass is then mixed with 10% of hot H<sub>2</sub>O, with or without 2½% of vinegar, and hydraulic pressure of 1000 lb. or more applied, yielding a clear water-white concn. pectin solution. E. B. HUGHES.

**Production and purification of [vegetable] phosphatides.** H. BOLLMANN (B.P. 356,384, 15.7.30. Ger., 21.10.29).—Phosphatides, together with the associated oil, are treated with H<sub>2</sub>O<sub>2</sub>, excess of the latter together with H<sub>2</sub>O being removed by subsequent distillation under reduced pressure. Considerable improvement in colour is obtained without impairing the taste or other qualities of the phosphatides. Capacity for emulsification is much improved. E. B. HUGHES.

**Fumigant.**—See XVI. **Synthesis of vitamins.**—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Study of rhubarb and determination of some of its constituents.** P. VALAER (Amer. J. Pharm., 1931, 103, 495—503).—Free and combined emodin are determined by a modification of the Bornträger reaction. Free emodin is determined by making the solution just acid with 0.1N-H<sub>2</sub>SO<sub>4</sub> and extracting with Et<sub>2</sub>O. The yellow colour of the Et<sub>2</sub>O extract is read in a Lovibond tintometer, using a  $\frac{1}{8}$ -in. cell. The Et<sub>2</sub>O extract is mixed with an equal vol. of aq. NH<sub>3</sub> solution and, after  $\frac{1}{2}$  hr., diluted to 5 times its vol. with H<sub>2</sub>O, the red colour of the NH<sub>3</sub> solution being read in a  $\frac{1}{8}$ -in. cell. Total emodin is determined by preliminary hydrolysis before extraction with Et<sub>2</sub>O. Before hydrolysis the yellow and red readings are in the ratio 1 : 1, whilst after hydrolysis the ratio is 6 : 5. A method which yields gravimetric results is not considered so satisfactory. T. McLACHLAN.

**Knudson and Dresbach's colorimetric evaluation of *Digitalis* leaves.** B. J. OCKELOEN and J. C. TIMMERS (Pharm. Weekblad, 1931, 68, 820—824).—Satisfactory results were obtained by matching the colour of the alkaloid picrate solution against K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solutions, the titre of which gives a measure of the val. of the *Digitalis* prep. (Cf. A., 1922, ii, 882.) S. I. LEVY.

**Determination of formaldehyde in certain [pharmaceutical] preparations, and making up**



**Liquor formaldehydi saponatus**, D.A.B. VI. J. BÜCHI (Pharm. Ztg., 1931, 76, 1063).—The influence of the presence of EtOH, MeCHO, and COMe<sub>2</sub> on the results obtained by five selected methods of analysis is recorded. Application of the results to analysis of the above prep. showed a CH<sub>2</sub>O content too low by 3%; loss of CH<sub>2</sub>O is found to occur by the action of the KOH during prep. and a modified way of making up is suggested. S. I. LEVY.

**Philippine pine-needle oil from *Pinus insularis* (Endlicher)**. I. DE SANTOS, A. P. WEST, and P. D. ESQUERRA (Philippine J. Sci., 1931, 46, 1—5; cf. B., 1931, 935, 768).—Steam-distilled needles (and twigs) of the Benguet pine yielded only 0.043% of dextro-rotatory greenish-yellow oil having  $d_D^{20}$  0.0582,  $[\alpha]_D^{20}$  +20.53°,  $n_D^{20}$  1.4700, acid val. 1.38, sap. val. 7.67, esters (as bornyl acetate) 1.75%; 4.9% and 85.3% distil below 155° and 164°, respectively. The oil is sol. in 10 pts. of 90% EtOH and appears to consist largely of  $\alpha$ - and  $\beta$ -pinene. E. LEWKOWITSCH.

**Root-rot in tobacco**.—See XVI.

#### PATENTS.

**Synthesis of vitamins**. C. M. RICHTER (B.P. 356,793, 6.6.30).—Vitamins-A and -D are synthesised in cholesterol, out of contact with O<sub>2</sub>, on exposure, in a transparent (e.g., quartz) vessel, to infra-red or ultra-violet light. Infra-red light favours production of vitamin-A; ultra-violet that of vitamin-B. [Stat. ref.] J. LEWKOWITSCH.

**Alcohols. Complex salts [for injection]. Quinine double salt**.—See III.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Optical methods for reducing the effects of photographic plate graininess, with special reference to spectral-line and star-image measurements**. F. E. WRIGHT (J. Opt. Soc. Amer., 1931, 21, 485—496).—A crit. review. A method for producing an oscillating image by means of an electromagnet is described. J. LEWKOWITSCH.

**Cellulose nitrate film**.—See V.

#### PATENTS.

**Protective paper strips for photographic film rolls for daylight loading**. I. G. FARBENIND. A.-G. (B.P. 356,981, 23.10.30. Ger., 25.10.29).—One or more layers of parchmented paper, coloured in the usual manner, are used. J. LEWKOWITSCH.

**Manufacture of photographic bleaching-out layers**. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 352,196, 2.6.30).—Thio- or seleno-xanthonium dyes are used instead of pyronine dyes to give a comparatively pure white bleach-out. Examples are: 3:7-diamino- (yellow-red) and 3:7-tetramethyldiamino-10-cyano- (blue) -thioxanthonium chlorides, and 3:7-diamino-1:4:6:9-tetramethyl- (purple), 3:7-tetraethyldiamino-9-cyano-2:8-dimethyl- (purple), 3-amino-7-dimethylamino-1:4-dimethyl- (purple), 1-chloro-3-amino-7-dimethylamino- (red-violet), and 3-amino-7-dimethylamino-2-methoxy- (purple) -seleuoxanthonium chlorides. C. HOLLINS.

**Development of light-sensitive layers**. E. GRONAU, Assr. to E. DIETZGEN Co. (U.S.P. 1,798,414, 31.3.31. Appl., 17.11.25. Ger., 6.1.25).—The layer is moistened with a fine film of liquid (aq. NH<sub>3</sub>) and treated with NH<sub>3</sub> gas at the same time. Suitable apparatus is described. J. LEWKOWITSCH.

**Production of negatives by other means than photography**. G. J. R. JOYCE (B.P. 356,850, 28.6.30).—Carbon duplicating paper through which writing or line-drawings have been traced is mounted on glass or celluloid and used as a negative. J. LEWKOWITSCH.

**Photographic printing**. I. G. FARBENIND. A.-G. (B.P. 356,701, 8.5.30. Ger., 8.5.29).—An optical system is described whereby a print may be made through a lenticular surfaced "negative" film on to another lenticular film without lateral inversion, and with correct tone-value reproduction, with or without a colour screen. Special diaphragms may be employed to minimise diffraction effects. (Cf. B.P. 353,121.) J. LEWKOWITSCH.

**Photographic etching**. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE CORP. (U.S.P. 1,797,210—1, 17.3.31. Appl., [A] 16.12.26, [B] 9.11.28).—(A) A light-sensitive resist for etching is prepared by treating asphalt, pine resin, etc. in one step with SCl<sub>2</sub> in CS<sub>2</sub> solution. The light-insensitive portions are removed by washing with an ether. Aldehydic substances, e.g., lavender oil, may be added. (B) Application of the above. J. LEWKOWITSCH.

**Polymethine dyes as sensitisers**.—See IV.

### XXII.—EXPLOSIVES; MATCHES.

**Storage of nitrocellulose**. W. FERMZIN (Chem.-Ztg., 1931, 55, 729—730).—A determination of the swelling capacity of nitrocellulose gives a truer interpretation of its condition during storage than do stability tests, since it is effected at more nearly corresponding temp. A simple apparatus, comprising a movable wt. and scale, is described. The max. swelling was 32.3% in 95% and 13.1% in 75% alcohol, 7% in aq. glycerin, 4% in CHCl<sub>3</sub>, and 12.2% in H<sub>2</sub>O. Swelling is influenced by impurities in the nitrocellulose rather than by its N content or fibre length, and increases with temp. In conjunction with the Bergmann-Junk stability test, a determination of the swelling provides an adequate safeguard against risk from decomp. Nitrocellulose wetted with 35% of a damping medium may lose 5—8% of this medium through gravity separation in 3 months, and the rate of separation increases with the sp. gr. and b.p. of the liquid. If the nitrocellulose is treated with 35% of 40—60% EtOH or BuOH there is no appreciable separation after 4 months. W. J. WRIGHT.

**Cellulose nitrate film**.—See V.

#### PATENTS.

**[Manufacture of high-density] nitrocellulose [from cellulose pulp]**. N. PICTON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 356,252, 5.6.30. Addn. to B.P. 336,235; B., 1931, 46).—Pulp-board made from cotton cellulose is cut up and nitrated as in the prior patent. Hard pulps may require up to 80% of HNO<sub>3</sub> in the acid mixture to ensure penetration, or soft pulps up to 40%. D. J. NORMAN.

**Propellent powders.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 356,256, 5.6.30).—Nitrocellulose, containing preferably  $>12.9\%$  N, is combined with a polynitrotoluene, *e.g.*, dinitrotoluene, and a high-boiling org. ester, *e.g.*, Bu phthalate, both the polynitrotoluene and the org. ester being solvents for the nitrocellulose, non-volatile, non-hygroscopic, and insol. in  $H_2O$ , and their mixture being liquid below  $80^\circ$ . A suitable composition consists of nitrocellulose 80–85, dinitrotoluene 10, Bu phthalate 7–10, and  $NHPh_2$  1 pt. The powder has good stability, even after  $H_2O$  treatment. W. J. WRIGHT.

**Explosive compositions.** A. STETTBACHER (B.P. 356,772, 11.6.30).—A plastic explosive of high brisance, but having a low sensitiveness to shock, is produced by incorporating 5–90% of finely-cryst. pentaerythritol tetranitrate with a mixture of 4–70% of nitroglycerin and 2–20% of dinitroglycol, gelatinised with 0.5–8% of collodion cotton, and adding 10–70% of nitrate or perchlorate of  $NH_4$ , Na, or K. Wood meal, vaseline, or aromatic nitrohydrocarbons may be added. W. J. WRIGHT.

**Percussion caps and the like.** H. W. BROWNSDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 356,765, 28.5.30).

### XXIII.—SANITATION; WATER PURIFICATION.

**Two-stage [sewage] sludge digestion.** I. Plant design and operation. A. M. BUSWELL, H. L. WHITE, and H. E. SCHLENZ. II. Solids balance. A. M. BUSWELL and G. E. SYMONS. III. Gas, grease, and cellulose balance. A. M. BUSWELL and E. L. PEARSON. IV. Use of tank liquor instead of sludge for seeding. A. M. BUSWELL, G. E. SYMONS, and F. L. PEARSON. V. Determination of settling solids. A. M. BUSWELL, A. L. ELDER, C. V. ERICKSON, and G. E. SYMONS (Ill. State Water Survey Bull., 1928–1929, No. 29, 17–27, 28–60, 61–76, 77–81, 82–91).—I. Diagrams and descriptions are given.

II. There can be obtained 63% digestion of total solids, 75% liquefaction, 63% digestion of protein, 83% digestion of  $SO_4$ , and a dehydration of 5% of total solids.

III. Grease digests to give gas.

IV. Seeding of a tank with liquor from another tank caused gasification to start immediately.

V. A method is described. CHEMICAL ABSTRACTS.

**Sodium aluminate in modern water treatment.** R. B. BEAL and S. STEVENS (J.S.C.I., 1931, 50, 307–313 T).—The factors involved in the production of a properly treated water together with the limitations of the simple lime-soda softener are surveyed. By the use of small quantities of Na aluminate the flocculent Mg aluminate ppt. is formed, having unique power to coagulate the finely-divided ppt. produced in the lime-soda softener. Previous attempts using acid coagulants have not been very successful, except in problems of clarification, owing to the increased hardness produced, thus necessitating larger quantities of softening reagents, as well as increasing the total solids fed to the boiler. Na aluminate increases very greatly the speed of chemical precipitation without over-treatment, and the reaction is brought to completion. Mg is removed completely in the softener, and the attendant formation of sol.

basic Mg compounds, which result in after-precipitation in filter-beds, pipe-lines, heaters, etc., is eliminated. By the use of Na aluminate,  $SiO_2$ , one of the worst of the scale-forming substances, is removed as an insol. aluminosilicate. Improved clarification and longer life of filter-beds are possible. Na aluminate solutions are also used in boiler plants where for some reason a softener has not been installed. Sufficient excess alkalinity is produced to prevent scale and corrosion. The Mg aluminate and aluminosilicates coagulate the finely-divided ppts., which are thus prevented from adhering to the boiler metal, and, in view of their size, drier steam is produced. Scale-formation and corrosion due to pollution of the condensate by raw water, and the access of Mg salts at this point, can be eliminated by conditioning the feed-water with a caustic solution of Na aluminate to form flocculent aluminates in the boiler. The  $CO_3/SO_4$  and the  $Na_2SO_4$  alkalinity ratios are discussed together with the relationship between the total solids and suspended solids which can be carried in a boiler water, since priming and foaming is a function of the surface tension of the water and the condition in which the suspended matter exists. Coagulation by the use of Na aluminate has important applications in the removal of colour and turbidity from water and of oil from condensate. Double coagulation with Na aluminate and alum is finding extended use for these purposes.

**Effect of heating and boiling on the temporary hardness of water.** A. V. FILOSOFOV (J. Appl. Chem., Russia, 1930, 3, 1055–1061).—Evolution of  $CO_2$  begins at  $55^\circ$ ; during heating from  $55^\circ$  to  $100^\circ$  the rate of decrease of hardness is const. On boiling, the decrease in hardness is at first rapid, but is negligible after 30–35 min. The results are expressed mathematically.

CHEMICAL ABSTRACTS.

**Filter-sand studies at Denver [waterworks].** O. J. RIPPLE (J. Amer. Water Works' Assoc., 1931, 23, 1317–1319).—Experiments showed that the coarsest sand filter (effective size 0.61 mm.) gave the best results of the sand filters, but a combined sand and coal filter (15 in. of sand at the bottom, effective size 0.50 mm.; 24 in. of coal on top, effective size 0.83 mm.) gave best results of any. C. JEPSON.

**Filter sand [for water].** J. W. ARMSTRONG (J. Amer. Water Works' Assoc., 1931, 23, 1292–1310).—Experimental graded sand filters of varying grain size were treated with a common influent of pretreated water. With this particular water the floc breaks up and adheres closely to fine-grained sand (0.33–0.45 mm. diam.) and tends to form a thin but hard crust which is not broken up when washing and forms mud-banks and balls in the bed. With coarser beds the floc settles lighter, is not broken up, penetrates deeper but is easily removed by washing, and gives longer filter runs. It is suggested that for pressure filters the size of sand grain generally used could be materially increased without adversely affecting the clarity of the effluent and with greater economy in working. C. JEPSON.

**Filtering materials [for waterworks] at St. Louis.** A. G. NOLTE (J. Amer. Water Works' Assoc., 1931, 23, 1311–1316).—Experiments similar to those with filter sand (cf. preceding abstract), carried out at

St. Louis, indicate that the filters containing the greatest proportion of sand between 0.5 and 1.0 mm. gave the best results as judged by the quality of effluent and the length of filter run. C. JEPSON.

**Filtering materials for waterworks.** W. E. STANLEY (J. Amer. Water Works' Assoc., 1931, 23, 1282—1291).—An introduction to a series of investigations carried out at various centres at the request of a Committee of the Sanitary Engineering Division of the American Society of Civil Engineers. An attempt has been made to standardise the equipment used, which consisted of glass-tube filters ( $1\frac{1}{2}$  in. diam.), and the mode of expression of results, which included consideration of sand sizes, washing rates, penetration of floc, length of filter run, filter depth, etc., and are believed to be applicable to large-scale plant. C. JEPSON.

**Manganese in water, its occurrence and removal.** R. S. WESTON (J. Amer. Water Works' Assoc., 1931, 23, 1272—1282).—The chief objections to the presence of Mn in potable waters are its liability to cause brown stains on clothing etc. and its interference with the o-tolidine test for  $\text{Cl}_2$ . But for the latter, its presence in many waters would have been unsuspected, as the amount present rarely exceeds 3 p.p.m. and is usually < 1 p.p.m. In industry it affects the bleaching process and is liable to cause a coating on the insides of pipes and aqueducts. It may readily be removed at reasonable cost by the methods in common use for the removal of Fe, provided suitable pretreatment is given. C. JEPSON.

**Water-purification problems in mining and manufacturing districts.** C. F. DRAKE (J. Amer. Water Works' Assoc., 1931, 23, 1261—1265).—The main difficulty experienced in the mining districts of Pennsylvania is due to rapid variations produced by irregular discharges of acid water from coal mines. Purification of such river water for potable purposes requires plant capable of dealing with peak conditions and the use of large amounts of  $\text{Na}_2\text{CO}_3$  to produce neutrality. C. JEPSON.

**Carbonate number in water analysis.** O. MAYER (Z. Unters. Lebensm., 1931, 62, 271—291).—A rapid scheme of analysis based mainly on the determination of the "carbonate number" ( $1000 \times$  carbonate hardness expressed as mg. CaO per 100 c.c./total solids) is described. "Lime-aggressive  $\text{CO}_2$ " is determined from the increase in the alkalinity val. of the filtered  $\text{H}_2\text{O}$  after 24 hr. in contact with finely-powdered washed marble. Analytical data illustrating the author's methods are given for 91 waters. J. GRANT.

**Determination of nitrate in drinking water.** W. MULDER (Pharm. Weekblad, 1931, 68, 995—997).—The intensity of the colour obtained by Schering's Na salicylate method is proportional to the  $\text{NO}_3^-$  concentration and the colour is stronger than that given by phenol-sulphonic acid. The correction factor necessary in presence of  $\text{Cl}^-$  is valid if not more than 2 mg. of  $\text{Cl}^-$  are present; with more  $\text{Cl}^-$  the correction becomes a const. factor, and accurate results may therefore be obtained by colorimetric comparison if sufficient  $\text{Cl}^-$  be added to both solutions.  $\text{NO}_2^-$ , at high concentrations, intensifies the colour. H. F. GILLBE.

**Determining F in insecticides.**—See VII. **Determining Pb dust in factories.**—See X.

#### PATENTS.

**Treatment of sewage.** W. RUDOLFS, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,797,879, 24.3.31. Appl., 16.5.29).—The addition of small amounts (3.0—5.0 p.p.m.) of  $\text{Fe}^{+++}$  salts to sewage or effluents is claimed to facilitate the separation of suspended solids and to produce a more granular type of sludge. C. JEPSON.

**Increasing the rate of anaerobic digestion of sewage solids.** W. RUDOLFS, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,797,157, 17.3.31. Appl., 16.5.29).—The addition of 1.0—5.0 p.p.m. of  $\text{Na}_2\text{Al}_2\text{O}_4$  or  $\text{Na}_3\text{AlO}_3$  to sewage containing approx. 20.0 p.p.m. of suspended matter accelerates the rate of digestion of the carbohydrate materials in the resultant sludge. The production of  $\text{CH}_4$  is increased, giving a higher thermal val. to the resultant gas, and the time required for complete digestion at room temp. is reduced by 30—40%. C. JEPSON.

**Apparatus for treating organic material [e.g., garbage].** J. H. FEDELER (U.S.P. 1,797,335, 24.3.31. Appl., 12.2.27).—The garbage, after being freed from tins etc. and dried by squeezing between rollers, is passed on a series of endless belts through zig-zag chambers in countercurrent to hot air. The dried material is reduced in bulk and may be used as a fertiliser or as a source of heat for the process. C. JEPSON.

**Treatment of wastes.** F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,799,444, 7.4.31. Appl., 29.3.26).—Atm. nuisance due to  $\text{H}_2\text{S}$  is prevented by aerating screened and detritus-free sewage in the presence of  $\text{Fe}(\text{OH})_3$ ,  $\text{Ca}(\text{OH})_2$ , and a foam-producing agent, e.g., saponin or pine oil. The  $\text{Fe}_2\text{S}_3$  produced is oxidised and the liberated S suspended in the froth which also prevents the escape of  $\text{H}_2\text{S}$ . After sedimentation the effluent is passed on for the necessary further treatment and the sludge is returned to the incoming sewage as in the activated-sludge process. C. JEPSON.

**Treatment of waste [pulp] liquors.** D. D. PEEBLES, Assr. to PEEBLES PROCESSES, INC. (U.S.P. 1,797,585, 24.3.31. Appl., 28.7.26).—The waste liquors from soda- and sulphate-pulp processes, after concentration to about 40—50% of solids in a vac. evaporator, are circulated through a dehydrator until the concentration reaches 90% of solids. This material is burned in a smelting furnace along with  $\text{Na}_2\text{SO}_4$  with production of  $\text{Na}_2\text{CO}_3$  for conversion into NaOH. Sufficient heat is generated to operate the whole plant, and all foul gases are discharged into the hot zone of the furnace and destroyed. C. JEPSON.

**Impregnation of filters [for gas- and smoke-masks].** H. A. KUHN and W. A. BOYLE (Assr.) (U.S.P. 1,798,164, 31.3.31. Appl., 28.10.26).—The filter is formed by impregnating a foraminous cellulose tissue with a finely-divided C product, e.g., lampblack or activated C passing 100-mesh. C. JEPSON.

**[Automatic] apparatus for mixing and delivering liquids [disinfectants for cisterns].** O. COWLEY and E. J. DICKER (B.P. 357,311, 10.7.30).

**Furnaces for waste materials.**—See II. **Sterilisation.**—See XI. **Fumigant.**—See XVI.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

DEC. 4 and 11, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Calculation of heat losses through the walls of industrial furnaces.** E. MAASE (Feuerfest, 1931, 7, 97—102).—The losses are due mostly to the thermal conductivity of the material, but a certain amount of heat may be lost by gases passing through the wall. The theory of calculations is explained, and tables and graphs are presented by means of which losses due to conduction and gas permeability and the external wall temp. can be directly determined. A simple graphic method for determining the temp. curves in an insulated wall is also given. The factors on which the thermal conductivity of a refractory brick depends are summarised. F. SALT.

**Passage of gas through walls of pyrometer protection tubes at high temperatures.** W. F. RÖESER (Bur. Stand. J. Res., 1931, 7, 485—494).—The rate of leakage of air through the walls of tubes of porcelain, fused  $\text{SiO}_2$ , glass, alundum, and metal when heated at various temp. not sufficiently high to cause deformation has been investigated. Sillimanite tubes are superior to the older porcelain tubes both in refractoriness and impermeability. R. CUTHILL.

**Pulp dryers.** M. N. SUIROMYATNIKOV (Sovet. Sakhar, 1929, 80—84).—Two types are described and illustrated.

CHEMICAL ABSTRACTS.

**Capillary systems. XIII. Influence of structure of technical capillary systems on physico-chemical processes. I, II.** E. MANEGOLD, R. HOFMANN, and K. SOLF (Kolloid-Z., 1931, 56, 267—295; 57, 23—39).—Measurement of the electrical resistance, velocity of dialysis, electro-osmotic phenomena, capillary rise, sieve effect, and permeability of several technical filters are described. The measurements show that the relations which have been developed for ideal capillary systems are applicable to these. E. S. HEDGES.

**$\text{SiO}_2$  grinding.**—See VIII. **Heat-treatment furnaces. Corrosion of metals by  $\text{NaCl}$ .**—See X.

### PATENTS.

**Open-hearth furnace.** N. F. EGLER (U.S.P. 1,798,871, 31.3.31. Appl., 5.9.29).—A method of converting a regenerative gas-fired furnace into one suitable for use with other fuels is described. A burner is inserted into the gas port, the gas riser is closed by a damper, and any air supplied with the fuel is not pre-heated. The gas regenerator is used for air which passes to the air riser through by-pass passages specially made for it. B. M. VENABLES.

**Rotary kiln [for low-temperature carbonisation etc.].** E. ROSER, Assr. to C. H. ELLSWORTH (U.S.P.

1,798,500, 31.3.31. Appl., 2.9.21. Ger., 1.2.19).—The kiln is externally heated and on the interior is provided with a helical rib to advance the material. The channel formed by the rib may be either wider towards the axis or crossed at intervals by partitions parallel to the axis; in the former case the hollow of the tapering rib may be open to the external heating gases. B. M. VENABLES.

**Rotary-kiln cooler.** F. LUTHER, Assr. to G. POLYSIUS (U.S.P. 1,797,831, 24.3.31. Appl., 28.10.26).—The cooler is an extension of the kiln and comprises an axial air space, through which the fuel supply extends, surrounded by an annular space divided into sectors in which the material is tumbled about.

B. M. VENABLES.

**Furnace regenerator.** F. R. MCGEE (U.S.P. 1,799,856, 7.4.31. Appl., 14.6.28).—A form of regenerator brick is described, the assemblage of a number of which produces straight vertical passages connected by minor horizontal ones to equalise the flow, which desideratum is also promoted by tapering the inlet and outlet passages. B. M. VENABLES.

**Heat-exchange device.** F. G. CORNELL, JUN., Assr. to JENSEN CREAMERY MACHINERY Co. (U.S.P. 1,799,356, 7.4.31. Appl., 9.4.29).—In a zig-zag apparatus comprising single outer tubes surrounding groups of inner tubes, the outer tubes are permitted to slide in the cross-headers connecting them, and the inner tubes take the longitudinal bursting strain.

B. M. VENABLES.

**Heat exchanger.** P. C. KEITH, JUN., Assr. to REFINERY ENGINEERS, INC. (U.S.P. 1,799,626, 7.4.31. Appl., 8.7.29).—A solid block of metal is formed with a number of parallel bores, and these are connected in two series of zig-zags, one for each fluid. B. M. VENABLES.

**Heat exchanger.** W. M. CROSS, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,799,734, 7.4.31. Appl., 17.12.24).—A reboiling unit for the base of a rectifier is arranged for zig-zag flow of the heating fluid. The header, which is provided with inlet and outlet connexions at one end of the tubes, is cellular and in one piece; at the other end a number of slidably supported headers are provided to allow for expansion. In the rectification of hydrocarbons, residual products may be used as the heating medium, in which case a secondary inlet is provided for use when the main inlet becomes choked.

B. M. VENABLES.

**Heat exchanger.** J. B. RATIBUN, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING Co. (U.S.P. 1,799,391, 7.4.31. Appl., 29.3.28).—In a jet heating system for circulating and heating a liquid by admixture of vapour, the mixing chamber is situated at an upper part of the

\* The remainder of this set of Abstracts will appear in next week's issue.

system and is provided with a liquid seal at the outlet for heated liquid, above which is another outlet for permanent gases which are prevented by the seal from being drawn into circulation. B. M. VENABLES.

**Preparing a charge of material to be sintered.** J. E. GREENAWALT (U.S.P. 1,799,163, 7.4.31. Appl., 30.4.29).—In a sintering machine, in order to obtain a bed of uniform permeability throughout the period of "blow," it is found advisable to charge the material in three layers of different grade, the coarsest being next to the grate. In some cases it may be sufficient to screen the mixture of new material, previously sintered returns, and fuel; in other cases it is necessary to screen the materials separately and place a greater proportion of coke in the top layer. B. M. VENABLES.

**Drying of [continuous sheet or strip] materials.** J. P. BROWN (U.S.P. 1,798,718, 31.3.31. Appl., 9.7.26).—The drying chamber is divided into sections, the transverse walls of which may be formed of the material itself (e.g., paper). The drying air enters at the same end as the material and passes across the sections in turn, being reheated between each stage to a temp. slightly higher than when entering the stage before. Finally the moist air is passed over a condenser, except a part which is by-passed under control of a humidistat, and the whole is reheated and re-used. B. M. VENABLES.

**Drying of articles [e.g., storage-battery plates].** W. L. REINHARDT, Assr. to WILLARD STORAGE BATTERY Co. (U.S.P. 1,799,248, 7.4.31. Appl., 10.11.24. Renewed 25.11.27).—The articles are dried in a series of chambers through which a current of heated inert gas is drawn. The first of the series is dried to a predetermined content of  $H_2O$ , then removed from the circuit, and subjected to a vac.; meanwhile another chamber containing raw goods is added to the other end and the process repeated. To protect the negative plates against oxidation after removal from the vac. they may be dipped before drying in a solution which leaves a film. The drying gas may conveniently be the vapour of a volatile liquid which is immiscible with  $H_2O$ , so that it can be recovered by condensation. B. M. VENABLES.

**Sealing of drying chambers and the like.** R. R. JONES, Assr. to FIRESTONE TIRE & RUBBER Co. (U.S.P. 1,799,375, 7.4.31. Appl., 1.6.27).—Inlet and outlet passages for strip material passing to and from a chamber for drying or other treatment are supplied with inert gas at a pressure above that existing both inside and outside the treatment chamber; devices such as squeegee strips are provided to avoid leakage as far as possible. B. M. VENABLES.

**Condenser [for use with refrigerators].** G. HILGER (U.S.P. 1,798,795, 31.3.31. Appl., 18.11.29).—In a condenser used for liquefying refrigerant by a falling stream of water, to render the lower part of the apparatus more active minor streams of the vapour are injected into the lower pipe-coils, and to avoid wasting tube surface in removal of superheat the main inlet of vapour at the top is in the form of an injector which draws cold vapour from the outlet of the condenser and mixes it with the hot arriving vapour. B. M. VENABLES.

**Condenser [for use with refrigerators].** G. H. WHITE (U.S.P. 1,798,824, 31.3.31. Appl., 12.6.29).—The vapour, e.g., refrigerant  $NH_3$ , is passed over the outside of a bundle of vertical tubes, the circulating water being caused to flow as quietly as possible in a film down the interior of the tubes while air is blown up them; additional means of contact between air and water is provided in the base of the apparatus, but no external cooling pond or tower is provided. B. M. VENABLES.

**Crusher.** R. C. NEWHOUSE, Assr. to ALLIS-CHALMERS MANUFACTURING Co. (U.S.P. 1,799,476—7, 7.4.31. Appl., [A] 15.9.22, [B] 22.12.24. Renewed [A] 22.12.27).—A high-speed gyratory crusher is provided with an electric motor above, driving the eccentric below by means of a rotating shaft within the gyrating shaft which is made hollow to accommodate it. The whole machine is flexibly suspended on slings or springs. B. M. VENABLES.

**Grinding mill.** N. NELSON, Assr. to BLATCHFORD CALF MEAL Co. (U.S.P. 1,799,435, 7.4.31. Appl., 26.3.28).—A centrifugal beater mill has the beaters formed alternately as transverse bars and as hammers having broad serrated faces. The air inlets near the shaft and outlets beyond the screen are adjustable. B. M. VENABLES.

**Apparatus for extracting soluble substances.** W. E. SANGER and O. H. WURSTER (Assec.) (U.S.P. 1,799,481, 7.4.31. Appl., 9.5.23).—The apparatus, suitable for the solvent extraction of fats and oils, comprises a rotatable jacketed digester. The treatment chamber is provided with a chordal filter diaphragm, and the jacket, adjacent the filtrate space, is fitted with a longitudinal rib stout enough to have a plugged hole formed through it, affording an alternative drain for filtrate; the rib also facilitates drainage of condensed heating steam. The apparatus is provided with the following valved connexions. Through one trunnion: a rotatable pipe leading to the filtrate space of the chamber, also a non-rotating pipe turned upwards within the chamber through which mixed vapours of  $H_2O$  and solvent are withdrawn, by vac. if desired. Through the other trunnion: an inlet for steam to the jacket opposite the rib, two outlets adjacent each side of the rib for condensate, also several injection pipes rotating with the shell and connected to a rotary valve so that only those pipes that are below the level of the pulp are supplied with treating fluid, e.g., internal steam. Charging and discharging of the solid matter are effected through manholes. B. M. VENABLES.

**Continuous extraction and filtration.** P. L. FAUTH GES.M.B.H. (B.P. 357,172, 12.6.30. Ger., 14.6.29).—Extraction of seed and like material and filtration of the extract are effected simultaneously in a substantially horizontal rotating cylinder of which at least part of the wall is permeable, the membrane being, e.g., silk impregnated against moisture. The material is caused to progress by helical blades and the solvent flows counter-current; the countercurrent effect may be emphasised by dividing the drum into a number of sections by means of constrictions. B. M. VENABLES.

**Deflocculation of colloids.** W. O. BORCHERT, Assr. to NEW JERSEY ZINC Co (U.S.P. 1,799,277, 7.4.31.

Appl., 1.4.25).—Finely-divided solids, *e.g.*, a mineral pulp, are mixed with an electrolyte and with sulphite waste liquor and the colloids thus deflocculated may be removed to any desired extent by decantation.

B. M. VENABLES.

**[Multiple-effect] evaporation of liquids.** D. D. PEEBLES (U.S.P. 1,799,478, 7.4.31. Appl., 5.2.25).—The liquid is heated and sprayed into a number of superposed flash-evaporating chambers in series downwardly, the pressure being reduced at each stage and the liquid collected in a pool before passing to the spray next below, but not being reheated in the evaporator. The liquid is returned to the top of the series by a pump and on its way is preheated by the vapours from all stages except the lowest and finally heated by steam or other external heat.

B. M. VENABLES.

**Vessels for containing acid and other liquids.** H. RÖMMLER A.-G. (B.P. 357,327, 28.7.30. Ger., 29.7.29).—The vessels are constructed of panel plates of pressed artificial resins joined by grooved strips of the same material and secured by pegs, screws, etc. preferably also of the same material.

B. M. VENABLES.

**Gas and liquid contact apparatus.** F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,798,822, 31.3.31. Appl., 14.9.28).—The gases enter through a central cylindrical screen and are then mixed with the liquid in a disintegrator of the intercalating-pin type, surrounding which is a centrifugal fan that, besides applying the draught, acts also as a moisture eliminator.

B. M. VENABLES.

**Gas and liquid contact apparatus.** S. G. STYRUD, Assr. to KOPPERS Co. (U.S.P. 1,798,563, 31.3.31. Appl., 4.4.29).—A scrubbing tower is divided into sections; at the top and between each section the descending liquid is collected and flushed intermittently on to the section below, to give a more uniform wetting with less liquor than if continuously dribbled on.

B. M. VENABLES.

**Gas and liquid contact apparatus.** C. M. ALEXANDER (U.S.P. 1,796,135, 10.3.31. Appl., 17.3.28).—Bubbling trays are described in which the downflows are in one vertical line, and on each tray weirs are provided to cause the liquid to flow across the centre and back by the sides.

B. M. VENABLES.

**Gas cleaner.** J. P. DOVEL, Assr. to G. P. DOVEL (U.S.P. 1,797,906, 24.3.31. Appl., 2.2.28).—In a cleaner (for blast furnaces etc.) of the type in which the gases come into contact with  $H_2O$  and wetted surfaces, reciprocating scrapers are provided in the valleys where the scale and sludge collect, the scrapers being dragged by a main and tail rope.

B. M. VENABLES.

**Separating the constituents of gaseous mixtures.** C. C. VAN NUYS and J. L. SCHLITT, Assrs. to AIR REDUCTION Co., Inc. (U.S.P. 1,799,937, 7.4.31. Appl., 12.10.27).—A large proportion of air (or other gaseous mixture) is separated into  $O_2$  and  $N_2$  of substantial purity by two rectifiers. The air is compressed and cooled by external means, part is liquefied by heat exchange with outgoing products, and the unliquefied part is expanded and liquefied by heat exchange with outgoing  $O_2$  and then rectified to give pure  $N_2$  gas, using a  $N_2$  reflux. The liquid effluent from the first

rectifier is passed to the second, which utilises the first-mentioned portion of liquid air as reflux and yields pure liquid  $O_2$  with some waste of gases approximating to air.

B. M. VENABLES.

**Separation of gases.** R. L. HASCHIE, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,798,733, 31.3.31. 12.11.27).—In, *e.g.*, the separation of  $SO_2$  from furnace gases, the impure moist gases are first passed through a small bed of adsorbent which is saturated with  $SO_2$  but capable of absorbing impurities and  $H_2O$  and then through the main adsorbent which has selectivity for  $SO_2$ . The first bed, being small, may be regenerated by heat, but the larger one, being protected from impurities, will continue to function by simple change of pressure.

B. M. VENABLES.

**Separation of vapours from gases by refrigeration.** M. FRÄNKEL (B.P. 356,889, 28.7.30. Ger., 27.7.29. Addn. to B.P. 294,354 and 308,288; B., 1928, 697; 1930, 492).—In the process described in the prior patents, the gas is admitted to the refrigerator or direct-contact cooler at different levels at different times, utilising more of the refrigerator as the incoming gas gets warmer.

B. M. VENABLES.

**Catalysing gaseous reactions.** E. B. MILLER, Assr. to SILICA GEL CORP. (U.S.P. 1,799,858, 7.4.31. Appl., 21.2.25).—The catalyst is carried in suspension in the gaseous current, and the mixture is heated or cooled during the reaction by transverse heat-exchanging tubes in which the cooling medium may often be the arriving gases. After the reaction the solid pulverulent catalyst is separated by a cyclone (followed if necessary by wet methods) and their activity continuously regenerated before returning to the circuit. The process is applicable to the manufacture of  $H_2SO_4$  and to the cracking of petroleum; in the latter case the catalyst may consist of  $SiO_2$  gel ( $H_2O$  adsorption 21% at  $30^\circ$ , partial pressure 22 mm.) having an active agent containing Fe.

B. M. VENABLES.

**Preparation of adsorbent [clay] material [for decolorisation of oils].** R. E. MANLEY and M. L. LANGWORTHY, Assr. to TEXAS Co. (U.S.P. 1,796,799, 17.3.31. Appl., 20.10.26).—The clay is treated on the countercurrent principle with hot 10%  $H_2SO_4$ .

A. R. POWELL.

**Treatment of decolorising agents.** R. C. PALMER, Assr. to NEWPORT Co. (U.S.P. 1,794,537, 3.3.31. Appl., 24.12.27).—Clay that has been used for decolorising rosin is revived by washing with petroleum naphtha, then with a 1:1 mixture of naphtha and EtOH, and again with the naphtha. EtOH is recovered by fractional distillation.

D. K. MOORE.

**Thermocouples for use in measuring temperatures.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of H. C. STAERLE (B.P. 357,413, 11.11.30. U.S., 12.11.29).—A thermocouple for temp. above  $2000^\circ$  is constructed of  $TaC_3$  (which will not recrystallise at high temp.) and graphite; the e.m.f. generated is about 1 millivolt per  $100^\circ$  difference.

**Colorimeter.** W. L. PATTERSON, Assr. to BAUSCH & LOMB OPTICAL Co (U.S.P. 1,799,539, 7.4.31. Appl., 30.1.26).—A colorimeter of the type in which the depth

of the liquids to be compared is varied by the greater or smaller immersion of transparent plungers is provided with a device by which the scales showing the effective depth of the liquid samples may be read by the same diffused light as is used for the matching.

B. M. VENABLES.

**Production of specimens or negatives for use in the microscopical observation of the surfaces of substances.** GUNZE SEISHI KABUSHIKI KWAISHA (B.P. 358,378, 30.12.30. Jap., 25.10.30).—An amyl acetate solution of celluloid is applied to the surface; after solidifying, the celluloid film is stripped off, and furnishes a replica of the surface.

J. LEWKOWITSCH.

**Refrigerant absorbents.** FRIGIDAIRE CORP., ASSCCS. of H. F. SMITH (B.P. 353,903, 29.4.30 U.S., 30.5.29).—See U.S.P. 1,791,515; B., 1931, 952.

**Producing fine-bubbled foam for fire-extinguishing purposes.** N. SANDOR (B.P. 357,639, 27.6.30).

**Dehydrating agent. Antifreeze solution.**—See VII. Tunnel kiln.—See VIII. Treatment of fumes.—See XIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Absorption and retention of hydrocarbons by solid fuels.** III. B. MOORE (Fuel, 1931, 10, 436—442; cf. B., 1931, 748).—No well-defined relation could be found between the chemical composition of the fuels and their capacities for absorbing and retaining the vapours of  $H_2O$ , paraffin hydrocarbons from pentane to decane,  $C_6H_6$ , PhMe, and xylene. Coke exhibited a relatively greater absorptive capacity for  $H_2O$  vapour than for hydrocarbon vapour, whilst the reverse was true for coals. The absorptive capacity of a coal for  $C_6H_6$ , PhMe, and xylene decreased with increasing mol. wt. of the hydrocarbon; that for the paraffin hydrocarbons passed through a max. for hexane and thereafter decreased with increasing mol. wt. There was no definite relation between the retentive capacities of the fuels and the physical or chemical characteristics of the hydrocarbons absorbed.

A. B. MANNING.

**Record of tests on the drying of coal.** R. A. MOTT (Fuel, 1931, 10, 424—435).—Details are given of a week's test on the "Universal" coal dryer. The dryer is made of cast Fe, is vertical, and uses a gravity feed from section to section. Each section contains a six-sectioned spider. The spiders are staggered in position and revolve in opposite directions in adjacent sections. The coal is heated by a countercurrent of hot gases from a gas-fired furnace. The dryer is simple to control and is economical in ground space. The principal results of the test were: capacity of the dryer 42 tons/hr.; average temp. of gases entering  $420^\circ$ ;  $H_2O$  content of coal entering and leaving the dryer 14.2% and 9.6%, respectively; consumption of heating gas (504.5 B.Th.U./cu.ft.) 20,200 cu. ft./hr.; power requirement 0.5 h.p./ton; evaporation efficiency 60.4%; heating efficiency 86.6%.

A. B. MANNING.

**Determination of the moisture content of hard coal and lignite.** C. HOLTHAUS (Arch. Eisenhüttenw., 1931—2, 5, 149—162).—The results obtained by various methods are critically discussed. Drying a small powdered sample at  $105^\circ$  yields incorrect results owing to

adsorption of air, oxidation of part of the coal, and volatilisation of the more volatile hydrocarbons, but correct results may be obtained by drying a large sample of uncrushed coal overnight at  $105^\circ$ . The most accurate results for powdered coal are obtained by drying in vac., by distillation with xylene, by drying at  $105^\circ$  and absorption of the  $H_2O$  in a dehydrating agent, and by extraction of the  $H_2O$  with abs. EtOH and light petroleum followed by determination of the cryohydric point.

A. R. POWELL.

**Gum resin production and dry distillation.** P. BOBROV (Trud. Vyat. Nauk. Issledov. Inst. Kraev., 1929, 5, 74—80).—When heated at  $245$ – $275^\circ$ , lignin gave chiefly products non-volatile in a high vac., 50% sol. in the usual org. solvents. Cellulose gave  $H_2O$ -sol. products of sweet-caustic taste; 42–45% were non-volatile. They gave reactions for carbohydrates, and were fermentable after treatment with dil. acid. The volatile matter contained much furfuraldehyde and hydroxymethylfurfuraldehyde. CHEMICAL ABSTRACTS.

**Testing of solid fuels in general and splint coals in particular by vacuum distillation at increasing temperatures.** L. B. DE MONGEOT (Giorn. Chim. Ind. Appl., 1931, 13, 411—413).—With a view to the classification of solid fuels, vac. distillation at temp. up to  $1000^\circ$ , followed by determination of the vols. and compositions of the gases obtained over successive temp. ranges, furnishes valuable information and gives indications concerning the val. of the fuel. Curves connecting the vol. of gas evolved with the temp. have their max. at  $800^\circ$  for anthracite and lignite, and at  $600$ – $700^\circ$  for splint coal and peat.

T. II. POPE.

**Evolution of gas from re-heated coke.** K. BUNTE and W. LUDEWIG (Gas- u. Wasserfach, 1931, 74, 893—900, 921—925).—The usual methods of determining the volatile matter content of cokes are unsatisfactory owing to insufficient duration of heating and consequent incomplete evolution of volatile matter. The evolution of volatile matter from coke was investigated by heating powdered coke (4 g. dried at  $105^\circ$  and contained in a porcelain boat placed in an electrically heated Pythagoras "Compo" tube) in pure  $N_2$  at low pressure. The evolved gas was withdrawn by a Hg pump, displaced into collecting vessels and its vol. determined, and analysed. The % of volatile matter was calc. from the amount and composition of the gas. Ten samples of coke from various sources (which are reported) were heated for 2 hr. at  $1000^\circ$ ,  $1100^\circ$ ,  $1200^\circ$ , and the amount and composition of the gas determined at intervals. The most rapid evolution of gas occurred during the first 60 min. (75% of gas evolved during first 25–50 min.) and only small amounts were evolved afterwards. The total amount of gas (i.e., volatile matter content) increased with rise of temp. It is concluded that the amount of volatile matter evolved increases with time of exposure at a given temp. and with rise of temp. when the duration of heating is fixed. Evolution of gas is probably due to pyrogenic decomp. of the coke constituents. Analyses of the gas evolved indicate that rise of temp. or longer exposure to a given temp. results in increase of  $H_2$  content of the gas. Results of analyses



of the coke before and after heat treatment are described and discussed. It is concluded that a technical method for the determination of the volatile matter content of coke can only be arbitrary. H. E. BLAYDEN.

**Reducing power of blast-furnace coke and a new apparatus for its determination.** M. RIEFFEL (*Chim. et Ind.*, 1931, 26, 280—288, 531—540).—The reducing power of coke is determined by measuring the CO:CO<sub>2</sub> ratio in the gas produced by passing a measured quantity (*e.g.*, 20 c.c.) of CO<sub>2</sub> through 25 c.c. of coke (1—2-mm. particles) heated at 950° in a vertical SiO<sub>2</sub> tube. The % CO in the gas mixture is the reducing power of the coke; for a good blast-furnace coke the min. val. is 40. Coke with a lower reducing power is hard and graphitic and is more suitable for foundry purposes, whereas coke with a higher reducing power than 75% is generally too soft for blast-furnace use.

A. R. POWELL.

**Heat-treatment of hydrocarbons with special reference to gaseous hydrocarbons.** A. E. DUNSTAN, E. N. HAGUE, and R. V. WHEELER (*J.S.C.I.*, 1931, 50, 313—318 r).—From a consideration of the paraffin hydrocarbons, C<sub>2</sub>H<sub>6</sub> to C<sub>6</sub>H<sub>14</sub>, it is concluded that in the primary decomp. rupture of the chain can take place at any position producing an olefine and the complementary lower paraffin, or at the limit H<sub>2</sub>. As the series is ascended the tendency for H<sub>2</sub> to be eliminated as a primary reaction product rapidly diminishes. With CH<sub>4</sub> the absence of a C-C linking causes it to differ from the C-chain paraffins and increases its stability. The suggested mechanism is: CH<sub>3</sub>·H = CH<sub>2</sub>· + H·H and then 2CH<sub>2</sub>· = CH<sub>2</sub>:CH<sub>2</sub> → aromatic hydrocarbons, and also CH<sub>2</sub>:CH<sub>2</sub> + H<sub>2</sub> = CH<sub>3</sub>·CH<sub>3</sub>. With the olefines, ethylene, propylene, and the two unbranched butylenes, the principal primary reactions, common to all, involve the formation of either the two-C or the four-C (or both) atom members of the olefine series. If conditions are drastic, secondary reactions occur with the decomp. of butylene to give butadiene and H<sub>2</sub> in one case and propylene and CH<sub>4</sub> in the other. An important secondary change with propylene is the formation of CH<sub>4</sub> and C<sub>2</sub>H<sub>4</sub>, probably by hydrogenation of the radicals formed by scission of the C chain at the single linking. Of the two butylenes it was found that Δ<sup>2</sup>-butylene was more stable than Δ<sup>1</sup>-butylene and the mode of decomp. is indicated. The paraffin hydrocarbons, with the exception of CH<sub>4</sub>, show a transition range of 700° to 750° in which the production of C<sub>2</sub>H<sub>4</sub> reaches a max. Below 725° each hydrocarbon follows its own course, but at the higher temp. there is a close similarity in behaviour. All evidence serves to indicate that the common source of the aromatic hydrocarbons produced above 750° is a C<sub>2</sub> unsaturated hydrocarbon, and that C<sub>5</sub>H<sub>8</sub>, and not C<sub>2</sub>H<sub>2</sub>, fulfils all the necessary conditions. With both paraffins and olefines C<sub>2</sub>H<sub>4</sub> would appear to be the starting point for the production of aromatic hydrocarbons. Details and tables are given of the production of liquid hydrocarbons by pyrolysis. From CH<sub>4</sub> the best yield of aromatic hydrocarbons (8·8%) was obtained at 1050°, whilst the hydrocarbons C<sub>2</sub>H<sub>6</sub> to C<sub>5</sub>H<sub>12</sub> produce 22—27% of aromatic liquids in the temp. range 800—900°. C<sub>6</sub>H<sub>14</sub> at 800—850° gives a 33·8% yield. With

the olefine hydrocarbons the conversion into liquids is equiv. to 36—40% of the olefine passed in the temp. range 700—800°. The "spirit" obtained consists chiefly of C<sub>6</sub>H<sub>6</sub> and some PhMe, styrene, and xylenes. The influence of pressure and dilution on pyrolysis reactions is considered.

**Chemical problems in refining petroleum.** I. M. D. TILITSCHÉEV and K. S. KURUNDIN (*Neft. Choz.*, 1930, 1, 586—598).—Mono-substituted homologues of C<sub>6</sub>H<sub>6</sub> were prepared from gasolines and kerosenes obtained by cracking, and the influence of the concentration of olefines relatively to C<sub>6</sub>H<sub>6</sub>, the amount and nature of the catalyst, the temp., and method of adding AlCl<sub>3</sub> or AlBr<sub>3</sub> was studied. Instead of fresh AlCl<sub>3</sub> or AlBr<sub>3</sub>, sludge obtained in previous experiments can be used. Physical properties of the products are recorded. All the mono-substituted C<sub>6</sub>H<sub>6</sub> homologues have *d* approx. 0·865. The higher homologues are difficult to sulphonate. CHEMICAL ABSTRACTS.

**Refining of mineral oils with absorbent substances.** B. SALADINI (*Atti III Cong. Naz. Chim. pura. Appl.*, 1929, 584—606; *Chem. Zentr.*, 1931, i, 2827).—C has little bleaching and scarcely any desulphurising effect. Fuller's earth has marked decolorising and desulphurising properties. SiO<sub>2</sub> gel has an even greater desulphurising effect, but less decolorising effect. Bauxite gives the best results. A. A. ELDRIDGE.

**Gasoline fractions of representative Japanese crude petroleum.** IV. [Determination of aromatic hydrocarbons.] M. MIZUTA (*J. Soc. Chem. Ind., Japan*, 1931, 34, 330—331 b; cf. B., 1931, 1034).—The aromatic content of straight-run gasolines has been determined by the aniline-point method (cf. Tizard and Marshall, *J.S.C.I.*, 1921, 40, 20 r) by using the following conversion factors for calculating the content from the difference in aniline point before and after sulphonation: C<sub>6</sub>H<sub>6</sub> 1·231, PhMe 1·182, xylene 1·223; and by using 97° and 120° as the cutting temp. for the C<sub>6</sub>H<sub>6</sub>, PhMe, and xylene fractions. The results are inaccurate with gasolines of high aromatic content. Methods of determining the aromatics by extraction with liquid SO<sub>2</sub> or by nitration were unsuccessful. A. B. MANNING.

**Metallic constituents of crude petroleum.** W. B. SHIREY (*Ind. Eng. Chem.*, 1931, 23, 1151—1153).—Analyses of a number of petroleum ashes are given. The quantity of rarer elements contained is not sufficiently high to make petroleum ash of value as a source of these metals. Ni was found in almost all the crude oil examined. V occurs in the more asphaltic crudes, but is not present in any quantity in paraffin-base crudes. A high V content is generally associated with a high Ni content. There appears to be no regularity in the amounts of other inorg. matter in petroleum ash.

T. A. SMITH.

**Isolation and determination of the cyclohexane in a midcontinent petroleum.** J. H. BRUUN and M. M. HICKS-BRUUN (*Bur. Stand. J. Res.*, 1931, 7, 607—615).—From an Oklahoma petroleum 0·33% of cyclohexane has been separated. R. CUTHILL.

**Gumming tendencies of pure olefines in gasoline.** H. A. CASSAR (*Ind. Eng. Chem.*, 1931, 23, 1132—1134).—A number of olefines and diolefines, free from peroxides,

were mixed with olefine-free gasoline and examined for gum formation under various conditions. Simple olefines in 20% concentration do not produce gum after oxidation for 4 hr. at 100° and under 100 lb. air pressure, but begin to do so after 20 hr. Diolefines give large quantities of gum when evaporated in a porcelain dish. The formation of gum is catalysed by the presence of peroxides.  $\text{H}_2\text{SO}_4$  transforms diolefines into gum, as also does  $\text{SO}_2$  when peroxides are present. Peroxides are decomposed by NaOH and a gasoline with gumming tendencies due to peroxides may be improved by NaOH treatment. A gasoline which does not contain peroxides may or may not be improved by treatment with alkali.  $\text{CH}_2\text{O}$  may also be used to destroy olefine peroxides. Removal of peroxides should be carried out before determination of the knock-ratings of olefines.

T. A. SMITH.

**Antidetoning properties of gasolines from Baku.** V. ELANSKI (Azerbaij. Neft. Choz., 1931, No. 2—3, 78—85).—The  $\text{C}_6\text{H}_6$ -heptane equiv. does not necessarily correspond with the aromatic equiv. determined by chemical analysis. Straight-run distillates have better antidetoning qualities than cracked products, except gasolines from Binagadin crude oils.

CHEMICAL ABSTRACTS.

**Testing antidetoning properties of gasolines.** J. KLIGERMAN (Azerbaij. Neft. Choz., 1931, No. 2—3, 72—78).—The chemical aromatic equiv. does not correspond with the  $\text{C}_6\text{H}_6$ -heptane (motor) equiv.

CHEMICAL ABSTRACTS.

**Detonation and auto-ignition temperature of gasolines.** N. KALANTAR (Azerbaij. Neft. Choz., 1931, No. 2—3, 85—91).—The auto-ignition temp. in  $\text{O}_2$  and particularly in air depends on the detonating properties. The average b.p. is inversely proportional to the  $\text{C}_6\text{H}_6$  equiv. Chemical analysis and  $d$  do not indicate antiknock properties. CHEMICAL ABSTRACTS.

**Oils for steel treatment.**—See X. Petroleum oil sprays.—See XVI.

PATENTS.

**Production of solid fuel.** TRENT PROCESS CORP. (B.P. 357,423, 24.11.30. U.S., 26.11.29).—A coal-oil amalgam is made by the process of B.P. 151,236 (B., 1921, 684 A) with a relatively light oil, e.g., gasoline, naphtha, and is then distilled to recover the oil. An oil binder is added to the hot residue, and after distillation of the more volatile constituents of the binder by the sensible heat of the residue the mixture of coal and binder is briquetted. The briquettes are finally carbonised.

A. B. MANNING.

**Manufacture of [fuel] briquettes.** G. KOMAREK, Assr. to KOMAREK-GREAVES & Co., G. MACPHAIL, and C. CORYELL (U.S.P. 1,796,465, 17.3.31. Appl., 25.7.29).—Petroleum coke is mixed with coke breeze, preferably low-temp. coke breeze, and briquetted. If the petroleum coke is relatively low in volatile matter content a binder may be added. If desired, the briquettes may subsequently be coked at 400—900°. A. B. MANNING.

**Coking retort oven.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,799,670, 7.4.31. Appl., 31.10.27).—The heating walls of the oven diverge slightly so as to provide vertical flues wider at the bottom than at the top, and

coking chambers relatively narrower at the bottom than at the top. The heating gases are supplied to the bottom of the flues and the waste gases withdrawn from the top. Steam-distributing channels below the chambers supply steam to the chambers through ports in the bottom thereof. The steam may be passed through superheating channels in the heating walls before being admitted to the distributing channels. By these means the coking operation in the lower part of the retort is completed before that in the upper part, and the lower part of the charge may be steamed relatively early in the coking period.

A. B. MANNING.

**Recording devices for coke ovens.** H. WADE. From H. KOPPERS A.-G. (B.P. 357,187, 18.6.30).—A device for recording the movements of the pusher and/or the leveller into and out of the ovens, and of the pusher mechanism along the battery, is described.

D. K. MOORE.

**Carbonisation of coal by the low-temperature process.** W. RUNGE, Assr. to INTERNAT. COAL CARBONIZATION Co. (U.S.P. 1,797,796, 24.3.31. Appl., 12.6.25).—Pulverised coal is carbonised by allowing it to fall through an upward current of heating gases at about 565° in a vertical retort. The upper part of the retort is cooled, e.g., by being water-jacketed, and the vapours condensed therein are returned to the carbonising zone wherein they are cracked. The coke produced falls into a chamber at the bottom of the retort.

A. B. MANNING.

**Carbonising process and apparatus.** TRENT PROCESS CORP. (B.P. 357,425, 24.11.30. U.S., 26.11.29).—The apparatus comprises a number of superposed, horizontal, tubular retorts connected in series and heated externally by hot combustion gases; each retort is provided with a screw conveyor. The material to be carbonised consists of pulverised coking coal, the agglomerating action of which has been reduced by the addition of a suitable proportion of hot carbonised coal. The lower tubes are water-cooled, and the steam thereby generated is passed through the carbonising zone in order to facilitate removal of the volatile products, which are withdrawn through the hollow shafts of the screw conveyors.

A. B. MANNING.

**Distilling plant [for wood].** J. T. MYERS (U.S.P. 1,795,404, 10.3.31. Appl., 10.8.28).—The wood is packed into an approx. rectangular, steel-walled retort the bottom of which is inclined from front to rear. The retort is heated by furnaces arranged on either side thereof. Steam-supply pipes are arranged within the top of the retort in such a manner as to direct the steam against the heated walls. An angle pipe leading from the bottom of the rear wall of the retort has a box-like trap at the end which dips into a tank below the level of the retort. The vapours evolved are withdrawn through a conduit opening near the centre of the bottom of the retort and are passed thence to a condenser.

A. B. MANNING.

**Production of coke and gas in intermittently operated oven chambers.** C. STILL (B.P. 357,057, 10.2.31. Ger., 11.2.30).—The volatile distillation products are withdrawn separately from the upper gas-collecting space and from ducts formed in the interior

of the charge (cf. B.P. 354,664; B., 1931, 957), the gaseous pressure in the upper space being maintained const., *e.g.*, at 0 mm. water-gauge, whilst the pressure in the ducts is gradually reduced to at least 100 mm. water-gauge below atm., and is again raised to 0 mm. before the end of the coking process. The quality of the products is improved and the time of carbonisation reduced.

A. B. MANNING.

**Production of carbon black.** C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON Co. (U.S.P. 1,798,614, 31.3.31. Appl., 14.3.21).—Hydrocarbon gases are burned in a furnace in a regulated supply of air forced thereto below the burners. On passing upwards in the furnace the air and gas enter a zone of hot products of combustion and burning gases. The hot gases and suspended C are withdrawn by regulated suction through a suitable outlet, are cooled, and the C is separated therefrom. The desired temp. and velocity conditions in the furnace are controlled by permitting a small, carefully regulated proportion of the hot gases to escape from the top of the furnace.

A. B. MANNING.

**Catalytic manufacture of carbon black.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 357,135, 13.6.30).—Carbonaceous gases, *e.g.*, CO or hydrocarbons, are passed through heated chambers made of material that will promote the decomp., *e.g.*, Cu, and of such form that the catalyst, *e.g.*, Ni or CO, falls through the gas stream. The deposited C is separated from the catalyst by flotation, dissolution of the latter by acid, or magnetic separation.

D. K. MOORE.

**Manufacture of amorphous carbon.** A. L. ULLRICH (U.S.P. 1,796,815, 17.3.31. Appl., 24.4.25).—Tar is heated to liquefy it, a solvent, preferably benzol, is added, and the solution is treated with a relatively heavy hydrocarbon oil, *e.g.*, fuel oil or kerosene, in sufficient quantity to ppt. the C particles. The ppt. is washed with a solvent such as light oil and the residual C is dried.

A. B. MANNING.

**Treatment of coal gas.** T. A. CLAPHAM (B.P. 356,763, 3.5.30).— $C_{10}H_8$  and moisture are removed from the gas by cooling it, first by indirect contact with gas that has already passed through the process, and secondly by indirect contact with a cold liquid, *e.g.*, brine cooled in a refrigerator. The cooling is carried out in chambers traversed by tubes through which the cooling medium is circulated. A solvent oil is sprayed into the chambers and carries the deposited  $C_{10}H_8$  from the apparatus with the condensed  $H_2O$ .

A. B. MANNING.

**Manufacture of combustible gas.** H. O. LOEBELL, Assr. to H. L. DOHERTY (U.S.P. 1,790,824, 3.2.31. Appl., 15.3.24).—A mixture of coal and coke is passed down a vertical shaft. A high-temp. zone is maintained at the mid-position by intermittent blasting with air, and between these operations steam is introduced, forming water-gas. This gas, in passing upward, distils volatile products from the descending coal. The heat in the products of the blasting is recovered. The coke and ash leaving the bottom of the shaft are quenched with steam or  $H_2O$  which rises upwards through the shaft.

D. K. MOORE.

**Producing a combustible gaseous mixture free from carbon monoxide.** H. KEMMER (B.P. 356,838, 21.6.30. Ger., 22.6.29).—Coal gas, water-gas, etc. are freed from CO by treating them first with steam, whereby the greater part of the CO reacts according to the equation  $CO + H_2O \rightarrow CO_2 + H_2$ , and then converting the residual CO into  $CH_4$  by interaction with  $H_2$ . The first stage is carried out in the presence of a suitable catalyst, *e.g.*, Fe containing  $Cr_2O_3$ , in conjunction with active C, or lignite coke, etc., and the second stage in the presence of a hydrogenating catalyst, *e.g.*, Ni, in conjunction with  $Al_2O_3$ , active C, etc. Before treatment in the second stage the gas is freed from  $H_2S$ .

A. B. MANNING.

**Manufacture of producer gas.** TRENT PROCESS CORP. (B.P. 357,424, 24.11.30. U.S., 26.11.29).—Anthracite is passed through a heating zone, wherein it is distilled, before its introduction into a gas producer, whereby the necessity for scrubbing the producer gas is obviated.

A. B. MANNING.

**Water-gas process.** W. J. EDMONDS, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,799,359, 7.4.31. Appl., 24.12.26).—Water-gas from a generator of the usual design is passed successively through a desulphurising tower, containing Fe or  $Fe_2O_3$ , a water-seal, a condenser, and a scrubbing tower. The gases enter the desulphurising tower at 300–650° and leave at 175–450°. The desulphurising material is revived at intervals by passing through it air, steam, and/or part of the blow gases from the generator.

A. B. MANNING.

**Oil-gas generator.** E. RECTOR, Assr. to RECTOR GASIFIER Co. (U.S.P. 1,799,247, 7.4.31. Appl., 22.11.26).—A generator designed for use in conjunction with an internal-combustion engine, and to operate continuously and automatically during operation of the engine, is claimed.

A. B. MANNING.

**Treatment of gases [for recovery of gasoline].** H. J. NICHOLS, JUN., and E. W. LUSTER, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,799,619, 7.4.31. Appl., 27.6.27).—Low-pressure refinery gas is compressed by the expansion of a similar high-pressure gas, and is cooled; the condensed gasoline is withdrawn, the gases are mixed at an intermediate pressure, and the residual gasoline therein is recovered, *e.g.*, by oil scrubbing. Other gases containing a volatile substance may be similarly treated.

A. B. MANNING.

**Cracking of oil.** R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,799,530, 7.4.31. Appl., 24.11.20. Renewed 24.3.27).—Preheated raw oil mixed with a definite proportion of reflux is cracked and passed into an expansion chamber. The vapours from this are fractionated and the reflux mixes with the raw oil. Throttle valves enable the system to be operated under a differential pressure.

D. K. MOORE.

**Oil [cracking] treatment.** PETROLEUM CONVERSION CORP. (B.P. 357,265, 24.6.30. U.S., 24.6.29).—Oil is vaporised and mixed with a carrier gas, heated to above the cracking temp. of the oil to bring about conversion thereof, and the temp. and vol. of one of the components of the mixture, *e.g.*, that of the oil vapour, are so regulated as to produce a mixture in which the % S does not exceed

a predetermined figure (1%) below which no substantial corrosion takes place. The resulting mixture is cooled to separate products suitable as motor fuel.

H. S. GARLICK.

**Cracking of petroleum oils.** D. G. BRANDT, Assr. to DOHERTY RES. Co. (U.S.P. 1,791,114, 3.2.31. Appl., 3.5.27).—The reflux from a dephlegmator is heated to cracking temp. in a reflux furnace, but as its velocity of circulation is too rapid to cause serious cracking, it passes into a separator. From this the liquid flows into the top and the vapour into the bottom of a cracking chamber. On the top of this is the dephlegmator, and the vapour rising through the liquid in the former is fractionated in the latter by heating coils through which the raw oil flows on its way to the bottom, or liquid part, of the separator. The vapours from the dephlegmator are condensed to give the low-boiling oils. The liquid is withdrawn from the bottom of the cracking chamber and some from the bottom of the separator, the whole being passed into the bottom of a concentrator in which it boils owing to reduction of pressure. The vapours are fractionated and then condensed, the residual liquid is discharged, and the reflux passed into a residuum furnace in which it is heated to cracking temp. and from which it passes into the separator. The uncondensed gases from the condenser receiving the vapours from the dephlegmator may be passed back into either the residuum or reflux furnace, thereby carrying more oil into the dephlegmator and giving more reflux if desired; since the deposition of C is accompanied by the formation of such gases, the deposition will be reduced according to the law of mass action.

D. K. MOORE.

**Treatment [cracking] of petroleum oils.** L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co (U.S.P. 1,793,443, 17.2.31. Appl., 25.2.29).—The oil is heated in a furnace in which the hottest gases come in contact with the tubes containing the coldest oil, thereby prolonging their life, and then passed into a vaporising chamber from which the heavy oil is withdrawn at the bottom; the vapours leaving the top are fractionated and condensed, giving light oil, and the reflux is returned to the furnace.

D. K. MOORE.

**Conversion [cracking] of petroleum oil.** W. R. HOWARD, Assr. to UNIVERSAL OIL PRODUCTS Co (U.S.P. 1,791,622, 10.2.31. Appl., 4.3.27).—Oil is cracked in the main retort fitted with agitators and the vapours are returned and pass through the oil in the still before being carried to the reflux tower by the vapour pipe. The condensate in the vapour pipe is returned to a reflux still situated inside the main retort in which further cracking takes place, and the residual liquid is pumped into an auxiliary still into which also flows the reflux from the reflux tower. This still is heated and cracking occurs. The vapours and those from the reflux still pass into the main vapour pipe. The liquid left in the auxiliary still may be returned to the main retort.

D. K. MOORE.

**Production of liquid hydrocarbons from solid fuels.** PHYSICAL CHEM. RES. Co. (B.P. 357,206, 30.5.30. Belg., 4.6.29).—Coal is passed through an apparatus wherein it is subjected successively to distillation at

about 500°, distillation at about 900°, and combustion in a furnace which acts as a producer giving water-gas. The gases obtained in the three stages are mixed together, H<sub>2</sub> is obtained by the decomp. of steam added thereto, and the mixture is submitted, at least partly, to the action of a high-potential electrostatic field, and, at least partly, to ultra-violet rays. The liquid hydrocarbons thereby produced are condensed.

A. B. MANNING.

**Production of liquid, in particular aromatic, hydrocarbons.** J. Y. JOHNSON. From I. G. FARBER-ENIND. A.-G. (B.P. 357,170, 10.6.30).—Gaseous paraffin hydrocarbons, in particular CH<sub>4</sub> or gases containing more than 80% of CH<sub>4</sub>, are subjected to thermal treatment at 700—1200° in the presence of a catalyst consisting of free Si.

A. B. MANNING.

**Refining of hydrocarbon oils with metal salts.** A. LACHMANN, Assr. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,826,139 and 1,826,142—7, 8.5.29).—The process described in B.P. 353,053 (B., 1931, 915) is carried out with aq. solutions of salts of Cu, Fe, Cr, Mn, Al, Ni, and Co, respectively.

**Treatment of light hydrocarbons to reduce the risk of ignition from spark formation.** W. KRITCHEVSKY and E. MORRILL (B.P. 356,717, 6.6.30. U.S., 20.6.29).—A small proportion of a compound of the type NXR<sub>3</sub>Y is added to the hydrocarbon, where R is H, an alkyl group, OH, or a substituted alkyl group, X is Ph, CH<sub>2</sub>Ph, an alkyl or substituted alkyl group, and Y is a fatty acid residue. NEt<sub>3</sub> oleate, NH<sub>3</sub>Et stearate, triethanol-ammonium oleate, etc. may be used. It is convenient to add them in the form of a solution in Pr<sup>6</sup>OII etc. Their addition increases the electrical conductivity of the hydrocarbon and so prevents the formation of dangerous charges of static electricity.

A. B. MANNING.

**Liquid fuel.** J. L. WHITEMAN (B.P. 357,453, 31.12.30).—The fuel comprises a crude oil or oils, e.g., petroleum, shale oil, and oils from coal distillation, and at least such proportions of one or more of the lower monohydric aliphatic alcohols, e.g., MeOH, EtOH, that the tar and other normally insol. constituents of the crude oil, e.g., wax, are dissolved or held in suspension. Small quantities of petrol, benzol, etc. may be present.

H. S. GARLICK.

**Colouring of gasoline or other petroleum distillates.** A. L. MOND. From PATENT FUELS & COLOR CORP. (B.P. 357,179, 17.6.30).—A stabilised solution of an org. dye that normally changes colour or separates out from gasoline or other petroleum distillate on storing is prepared by mixing with such dye a H<sub>2</sub>O-insol. soap of at least one of the metals Al, Mg, Ca, Zn, e.g., Al stearate, dissolving the mixture in benzol or other blending agent, and adding this solution to the gasoline etc. Several suitable dyes are mentioned.

H. S. GARLICK.

**Mixing material to be briquetted with a liquid binding medium [with a rotary conveying pump].** E. KLEINSCHMIDT (B.P. 357,863, 29.11.30. Ger., 18.12.29. Addn. to B.P. 322,220).

**Devices for moulding coking materials.** G. HILGER (B.P. 357,591, 25.6.30).

**Apparatus for charging horizontal coke ovens with rammed coal cakes.** C. STILL (B.P. 358,330, 11.11.30. Ger., 16.11.29).

**Process and means for discharging retort ovens.** "INTERTRUST" COMP. GÉN. DE DISTILLATION ET COKÉFACTION À BASSE TEMP. ET MINÈRE SOC. ANON., Assees. of INTERNAT. HOLDING DE DISTILLATION ET COKÉFACTION À BASSE TEMP. ET MINÈRE (HOLCOBAMI) (B.P. 358,385, 12.1.31. Belg., 24.1.30).

**Burner for pulverulent or gaseous fuels.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 358,092, 2.7.30. Ger., 2.7.29. Addn. to B.P. 304,142).

**Construction of burners for gas-fired ovens or kilns for use in manufacture of tiles, pottery, and other ware.** H. STEELE (B.P. 358,292, 15.10.30).

**Apparatus for burning liquid fuels.** F. WATSON. FROM AKTIEBOLAGET BENZO-GAZ (B.P. 357,689, 22.7.30).

**Kiln for low-temp. carbonisation.** Heat exchanger [for stills]. Catalysing gaseous reactions. Clay for decolorisation of oils.—See I.  $H_2$ .—See VII. Graphite crucibles.—See VIII. Bituminous concrete. Covering for road surfaces.—See IX. Sand moulds. Steel alloy [for oil cracking].—See X. Dielectric material. Carrying out chemical reactions.—See XI. Resin from coal.—See XIII. Explosive gas mixtures.—See XXII.

### III.—ORGANIC INTERMEDIATES.

**Quantitative study of the preparations of guanidine nitrate and nitroguanidine.** G. B. L. SMITH, V. J. SABETTA, and O. F. STEINBACH, JUN. (Ind. Eng. Chem., 1931, 23, 1124—1129).—Fusion of dicyanodiamide and  $NH_4NO_3$  at 162—165° for 1 hr. results in max. conversion into guanidine nitrate, m.p. 217° (corr.), which may be separated from the crude fusion melt in 86% yield by crystallisation from  $H_2O$  at 80° followed by hot MeOH. Alteration of these conditions leads to decreased yields of product and contamination with amorphous material. Pure guanidine nitrate, ground to uniform fineness, added to 3 pts. by wt. of conc.  $H_2SO_4$  below 0° gives a 92% yield of nitroguanidine. The reaction must not be prolonged more than 1 hr., otherwise hydrolysis proceeds more rapidly than nitration. F. R. SHAW.

**Mechanism of catalytic hydrogenation of phenol under high pressure.** S. ANDO (J. Soc. Chem. Ind., Japan, 1931, 34, 320—322 B).—With a  $PhOH:5H_2$  mixture and an initial pressure of 117 atm. the principal product was cyclohexanol when the max. temp. was 260°, and cyclohexane when the max. temp. was 470°. With a  $PhOH:2H_2$  mixture, an initial pressure of 48 atm., and a max. temp. of 260°, the principal product was cyclohexanone. From these results and a study of the hydrogenation of cyclohexanol etc. it is concluded that the reactions occur in the order:  $PhOH + 2H_2 \rightarrow C_6H_{10}O + H_2 \rightarrow C_6H_{11}OH \rightarrow C_6H_{10}$  (cyclohexene)  $+ H_2 \rightarrow C_6H_{12}$ . In the absence of  $H_2$  the reaction  $C_6H_{11}OH \rightarrow C_6H_{10}O + H_2$  tends to occur. A marked fluctuation in the temp.-pressure curve, which occurs at about 335° in the experiments with  $PhOH$ , even in  $N_2$ , is

attributed to a physical change associated with the crit. phenomena of the liquids in the autoclave.

A. B. MANNING.

**Catalytic oxidation of *p*-cymene in the vapour phase.** C. E. SENSEMAN and J. J. STUBBS (Ind. Eng. Chem., 1931, 23, 1129—1131).—Oxidation of *p*-cymene with air in presence of  $V_2O_5$  gives  $H_2O$ ,  $CO_2$ ,  $CH_2O$ ,  $HCO_2H$ , *p*-toluic and terephthalic acids. The optimum conditions for the production of *p*-toluic acid are: temp. 375°; space velocity 300 (litres of air used per hr. per litre of catalyst space), with 3—6 times the theoretical amount of air. Higher yields of acids are obtained through the use of  $V_2O_5$  deposited on porcelain than by using granular fused oxide.  $Sn$  vanadate and finely-divided  $MnO_2$  are less efficient. A method is given for the separation and determination of *p*-toluic acid from any  $H_2O$ -sol. acid. F. R. SHAW.

**cycloHexane from petroleum.**—See II. Derivatives of  $\alpha$ -pinene.—See XIII.  $Et_2O$ ,  $EtOH$ ,  $CHCl_3$ , and  $CS_2$  as bactericides.—See XIX. Acetylsalicylic acid solutions and derivatives.—See XX. Saponins.—See XXIII.

#### PATENTS.

**Production of formaldehyde from methane and carbon dioxide.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 353,076, 23.4.30. Ger., 29.4.29).—A mixture of  $CH_4$  and an equal or greater vol. of  $CO_2$ , preferably preheated, is passed one or more times between the plates of a condenser in a high-frequency circuit of high voltage, e.g., frequency  $1.5 \times 10^6$  and voltage 80,000, with or without catalyst ( $MgCO_3$ ); the yield is 150—210 g. of  $CH_2O$  per cu. m. of  $CH_4$  at a gas speed of 1.5 cm./sec. C. HOLLINS.

**Production of acetaldehyde [from alcohol].** K. WIESLER, A. M. PEAKE, and BRIT. INDUSTRIAL SOLVENTS, LTD. (B.P. 353,071, 17.4.30).— $EtOH$ , steam, and air (etc.) are passed over  $Cu$  at 420—240°,  $Ag$  at 450—500°, or  $AgVO_3$  or  $Cu(NO_3)_2$  at 360—400°. The reaction proceeds without further supply of heat, and the temp. is regulated by the steam present. Suitable apparatus is figured. C. HOLLINS.

**Manufacture of acetic and other organic acid anhydrides.** U.S. INDUSTRIAL ALCOHOL CO. (B.P. 353,381, 13.3.31. U.S., 28.5.30).—The production of  $Ac_2O$  from  $NaOAc$  or  $Ca(OAc)_2$  and  $SO_2Cl_2$  or  $SO_2$  and  $Cl_2$  is effected in liquid  $SO_2$  (2—4 pts. for 1 pt. of acetate) at 0—20°; the solution of  $Ac_2O$  in  $SO_2$  is filtered readily and fractionated. C. HOLLINS.

**Concentration of acetic and other lower aliphatic acids.** CELLULOSE ACETATE SILK CO., LTD., and D. HAYES (B.P. 352,585, 24.4.30).—The dil. acid is distilled with  $CH_2Cl_2$ , the residue being 99% acid. The  $CH_2Cl_2$  layer of the distillate may be returned to the fractionating column. C. HOLLINS.

**Manufacture of alkyl chlorides and/or bromides from olefines or mixtures containing olefines.** E. H. STRANGE and T. KANE (B.P. 353,032, 20.1. and 22.5.30).—The addition of  $HCl$  or  $HBr$  to olefines below  $C_7$  is effected in presence of adsorbent materials, such as active  $C$ , active  $SiO_2$ , "glauconil," or kieselguhr. The olefines may be diluted with paraffins, and the reactants

are preferably dried. *E.g.*, propylene (2 c.c. per g. of active C) and HCl are passed over active C at 70°, Pr<sup>o</sup>Cl being recovered by heating the adsorbent to 150°; C<sub>2</sub>H<sub>4</sub> and HCl combine best under pressure.

C. HOLLINS.

**Grinding of organic peroxides.** N.V. INDUSTRIEEL MAATS. V./H. NOURY & VAN DER LANDE (B.P. 358,349, 25.11.30. Holl., 13.12.29).—The (Bz) peroxide for treatment of flour etc. is ground in the presence of a non-reacting, hygroscopic solid, *e.g.*, CaHPO<sub>4</sub>, Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, or CaSO<sub>4</sub>·2H<sub>2</sub>O.

L. A. COLES.

**Continuous production of esters of ethyl alcohol.** HOLZVERKOHLLUNGS IND. A.-G. (B.P. 352,647, 22.5.30. Ger., 23.7.29).—The vapours of aq. EtOH distilled from the fermentation mash are mixed with acid vapour (*e.g.*, aq. AcOH) in a column where esterification takes place and the ester is separated, EtOH excess being returned to the system. Suitable apparatus is described.

C. HOLLINS.

**Production of trialkyl phosphates.** W. J. BANISTER, ASST. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,799,349, 7.4.31. Appl., 18.11.29).—Al butoxide produced by the interaction of Al and BuOH in the presence of HgCl<sub>2</sub> is dissolved in C<sub>6</sub>H<sub>6</sub> and the solution treated with a solution of POCl<sub>3</sub> in C<sub>6</sub>H<sub>6</sub> at 5–8°. When reaction ceases the product is treated with ice and the Bu ester solution separated is washed free from AlCl<sub>3</sub> with H<sub>2</sub>O, then with dil. NaOH solution to remove HCl, and finally with H<sub>2</sub>O. The C<sub>6</sub>H<sub>6</sub> is separated by distillation and the ester purified by distillation under 15 mm. (b.p. 170–174°).

A. R. POWELL.

**Extraction of ethylene glycol or its homologues.** CHEM. FABR. KALK G.M.B.H., and H. OEHME (B.P. 352,651, 24.5.30).—The aq. glycol-CaCl<sub>2</sub> solution from ethylene chlorohydrin and CaO is evaporated in vac. to 18–20% glycol and 40–45% CaCl<sub>2</sub> and this liquor is distilled at 250–400° in vac. with continuous stirring, or the liquor passes through a tubular vac. evaporator with a final temp. of 280–320°, anhyd. CaCl<sub>2</sub> being recovered and the distillate (25–35% glycol) rectified.

C. HOLLINS.

**Manufacture [separation] of ethylidene diacetate.** COURTAULDS, LTD., and J. BROWN (B.P. 353,318, 20.9.30).—The crude product from C<sub>2</sub>H<sub>2</sub> and AcOH is agitated with an aq. salt solution (*e.g.*, NaOAc) of *d* greater than that of ethylidene diacetate, and the upper layer is separated and dried over anhyd. NaOAc. C. HOLLINS.

**Production of hexamethylenetetramine from methane.** GUTHEHOFFUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 352,978, 7.1.30. Ger., 7.1.29).—In the process of B.P. 343,461 (B., 1931, 621) when N<sub>2</sub> is present, hexamethylenetetramine is formed. *E.g.*, CH<sub>4</sub> is led with 5 pts. of air at 40 cm./sec. through a tube between Cu electrodes and an intermediate electrode of Ag-Cu alloy exposed to a field of frequency 1 × 10<sup>6</sup> and voltage 500,000; 1 cu. m. of CH<sub>4</sub> gives 65 g. of CH<sub>2</sub>O and 1.5 g. of hexamethylenetetramine.

C. HOLLINS.

**Manufacture of [diaryl]formamidines.** IMPERIAL CHEM. INDUSTRIES, LTD., T. BIRCHALL, and E. H. RODD (B.P. 353,138, 16.5.30).—HCO<sub>2</sub>H and an arylamine (NH<sub>2</sub>Ph) in excess are refluxed in presence of H<sub>3</sub>BO<sub>3</sub> or a borate and/or Fe.

C. HOLLINS.

**[Manufacture of] wetting, cleansing, foaming, and dispersing agents.** H. T. BÖHME A.-G. (B.P. 353,232, 9.7.30. Ger., 21.9.29).—Unsaturated or hydroxylated primary amines above C<sub>8</sub> are sulphonated either with conc. H<sub>2</sub>SO<sub>4</sub> at 0° to give esters, or more energetically (*e.g.*, with ClSO<sub>3</sub>H or oleum in excess) to give true sulphonic acids. Examples are octadecenylamine and hydroxyoctadecylamine (from olein alcohol hydrobromide).

C. HOLLINS.

**Production of finely-divided phenolphthalein and compositions containing the same.** E. B. PUTT (B.P. 352,934, 4.4.30).—Phenolphthalein is dissolved in alkali and precipitated by acid (AcOH) in presence of colloid (acacia) sufficient to delay precipitation without gelling. A definite proportion of phenolphthalein by-products (*e.g.*, 1- and 2-hydroxyanthraquinones, fluoran, etc.) is preferably added, the mixture being an effective laxative.

C. HOLLINS.

**Manufacture of 6'-chloro-6-methyl-1 : 2 : 3 : 4-tetrahydronaphthalene and 1 : 2 : 3 : 4-tetrahydro-β-naphthaldehyde [ar-tetrahydro-β-naphthaldehyde].** I. G. FARBENIND. A.-G. (B.P. 353,260, 29.7.30. Ger., 29.7.29. Addn. to B.P. 337,289; B., 1931, 149).—Tetrahydronaphthalene gives with CH<sub>2</sub>O and HCl the 6-chloromethyl compound, b.p. 139–141°/11 mm., which is oxidised smoothly by Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and NaOH to ar-tetrahydro-β-naphthaldehyde, b.p. 142–143°/11 mm. (semicarbazone, m.p. 222–223°).

C. HOLLINS.

**Manufacture of [diphthaloyl]naphthalene derivatives.** I. G. FARBENIND. A.-G. (B.P. 353,113, 5.5.30. Ger., 3.5.29).—1 : 5-Di-(*o*-cyanobenzoyl)naphthalene, m.p. 257°, and its halogen derivatives, *e.g.*, 1 : 5-di-(*p*-chloro-*o*-cyanobenzoyl)naphthalene, obtainable from the corresponding *o*-bromobenzoyl compounds and Cu<sub>2</sub>(CN)<sub>2</sub>, are cyclised by heating in H<sub>2</sub>SO<sub>4</sub> or with other acid condensing agents to give 1 : 2 : 5 : 6-diphthaloylnaphthalene; the products may be vatted.

C. HOLLINS.

**Manufacture of hydroaromatic carboxylic acids.** SCHERING-KAHLBAUM A.-G. (B.P. 353,373, 9.1.31. Ger., 11.1.30).—An (alkali) aromatic carboxylate is hydrogenated in aq. solution in presence of Ni. *trans*-Hexahydrophthalic acid, m.p. 213–215°, tetrahydronaphthalic acid, m.p. 184–187°, and hydronaphthylcyclohexylacetic acids (from naphthoic acid) are described.

C. HOLLINS.

**Liquid hydrocarbons.**—See II. Higher fatty acids esters.—See V. Purifying acids [AcOH].—See VII. Carrying out chemical reactions.—See XI. Fatty acid derivatives.—See XII.

#### IV.—DYESTUFFS.

##### PATENTS.

**Manufacture of vat dyes [from naphthalene-1 : 4 : 5 : 8-tetracarboxylic acid].** I. G. FARBENIND. A.-G. (B.P. 353,124, 9.5.30. Ger., 10.5.29. Addn. to B.P. 341,357; B., 1931, 386).—The mixture of vat dyes from naphthalenetetracarboxylic acid and *o*-diamines is separated by crystallising the mixed salts, *e.g.*, hydrochlorides, from an org. solvent, *e.g.*, chloroacetic acid, at 70–80°. Preferably the dye mixture

is dissolved in molten chloroacetic acid and HCl or  $\text{H}_2\text{SO}_4$  is added, whereupon the less sol. salt at once separates.

C. HOLLINS.

**Manufacture of substantive-dyeing disazo dyes containing copper.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 352,956, 14.4.30).—Dianisidine or diphenetidine is tetrazotised and coupled with 1 mol. of J-acid (or a derivative) and 1 mol. of a naphtholdisulphonic acid (or a nuclear halogeno- or alkoxy-derivative) and the dye is boiled with aq.  $\text{CuSO}_4$  until no further change in dyed shade is produced, the methoxy- or ethoxy-groups being thereby hydrolysed. Examples are Cu compounds from: dianisidine  $\rightarrow$  1:3:6-naphtholdisulphonic acid and *p*-anisyl-J-acid (blue on cotton, silk, and viscose silk); dianisidine  $\rightarrow$  1-hydroxy-8-ethoxynaphthalene-3:6-disulphonic acid and phenyl-J-acid (blue).

C. HOLLINS.

**$\text{C}_{10}\text{H}_8$  derivatives.**—See III. Printing inks.—See XIII.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Examination and commercial analysis of cotton cloths.** R. H. KAY (Analyst, 1931, 56, 647—652).—The methods in use for the examination and analysis of cotton cloths are described fully, together with certain modifications which enable the results to be obtained more rapidly.

T. McLACHLAN.

**Preservation of textile fish nets.** A. C. ROBERTSON (Ind. Eng. Chem., 1931, 23, 1093—1098).—Deterioration of nets is largely due to bacterial action, and may be lessened by washing off most of the contamination and drying the net thoroughly. Differences in the physical requirements of the various types of nets necessitate different kinds of preservatives, but a 25% solution of Cu resinate in water-gas tar oil for flexible, and a paint dip containing 5½% of finely-divided  $\text{Cu}_2\text{O}$  for the stiffer trap and pound, nets are the most satisfactory. Crystal-violet, malachite-green, and some other  $\text{CHPh}_3$  dyes inhibit bacterial growth in cultures isolated from nets, but must be suitably mordanted to render them insol. Greater attention should be paid to this use of dyes because the dyed thread is as light and flexible as the original yarn and may prove very valuable in the preparation of gill nets.

B. P. RIDGE.

**Jute-lignin. I. Methods of isolation.** P. B. SARKAR (J. Indian Chem. Soc., 1931, 8, 397—405).—The following known methods were used: (a) 42% HCl at room temp., (b) 4% NaOH under pressure at 185°, (c) 72%  $\text{H}_2\text{SO}_4$  at room temp., (d) 2% aq.-alcoholic NaOH at room temp., and (e) HCl (d 1.19) and  $\text{P}_2\text{O}_5$ ; method (a) was the most satisfactory.

H. BURTON.

**Chemical investigations of bamboo. IV. Bamboo cellulose.** S. OGURI and M. NARA (J. Soc. Chem. Ind., Japan, 1931, 34, 328—329 B. Cf. B., 1931, 1005).—The proportions of residual cellulose obtained after treatment with NaOH and  $\text{CS}_2$  were found to diminish with increase in concentration of the alkali used, and are generally lower in the case of bamboo than of cotton cellulose, showing that the former has the greater chemical affinity for alkali.

F. R. ENNOS.

**Industrial utilisation of cellulose formate.** G. Tocco (Giorn. Chim. Ind. Appl., 1931, 13, 414—418).—Unlike cellulose acetate, the formate (B., 1931, 876) may be dyed by means of the same dyes as cellulose itself. Dry-spinning of  $\text{HCO}_2\text{H}$  solutions of cellulose diformate yields pellicles of pure cellulose of excellent mechanical properties. Owing to the relatively high b.p. of  $\text{HCO}_2\text{H}$ , it is convenient to effect the spinning in a vac., and a machine devised for this purpose is described. This spinning of the  $\text{HCO}_2\text{H}$  solution renders possible incorporation of protein substances in the cellulose, perfectly transparent solid solutions being thus formed; the presence of casein confers marked plasticity on the films and diminishes the inflammability. The pure cellulose films regenerated from cellulose formate are stronger than those of cellophane and far less sensitive to the action of  $\text{H}_2\text{O}$ . They are, moreover, highly transparent and only faintly coloured, and do not require to be glycerined. The manufacture is economical, the cellulose being recovered quantitatively and the only losses being those of acid and catalyst. Films 1 mm. or more thick are obtainable, whereas those of cellophane, obtained by coagulation, scarcely exceed 0.4 mm. The constructional material of the plant used presents difficulties, owing to corrosion troubles.

T. H. POPE.

**State of solution of cellulose derivatives.** R. O. HERZOG and B. LANGE (Proc. World Eng. Congr., 1929, 31, 501—508; cf. A., 1929, 505).—Solutions of acetylated and methylated cellulose in  $\text{CHCl}_3$  and  $\text{CCl}_4$  were examined in Tyndall light and the degree of depolarisation was measured. Depolarisation is dependent on concentration, the effect on dilution being masked by change in particle size. The influence of solvent and temp. is considerable.

T. T. POTTS.

**Cellulose acetate. VI. Analysis and some physico-chemical properties of the acetylation bath. VII. Acetylation of cellulose. VIII. Ripening of cellulose acetate.** K. ATSUKI and N. ISHII (J. Soc. Chem. Ind., Japan, 1931, 34, 331—335 B, 335—339 B).—VI. The  $\text{Ac}_2\text{O}$  content of the acetylation bath was determined by measuring the rise of temp. observed during the reaction with  $\text{PhNH}_2$ ,  $\text{H}_2\text{SO}_4$  and AcOH being determined in the usual way. Measurements of conductivity, sp. gr., viscosity, surface tension, and f.-p. depression of mixtures of two or more components of the bath show that  $\text{H}_2\text{SO}_4$  forms a compound with  $\text{Ac}_2\text{O}$  which can dehydrate and combine with org. compounds, and that  $\text{Ac}_2\text{O}$  forms mol. associations in AcOH, the degree of association being higher in presence of  $\text{H}_2\text{SO}_4$ .

VII. The velocity of acetylation of cellulose after the fibres are disintegrated is given by the formula  $K = [2 \cdot 3026 / (A - B)t] \log [(A - x)B / (B - x)A]$ , where  $A$  and  $B$  are the molar nos. of cellulose and  $\text{Ac}_2\text{O}$ , respectively,  $t$  is the reaction time, and  $x$  is the equiv. of  $\text{Ac}_2\text{O}$  consumed during acetylation. At 30°  $K = 0.0095$ . The function of the  $\text{H}_2\text{SO}_4$  is considered to be that of a catalyst in the colloidal solution.

VIII. The hydrolysis of cellulose triacetate and degradation of cellulose which occur during the ripening process are most marked in a bath containing  $\text{Ac}_2\text{O}$  in presence of AcOH and  $\text{H}_2\text{SO}_4$ , only moderate in one



with traces of  $\text{Ac}_2\text{O}$  or  $\text{H}_2\text{O}$ , and very slight in a bath containing excess of  $\text{H}_2\text{O}$ .  $\text{H}_2\text{SO}_4$  is essential to ripening, its activity as a catalyst depending on the ratio  $\text{H}_2\text{SO}_4/\text{H}_2\text{O}$ . F. R. ENNOS.

**Comparison of the properties of various cellulose acetates and the artificial silks made therefrom.** F. OHL (Textilber., 1931, 12, 647—651).—Four technical cellulose acetates, viz., (A) Zellit L (I.G.), and the  $\text{COMe}_2$ -sol. products made by (B) Ver. f. chem. Ind., (C) Wacker Ges.m.b.H., and (D) Soc. Fabr. Soie "Rhodiaseta" were investigated; their  $\text{AcOH}$  and  $\text{H}_2\text{SO}_4$  contents were 53.97, 54.82, 53.20, and 53.80%, and 0.092, 0.019, 0.022, and 0.098%, respectively. When heated, A and D commence to yellow at  $190^\circ$  and melt and carbonise just above  $200^\circ$ , whereas B and C yellow at  $210^\circ$  and carbonise at  $240^\circ$ , this behaviour being in agreement with their lower content of  $\text{H}_2\text{SO}_4$ . In  $\text{COMe}_2$  solution at  $20^\circ$ , B had the highest viscosity, and this decreased least with rise of temp. to  $50^\circ$  and also on being kept for 7 days. Of the artificial silk threads spun from  $\text{COMe}_2$  solutions of A, B, C, and D, the threads from B had the greatest tensile strength and suffered the smallest % loss of strength and lustre when wetted and when boiled with  $\text{H}_2\text{O}$ ; they also had the most desirable cross-section. When boiled in  $\text{H}_2\text{O}$ , threads from A and D lost considerable lustre and became slightly and very woolly, respectively, those from B lost but little lustre and became slightly woolly, whilst those from C lost much lustre and became very woolly. Cellulose acetate B yielded the most satisfactory silk, and it is concluded that for production of the highest quality of silk the cellulose acetate should be uniform in structure, have small  $\text{H}_2\text{SO}_4$  and high  $\text{AcOH}$  contents, and yield  $\text{COMe}_2$  solutions having a high viscosity which decreases but little with increase of temp. and during storage. A. J. HALL.

**Shape of cross-section of viscose silk [as affected by conditions of spinning].** A. JÄGER (Kunstseide, 1931, 13, 325—328, 352—356).—The composition (e.g., contents of cellulose,  $\text{NaOH}$ , and  $\text{CS}_2$ ) and viscosity of a viscose solution have much less influence on the shape of the cross-section of viscose filaments spun from it than has its ripeness or the composition of the spinning (coagulating) bath; this solution largely affects the cross-section of cellulose acetate silk. With a viscose solution ( $\text{NH}_4\text{Cl}$  ripeness = 14.2) and a spinning bath containing 100—200 g.  $\text{H}_2\text{SO}_4$  and 220—440 g.  $\text{Na}_2\text{SO}_4$  per litre at  $45^\circ$  the resulting circular cross-sections become more ribbon-like as the concentration of both  $\text{H}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$ , or  $\text{Na}_2\text{SO}_4$  alone, is increased; with increase of  $\text{H}_2\text{SO}_4$  the cross-sections become kidney-shaped. With a riper viscose solution ( $\text{NH}_4\text{Cl}$  ripeness = 9.9) the cross-sections also become ribbon-like with increase of  $\text{H}_2\text{SO}_4$  in the spinning bath, and the results obtained show that Bronnert's experiments (B., 1922, 540 A) must have been carried out with fresh (unripe) viscose solutions. The addition of  $\text{MgSO}_4$  and particularly of  $\text{ZnSO}_4$  to the spinning bath confers softness of handle on the resulting silk; the presence of  $(\text{NH}_4)_2\text{SO}_4$  favours a circular cross-section. Salts of Al, Fe, Cr, Ni, Mn, and Cu have an effect similar to, but much

smaller than, that of Mg and Zn salts. The temp. of the spinning bath exerts a comparatively small effect on the shape of the cross-section of the silk. The cross-section of a viscose silk filament is largely determined by the conditions existing at the point of its extrusion from the spinning nozzle, so that although stretching of the silk induces the ribbon-like cross-section to curl and thus resemble a circular section, this effect is only appreciable when the stretching is applied close to the spinning nozzle. Cross-sections of viscose filaments are conveniently prepared by coating a bundle of filaments with Peru glue and then embedding in paraffin wax before cutting. A. J. HALL.

**Making pulp for Japanese paper by use of chlorine.** S. OCHI, I. FUJIMOTO, and H. MURASUGI (Proc. World Eng. Congr., 1929, 31, 515—521).—Chlorination followed by alkaline treatment of a number of fibrous materials indigenous to Japan gives good yields of satisfactory pulps which may be used for the manufacture of papers having the usual Japanese characteristics. Coniferous woods are unsuitable.

T. T. PORTS.

**Bleaching of sulphite pulp with chlorine and calcium hypochlorite.** H. RAUCHBERG (Papier-Fabr., 1931, 29, 490—497, 516—524, 535—541).—Parallel bleaching experiments have been carried out on sulphite wood pulps using  $\text{Cl}_2$  and  $\text{Ca}(\text{OCl})_2$ , respectively. For regular grades of pulp not requiring high freedom from incrustants, bleaching with  $\text{Cl}_2$  is advantageous. The reaction is rapid, 62% of the bleaching being complete in 10—20 sec. Above the optimal  $\text{Cl}_2$  consumption the reaction becomes slow and uneconomical, hypochlorite bleaching being more efficient in this range.  $\text{Cl}_2$  acts equally in chlorination and oxidation of incrustants, in distinction to hypochlorite bleaching, sol. chlorination products being formed. The yield from  $\text{Cl}_2$  bleaching is the lower. The greater amount of washing required by  $\text{Cl}_2$ -bleached pulp is a disadvantage and is the cause of slight losses. An extensive bibliography is given.

T. T. PORTS.

**Determination of lignin in wood pulp.** A. NOLL and F. HÖLDER (Papier-Fabr., 1931, 29, 485—490).—A detailed account is given of the study of the effect of state of division of fibre, addition of  $\text{EtOH}$  and/or  $\text{NPhMe}_2$ , and concentration of  $\text{H}_2\text{SO}_4$  on the purity and yield of lignin obtained by acid hydrolysis of wood pulps, resulting in the formulation of a rapid method previously described (B., 1931, 1005). T. T. PORTS.

**Strength testing of wood pulps.** W. NIPPE (Papier-Fabr., 1931, 29, 418—430).—Existing methods for determining the strength of wood pulps are critically examined, with particular reference to the individual properties of pulp emphasised by each method. No single method yields a complete measure of the strength of the original fibre, and an analysis of the factors involved has been attempted by means of the examination of given pulps by 3 different methods, the resulting range of tensile strengths being different for each method. The measurement of strength of individual fibres has been found impracticable as a means of pulp-strength testing. Degree of beating, swelling, orientation, and felting are examined as decisive factors in the

3 methods employed, and in the absence of better technique the method of Wenzl, consisting of plotting strength with time of beating, is held to be the most complete.

T. T. POTTS.

**Evaporation of [sulphite- and sulphate-]pulp-mill waste liquors.** F. A. OETKEN (Papier-Fabr., 1931, 29, 545—549).—Recent methods are discussed, with particular reference to the avoidance of a ppt. of  $\text{CaSO}_4$ . In the Siemens process, d.c. of 5—10 volts is supplied to the evaporator so that the + terminal is connected to 2 electrodes placed in the liquor stream, the — terminal and the metal of the plant being earthed. The Agfil process employs interrupted current of 0.1—2 volts generated by a thermal battery, the arrangement of the circuit being similar to that of the Siemens except that a slight leakage current is allowed to pass from the + terminal over the metal work to earth. It is claimed that formation of  $\text{CaSO}_4$  scale is avoided in differing degree by the above processes, depending on local conditions. The Lurgi system depends on the formation of "metastable supersaturated" solutions of  $\text{CaSO}_4$  in the evaporator, with rapid crystallisation at suitable points in the liquor stream. The process is claimed to be efficient and economical, details of cost tests being given.

T. T. POTTS.

**Testing of paper half-stuffs for strength.** W. WIRTH (Papier-Fabr., 1931, 29, 524—526).—Wood half-stuffs made into sheets and examined by Humm's method for "punching length" (B., 1930, 609) do not give concordant results, but produce in some cases the opposite of the effect claimed. "Punching length" is independent of degree of beating, but may be a measure of individual fibre strength.

T. T. POTTS.

**Accelerated ageing test for paper.** R. H. RASCH (Bur. Stand. J. Res., 1931, 7, 465—475).—Determination of the strength and various chemical properties of a variety of bond and ledger papers has shown that, in general, a high Cu no. is associated with a low  $\alpha$ -cellulose content, and *vice versa*, and that a permanent paper is characterised by a low Cu no. and a high  $\alpha$ -cellulose content. When the papers were arranged in order of stability, as deduced from the effects of heating for 72 hr. at 100° on the strength and  $\alpha$ -cellulose content, the result apparently corresponded with that which would have been expected from observations on natural ageing. Ageing by heating at 60° for 860 hr. and by exposure to sunlight also gave the same order of stability.

R. CUTHILL.

**Permanence of current commercial book papers.** J. O. BURTON (Bur. Stand. J. Res., 1931, 7, 429—439).—Determination of the resistance of various book papers to ageing caused by heating at 100° for 72 hr. has shown that the permanence of a paper depends primarily on its chemical purity, and has little relation to the source or initial quality of the fibre. If the acid content is greater than the equiv. of 0.1%  $\text{SO}_3$ , both the  $\alpha$ -cellulose content and the strength, as measured by the folding-endurance, diminish considerably as a result of the ageing.

R. CUTHILL.

**Wallboard etc.**—See IX. **Rancidity and porous packings.**—See XII. **Acid caseins [for paper].**—See XIX.

## PATENTS.

**Manufacture of aralkyl ethers of cellulose.** IMPERIAL CHEM. INDUSTRIES, LTD., D. TRAILL, and A. S. LEVESLEY (B.P. 352,999, 16.4.30).—A cellulose aralkyl (*e.g.*,  $\text{CH}_2\text{Ph}$ ) ether of uniform low viscosity substantially free from highly degraded products is produced by heating a high-viscosity ether with dil. acid ( $\text{HCl}$ ) under pressure (20—50 lb./sq. in.); a wetting agent (oleic acid, glycerol) may be added and a short pretreatment with a swelling agent (aromatic hydrocarbons) given.

C. HOLLINS.

**Purification of cellulose esters.** F. OLSEN (U.S.P. 1,798,270, 31.3.31. Appl., 1.3.29).—The ester, which contains <1% of acid, is pulped in a beating machine, boiled for about 8 hr. in  $\text{H}_2\text{O}$  of approx. 0.25% acidity, and then subjected to alternate boils and washes with fresh  $\text{H}_2\text{O}$  for approx. 12 hr.

B. P. RIDGE.

**Manufacture of higher fatty acid esters of polymeric carbohydrates [*e.g.*, cellulose].** I. G. FAREENIND. A.-G. (B.P. 353,193, 19.6.30. Ger., 4.7.29).—The acylation of cellulose, starch, etc. is affected with acyl halides at 80—100° in presence of <1 equiv. of pyridine or other *tert.* base. The process is especially suitable for the prep. of monoacyl derivatives, the solubility of which is favourably influenced by the acyl halide more than by *tert.* base halide. In the examples, chlorides of coconut oil, stearic and lauric acids are used.

C. HOLLINS.

**Apparatus for the washing and after-treatment of spinning cakes [of artificial silk].** VEREIN. GLANZSTOFF FABR. A.-G. (B.P. 357,470, 13.2.31. Ger., 18.8.30).—Means are described for the automatic mechanical transfer of the cakes, threaded on carrying bars, from the sprinkling to the immersion machine in which washing and after-treatment are carried out.

F. R. ENNOS.

**Manufacture of films and the like from cellulose acetate solutions.** W. H. PEASE (B.P. 357,079, 15.5.30).—A solution of cellulose acetate in 5—9 pts. of Me Et ketone, together with a plasticiser ( $\text{ZnCl}_2$ , oils, fats, or waxes) and, if desired, additional materials such as fireproofing, colouring, or metallising materials, is allowed to evaporate in a closed chamber at 30—75° with a humidity of 50—95%, the outer room temp. being 20—40°.

F. R. ENNOS.

**Treatment of ligneous vegetable materials.** K. G. BERGSTRÖM and A. F. ZENNSTRÖM (B.P. 357,329, 29.7.30. Finl., 18.9.29).—Before digestion by ordinary processes, the raw material, *e.g.*, wood, is treated for 6—24 hr. at 15—20° with aq.  $\text{NaOH}$  or  $\text{Na}_2\text{CO}_3$  (*d* 1.007—1.014) to remove  $\text{AcOH}$ , which latter is recovered. Woods rich in turpentine and resin are further treated first with steam to separate the turpentine, and then with a hot alkaline liquor, *e.g.*, the acetate liquor previously obtained (with addition of a further quantity of alkali), to remove resins.

D. J. NORMAN.

**Opening and preparing artificial staple fibres.** M. F. THOMA (U.S.P. 1,799,399, 7.4.31. Appl., 27.12.26).—The masses are treated at moderate temp. in a bath of  $\text{H}_2\text{O}$  containing an oil soap with a trace of  $\text{H}_2\text{SO}_4$  in it, glycerin,  $\text{NaOH}$ , and pinol, whereby the fibres are loosened, crinkled, softened, and rendered more lustrous.

Alternatively, sulphonated oils, oleic acid, etc. may be used as softeners instead of glycerin. B. P. RIDGE.

**System for cooking fibrous material.** T. L. DUNBAR (U.S.P. 1,798,525, 31.3.31. Appl., 1.8.29).—Hot relief gases from digesters are discharged into a mixing chamber into which cold acid liquor is fed continuously, so that the latter is preconditioned and preheated. The liquor is then withdrawn, forced into an accumulator maintained at superatm. pressure, and used to precook fibrous material. B. P. RIDGE.

**Manufacture of resistant pulp for papermaking.** J. D. RUE, Assr. to CHAMPION FIBRE Co. (U.S.P. 1,798,987, 31.3.31. Appl., 1.9.28).—Partly bleached, raw, alkaline wood pulp is thickened, digested with a caustic alkaline solution, the pulp is separated, part of the residual alkaline solution conc., the remainder mixed with part of the conc. solution with addition of more NaOH, and re-used as alkaline digesting liquid. The remainder of the conc. liquor is used as cooking liquid in the prep. of more alkaline wood pulp for a further cycle of operations. B. P. RIDGE.

**Sizing of papermaking fibres.** J. A. DE CLEW, Assr. to PROCESS ENGINEERS, INC. (U.S.P. 1,799,216, 7.4.31. Appl., 15.11.28).—Streams of dil. solutions of size and alum are either allowed to mix with each other just before entering a stream of pulp stock, or are added along with the stock as it enters the Jordan engine or other rapid mixer. B. P. RIDGE.

**[Treatment of residual waste liquors from] production of chemical pulp.** L. BRADLEY and E. P. McKEEFE, Assrs. to BRADLEY-McKEEFE CORP. (U.S.P. 1,797,678, 24.3.31. Appl., 2.9.26).—The conc. liquors resulting from the digestion of cellulose material, which contain most of their Na either as sulphite or as S-free compounds, are subjected to a furnacing treatment under reducing conditions, thereby forming a product containing mainly  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{S}$ . This is then causticised or sulphited in order to give caustic or sulphite cooking liquors. B. P. RIDGE.

**Panels or backings for oil paintings.** H. WADE. From BAKELITE CORP. (B.P. 357,204, 20.5.30).—A fibrous base, e.g., pulpboard or superimposed sheets of paper or fabric, is impregnated with a reactive synthetic resin and subjected to heat and pressure.

D. J. NORMAN.

**Production of slabs from fibrous material.** E. PLETSCHER (B.P. 357,168, 14.4.30. Switz., 14.4.29).—Wood wool is moistened (dipped into  $\text{H}_2\text{O}$ ) and covered with a thin layer of dry hydraulic cement and/or gypsum. The mass is then stirred and left to harden in a mould under moderate pressure. D. J. NORMAN.

**Manufacture of [high-twist] (A) crêpe yarns and fabrics, (B) textile materials.** BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 357,576—7, 24.5.30).

**Machines for spinning artificial silk.** C. HAMEL A.-G., and E. HAMEL (B.P. 358,045, 27.6.30).

**Production of [lengths of] artificial filaments and the like [to be cut up into staple fibre].** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 357,565, 16.6.30).

**Apparatus [electrically-insulated acid-proof vat and rollers] for manufacture of films or webs from cellulose products.** E. BLEIBLER (B.P. 357,820, 24.10.30. Ger., 26.10.29).

**Drying of sheets [of paper].**—See I. S product. —See VII. Laminated glass. Preventing moisture condensation on glass.—See VIII. Nitro-cellulose solutions.—See XIII. Floor-covering material.—See XIV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Solid-solution theory of dyeing.** W. D. BANCROFT and J. W. ACKERMAN (Proc. Nat. Acad. Sci., 1931, 17, 480—482).—The adsorption of Na alizarate by various hydrous oxides is studied, and it is shown that Witt's theory of solid solutions in dyeing loses its only support if it is assumed that textiles and mordants can act as ionising adsorbents. A. J. MEE.

**Preservation of fish nets. Cellulose formate. Bleaching of sulphite pulp.**—See V. Dyed leather and dermatitis.—See XV.

## PATENTS.

**Manufacture of fabrics exhibiting crêpe effects.** BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 357,084, 7.5.30).—Fabrics containing highly twisted crêpe threads with filaments made from an org. derivative of cellulose are padded, locally if desired, with liquids or solutions which exert a dehydrating action on the cellulose derivative, e.g., abs. EtOH, conc. aq. solutions of  $\text{CaCl}_2$ ,  $\text{ZnCl}_2$ , NaOAc,  $\text{NaH}_2\text{PO}_4$ ,  $\text{NH}_2\text{Me}$ , HCl, etc., and after keeping for, e.g.,  $\frac{1}{2}$  hr. when abs. EtOH is used or  $\frac{1}{2}$ —2 hr. when 30% aq.  $\text{CaCl}_2$  is used, are washed and dried without tension. Alternatively, the threads may be treated with these dehydrating liquors before weaving or even before twisting, the woven fabric made therefrom being subsequently washed with  $\text{H}_2\text{O}$ . D. J. NORMAN.

**[Manufacture of crêped] textile materials.** BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and C. W. NORTH (B.P. 357,169, 16.5.30).—Threads composed of org. derivatives of cellulose are coated either with a layer of cellulose, e.g., by surface hydrolysis, or by coagulation of viscose or cuprammonium solutions thereon, or with a cellulose ester or ether in which the esterifying or etherifying radicals contain OH or  $\text{CO}_2\text{H}$  groups, e.g., cellulose acetate tartrate, and, after highly twisting and weaving with other fibres, the resulting fabric is scoured with water or soap solution to cause swelling of the coating. F. R. ENNOS.

**[Rendering] textiles [water-repellent].** A. NATHANSOHN (B.P. 356,878, 18.7.30. Ger., 14.9.29).—Textiles esterified as described in B.P. 355,256 (B., 1931, 1047) are heated for up to 24 hr. at not above  $120^\circ$  either during or after the drying process, whereby an increased degree of  $\text{H}_2\text{O}$ -repellency is obtained.

A. J. HALL.

**Manufacture of preparations for treatment of textiles.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,989, 9.4.30).—Sulphuric esters of cetyl and other alcohols above  $\text{C}_8$  or their salts are mixed with Na, K, Mg,  $\text{NH}_4$ , or amine  $\text{H}_2\text{O}$ -sol. salts of acids

stronger than  $\text{H}_2\text{CO}_3$ , e.g.,  $\text{Na}_2\text{SO}_4$ ,  $\text{NaOAc}$ ,  $\text{Na}_3\text{PO}_4$ ,  $\text{Na}_2\text{SiO}_3$ ,  $\text{MgSO}_4$ , or  $\text{NaHSO}_4$ . Improved scouring effects (etc.) are obtained. C. HOLLINS.

Colouring gasoline etc.—See II. Wetting etc. agents.—See III. Cleansing agent. S product.—See VII. Fatty acid derivatives.—See XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Poisoning of platinum catalysts for oxidation of ammonia.** J. Y. YEE and P. H. EMMETT (Ind. Eng. Chem., 1931, 23, 1090—1092).—In these experiments an 80-mesh Pt gauze at 800—900° was used, giving a normal conversion of 90—92%. Up to 0.4% addition of  $\text{C}_2\text{H}_2$  was without effect, 0.02 p.p.m. of  $\text{PH}_3$  shows a definite poisoning effect, and with 0.2 p.p.m.  $\text{PH}_3$  this effect permanently damages the gauze. Only traces of  $\text{NH}_3$  were found in the converted gases. The alleged protective effect of adding 0.07%  $\text{H}_2\text{S}$ , which results in an increase of 3—4% in the conversion efficiency, is also found with unpoisoned gauzes in absence of  $\text{PH}_3$ . The increase is more than can be accounted for by the oxidation of the  $\text{H}_2\text{S}$ , and remains unexplained.

C. IRWIN.

**Salt industry of Rio.** M. DA SILVA PINTO and R. R. FILHO (Bol. Serv. Geol. Min. Brasil, 1930, No. 52, 143 pp.).—A fully detailed account, with, especially, a description of the application of phase-rule principles to the isolation and purification of the crude salt, some specimens of which contain only about 85%  $\text{NaCl}$ . Details are given also of the Duflos process of sterilisation, which consists in heating the salt at 200°, whereby in addition the  $\text{MgCl}_2$  present is converted into insol. and non-hygroscopic  $\text{Mg}(\text{OH})\text{Cl}$ , and of the del Vecchio process of sterilisation by electrolysis at a voltage at which only the  $\text{MgCl}_2$  is decomp. The working up of by-products is described, and the commercial and economic aspects of the industry are discussed.

H. F. GILLBE.

**Composition of mechanical separates from ground phosphate rock.** W. L. HILL, H. L. MARSHALL, and K. D. JACOB (Ind. Eng. Chem., 1931, 23, 1120—1124).—Samples of American rock phosphate were ground to pass 100-mesh and then classified according to particle size: (1) above 50  $\mu$ , (2) 5—50  $\mu$ , (3) below 5  $\mu$ . With Florida phosphates the  $\text{SiO}_2$  was found to be conc. in class (1), but with Western phosphates in classes (2) and (3). Fe and Al were found to be conc. in classes (2) and (3), whilst Ca and P were higher in classes (2) and (3) with Florida rock and in class (1) with Western rock. F remained uniform in relation to P content, the samples containing no  $\text{CaF}_2$  as such. There was in most cases a small progressive increase in citrate-sol. phosphate as particle size decreased.

C. IRWIN.

**Continuous precipitation of phosphoric acid extracts with milk of lime.** L. V. VLADIMIROV (Udobr. Urozhai, 1930, 2, 922—926).—An apparatus is described. The use of high-grade  $\text{CaO}$  shortens the reaction time.

CHEMICAL ABSTRACTS.

**Production of soluble phosphates from Chuvash phosphate rock.** S. I. VOLFKOVICH and A. P. METAL-

NIKOV (Udobr. Urozhai, 1931, 3, 61—64).—Phosphate rock (21—22%  $\text{P}_2\text{O}_5$ ) of high carbonate and sesquioxide content (100 pts.) was heated with  $\text{Na}_2\text{CO}_3$  (25 pts.) at 1000—1100°, when 96% of the  $\text{P}_2\text{O}_5$  became sol. in citric acid.

CHEMICAL ABSTRACTS.

**Silico-superphosphate.** A. HOCK (Superphosphat, 1931, 7, 174—176).—Admixture of 4% of kiesulguhr with superphosphate increases its efficiency on light soils and also improves the ease of distribution. The product gives better results than mixtures of superphosphate with basic slag.

A. G. POLLARD.

**Preparation of manganese dioxide.** III. Comparison of properties of samples of different origin. Y. KATO and T. MATSUHASHI (J. Soc. Chem. Ind., Japan, 1931, 34, 312—314 B; cf. A., 1930, 308).—Various samples of  $\text{MnO}_2$  were compared as to (1) catalytic effect in the thermal decomp. of  $\text{KClO}_3$ , measured by temp. rise when heated with a const. electric current; (2) oxidation potential in  $\text{NH}_4\text{Cl}$  solution; (3)  $\text{O}_2$  vapour tension, using  $\text{MnO}_2$  dried at 110°; (4) X-ray photography. The capacities of  $\text{MnO}_2$  dry batteries charged with different samples were compared. The samples fell into two groups of high and low activity.

C. IRWIN.

**Chemical treatment of low-grade chromites.** F. F. WOLF and E. N. PINAEVSKAIA (J. Chem. Ind. Russ., 1931, 8, 949—955).—Saranov chromite, containing about 13%  $\text{Cr}_2\text{O}_3$ , is heated at 1100° with  $\text{Na}_2\text{CO}_3$  and dolomite (proportions 1:0.8:0.8) and the product is extracted with  $\text{H}_2\text{O}$ , when 98% of Cr and 75% of Al present is obtained in the extract, which is neutralised to ppt.  $\text{Al}(\text{OH})_3$ . The calcined ppt. contains 83.2%  $\text{Al}_2\text{O}_3$ , 0.17%  $\text{Cr}_2\text{O}_3$ , 1.4%  $\text{SiO}_2$ , 0.15%  $\text{SO}_3$ , and 13.64%  $\text{Na}_2\text{O}$ ; Fe, Mg, and Ca are absent, so that the product is suitable for the prep. of pure Al salts. The filtrate is worked up for Cr salts in the usual way.

R. TRUSZKOWSKI.

**Processes for decomposing ferrous titanate.** I. Y. KATO and T. MURAKAMI (J. Soc. Chem. Ind., Japan, 1931, 34, 310—311 B).—A résumé of published processes for the dissolution of ilmenite. These fall into 4 classes: (1) those in which both Fe and Ti are dissolved, (2) those in which Fe alone is dissolved after alkali fusion, (3) volatilisation of the chlorides, (4) reduction of Fe with CO.

C. IRWIN.

**Analysis of aluminosilicates.** E. DELFELD (Bull. Soc. chim. Belg., 1931, 40, 427—436).—Various methods are reviewed, and details are given of the most satisfactory procedure for the determination of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{CaO}$  and  $\text{MgO}$ , alkalis, and  $\text{TiO}_2$ . The ordinary method based on fusion with  $\text{Na}_2\text{CO}_3$ — $\text{K}_2\text{CO}_3$  is said to be sufficiently accurate for all industrial analyses.

H. F. GILLBE.

**Commercial possibilities of metallic peroxides as a source of hydrogen peroxide.** L. W. BLUNDELL (Trans. Inst. Chem. Eng., 1930, 8, 191—201).—A plant is described which is capable of giving a daily output of 200 gals. of 12-vol.  $\text{H}_2\text{O}_2$ , using  $\text{BaO}_2$  and  $\text{H}_3\text{PO}_4$ , with recovery of the latter. This acid is preferred owing to its stabilising effect on the product and the ease of filtration of its Ba salt. The purity of the raw materials and the fine grinding of the  $\text{BaO}_2$  are

important. On the top floor are storage-bin hoppers and mill for  $\text{BaO}_2$ , on the next the mixers and acid tanks, and on the ground floor filter-presses etc. The mixers in which  $\text{BaO}_2$  is hydrated are of cast Fe with heavy cast-Fe blades, the reaction vessels being of pitch-pine and having two concentric panels rotating in opposite directions. A wooden frame-press is used for  $\text{Ba}_3(\text{PO}_4)_2$  filtration. Al is the only metal suitable for contact with  $\text{H}_2\text{O}_2$  solutions as almost all others catalyse decomp. The  $\text{Ba}_3(\text{PO}_4)_2$  is digested with  $\text{H}_2\text{SO}_4$  in an enamelled cast-Fe jacketed pan, settled, and the clear liquor is siphoned off and conc. in a similar pan. The main reaction vessel is kept at  $10$ – $15^\circ$  (by cooling coils) during the working of a batch of  $\text{BaO}_2$ . The  $\text{BaO}_2$  is fed in as a cream which must be quite free from grit. The neutralisation of the filter-pressed  $\text{H}_2\text{O}_2$  solution is effected with  $\text{Ba}(\text{OH})_2$  and  $\text{H}_2\text{SO}_4$  and the liquor is again filter-pressed.  $\text{NH}_4\text{AcPh}$  may be added as a stabiliser. Outlay and working costs are estimated.

C. IRWIN.

**Thermal decomposition of hydrogen sulphide. Decomposition by the electric arc in presence or absence of carbon dioxide.** P. FERRERO and J. CORBAZ (Helv. Chim. Acta, 1931, 14, 912–935).—The decomp. of a stream of  $\text{H}_2\text{S}$  by the electric arc has been studied from the viewpoint of technical requirements. Electrodes of Pt or Cu only are admissible. With pure  $\text{H}_2\text{S}$  the yield of S was 50–60% according to the speed of flow, the energy yield only 28%. Dilution with  $\text{N}_2$  (25%  $\text{H}_2\text{S}$ ) increased the chemical yield considerably and diminished the energy yield slightly. In presence of  $\text{CO}_2$  (70%) the decomp. of  $\text{H}_2\text{S}$  was nearly complete, but the energy yield still low. It is concluded that the conditions which would make the process of commercial value are not realisable in practice. F. L. USHER.

**Fe oxide pigments.**—See XIII. **Roasting phosphorites for plants.**—See XVI.

## PATENTS.

**Purification of solutions [acids] by electrolytic means.** I. G. FARBENIND. A.-G. (B.P. 357,630, 26.6.30. Ger., 28.6.29).—As or Hg are removed from  $\text{H}_3\text{PO}_4$ ,  $\text{H}_2\text{SO}_4$ , or  $\text{AcOH}$  by passing the acid slowly through a trough containing a series of transverse gauze cathodes between which are disposed strip or wire anodes; between these and the cathodes is passed a current at 2–3 volts. In the case of  $\text{H}_3\text{PO}_4$  Cu-gauze cathodes and Pt-wire anodes are used, the  $\text{As}_2\text{O}_3$  being reduced to finely-divided As and subsequently removed by filtration.

A. R. POWELL.

**Production of salts of hydrofluosilicic acid.** M. BUCHNER, Assoc. of F. HILDERBRANDT (B.P. 357,922, 23.2.31. Ger., 24.2.30).—A fluoride is mixed with, or suspended in, a solution containing 30 wt.-% or upwards of  $\text{H}_2\text{SiF}_6$ , and into the mixture is introduced  $\text{SiF}_4$ , the reaction being effected with heat or under pressure, if desired. The function of the  $\text{H}_2\text{SiF}_6$  is partly catalytic.

W. J. WRIGHT.

**Production of hydrocyanic acid.** GES. F. KOHLENTECH. M.B.H. (B.P. 356,724, 22.5.30. Ger., 7.6.29. Addn. to B.P. 356,190; B., 1931, 1009).—The N values of the reaction gases (obtained by way of the modified Raschen

process) are used for the manufacture of the  $\text{HNO}_3$  required in the production of HCN from  $\text{NH}_4\text{CNS}$ . The residual reaction solution is neutralised with  $\text{NH}_3$  to produce fertilising materials.

F. YEATES.

**Treatment of brine.** W. R. COLLINGS, Assr. to DOW CHEM. CO. (U.S.P. 1,796,920, 17.3.31. Appl., 12.9.28).—Brine containing  $\text{MgCl}_2$  and  $\text{CaCl}_2$  is evaporated to obtain NaCl crystals and a solution containing about 33%  $\text{CaCl}_2$  and 11%  $\text{MgCl}_2$ , which is further evaporated to yield crystals of tachydrite and a mother-liquor containing 53%  $\text{CaCl}_2$  and 5%  $\text{MgCl}_2$ . This liquor is treated with  $\text{Ca}(\text{OH})_2$  to separate the Mg as  $\text{Mg}(\text{OH})_2$ , leaving a pure  $\text{CaCl}_2$  solution. The tachydrite is dissolved in boiling  $\text{H}_2\text{O}$  and the solution cooled to  $30^\circ$  to separate  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  and leave a  $\text{CaCl}_2$  mother-liquor (*d* 1.24), which is returned to the tachydrite crystalliser.

A. R. POWELL.

**Obtaining sodium sulphate from natural deposits.** A. T. W. WARNKEN, Assr. to E. JOHNSON (U.S.P. 1,798,993, 31.3.31. Appl., 4.10.29).—Natural beds of  $\text{Na}_2\text{SO}_4$  are leached with shallow streams of  $\text{H}_2\text{O}$  during the day, the leach liquors accumulating in a reservoir in the bed in which  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  crystallises during the night.

A. R. POWELL.

**Leaching of phosphate rock.** KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G. (B.P. 357,580, 23.6.30. Swed., 11.7.29).—A liquor containing  $\text{H}_3\text{PO}_4$  and, if desired,  $\text{NH}_4$  phosphates, produced, *e.g.*, by treating phosphate rock with  $\text{H}_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$ , is added during the treatment of phosphate rock with  $\text{HNO}_3$ , and the Ca is precipitated by the addition of  $\text{K}_2\text{SO}_4$ , part of it being added during, and the rest after, the completion of the main reaction. Some of the final solution, after removal of the  $\text{CaSO}_4$  and part of the  $\text{KNO}_3$  as described in B.P. 338,079 (B., 1931, 61), may also be added in the initial stage.

L. A. COLES.

**Treatment of phosphate rock.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 357,814, 21.10.30. Norw., 28.10.29).— $\text{Ca}(\text{NO}_3)_2$  is crystallised without removal of insol. impurities from the liquor obtained by treating phosphate rock with  $\text{HNO}_3$ , and the crystals are removed on a wide-mesh filter or wire screen so that some of the insol. material passes through. The insol. material is removed separately from the mother-liquor and, after dissolution, from the  $\text{Ca}(\text{NO}_3)_2$  by filtration or centrifuging. Dil. waste liquors obtained during the process are used for irrigation in a plant for manufacturing the  $\text{HNO}_3$  for use in the process.

L. A. COLES.

**Preparation of a cleansing agent [containing trisodium phosphate].** I. G. FARBENIND. A.-G. (B.P. 357,533, 12.6.30. Ger., 15.7.29).—Siliceous ferrophosphorus, obtained in the electrothermal prep. of P, is heated to redness with  $\text{Na}_2\text{CO}_3$ ; the product is then leached with  $\text{H}_2\text{O}$  and the solution evaporated to dryness. [Stat. ref.]

W. J. WRIGHT.

**Preparation of detergents.** C. A. JENSEN. From ELECTRIC SMELTING & ALUMINUM CO. (B.P. 357,224, 18.6.30).— $\frac{3}{4}$ – $1\frac{1}{2}$  mol. of alkali carbonate is fused with 1 mol. of  $\text{SiO}_2$ ; sufficient alkali chloride may be added to remove impurities containing Fe or Al.

E. LEWKOWITSCH.

**Manufacture of potassium phosphate.** R. D. PIKE (U.S.P. 1,799,479, 7.4.31. Appl., 18.6.28).—A mixture of KCl (220 pts.),  $\text{H}_3\text{PO}_4$  containing 45%  $\text{P}_2\text{O}_5$  (158 pts.), and  $\text{H}_2\text{SO}_4$  (114 pts.,  $d$  1.7) is heated first at  $770^\circ$  in a rotary furnace until HCl ceases to be evolved, and then, after addition of coal (66 pts.), at  $850^\circ$  until  $\text{SO}_2$  is expelled. The resulting clinker is dissolved in  $\text{H}_2\text{O}$ , the insol. mud removed, and the solution treated with  $\text{H}_3\text{PO}_4$  to give  $\text{KH}_2\text{PO}_4$ , which is crystallised.

A. R. POWELL.

**Manufacture of alkaline-earth oxides.** I. G. FARBENIND. A.-G. (B.P. 357,795, 1.10.30. Ger., 1.10.29).— $\text{BaCO}_3$  or  $\text{SrCO}_3$  is mixed with about 6% of free C (e.g., soot), or of substances containing it, and heated in a rotary furnace at above  $700^\circ$  in an atm. of  $\text{H}_2$ .

H. ROYAL-DAWSON.

**Manufacture of barium hydroxide.** F. RÜSBERG and G. CLAUSS, Assrs. to RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,799,989, 7.4.31. Appl., 24.5.28. Ger., 22.6.27).— $\text{Ba}_2\text{SiO}_4$  produced by heating  $\text{BaSO}_4$  and  $\text{SiO}_2$  in steam is ground with  $\text{H}_2\text{O}$  in a ball-mill until reduced to an almost colloidal suspension and then boiled under reflux until the  $\text{Ba}_2\text{SiO}_4$  is hydrolysed and the residue settles readily. The solution contains 32–34% of  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ , which separates on cooling.

A. R. POWELL.

**Production of anhydrous calcium sulphate.** C. B. DURGIN, A. B. GERBER, and P. LOGUE, Assrs. to SWANN RES. INC. (U.S.P. 1,783,417, 2.12.30. Appl., 21.1.26).—To prevent discoloration of  $\text{CaSO}_4$  during dehydration, 1–5% of  $\text{CaHPO}_4$  is added to gypsum and the mixture calcined at  $760^\circ$ .

A. R. POWELL.

**Production of bleaching powder.** I. G. FARBENIND. A.-G. (B.P. 357,933, 14.3.31. Ger., 14.3.30).—About 3–7% of a vaporisable liquid, e.g.,  $\text{CCl}_4$ ,  $\text{CHCl}_3$ , or a mixture of these, which is inert towards  $\text{Cl}_2$ ,  $\text{Ca}(\text{OH})_2$ , or  $\text{CaOCl}_2$ , is added to the  $\text{Ca}(\text{OH})_2$  before chlorination, so as to restrict rise of temp. and formation of lumps, the inert liquid being subsequently expelled by heating in vac.

W. J. WRIGHT.

**Production of dry, high-grade calcium hypochlorite.** I. G. FARBENIND. A.-G. (B.P. 357,798, 6.10.30. Ger., 28.10.29).— $\text{Ca}(\text{OCl})_2$  solution with a low  $\text{CaCl}_2$  concentration is rendered slightly alkaline by addition of NaOH and then rapidly evaporated to dryness by spraying in a current of hot air. The evaporation may be conducted in two stages, incompletely anhyd. crystals being produced first and these being subsequently dehydrated.

W. J. WRIGHT.

**Manufacture of calcium magnesium chloride.** W. R. COLLINGS and J. J. SHAFER, Assrs. to DOW CHEM. CO. (U.S.P. 1,796,885–6, 17.3.31. Appl., 4.11.29).—(A) A solution containing  $\text{MgCl}_2$  and  $\text{CaCl}_2$  in a ratio  $< 2:1$  is added to a mother-liquor saturated with tachydrate and  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  and the solution is evaporated at  $45$ – $93^\circ$ , whereby a crystal meal consisting of a mixture of  $\text{CaCl}_2 \cdot 2\text{MgCl}_2 \cdot 12\text{H}_2\text{O}$ ,  $\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ , and  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  is obtained. (B) A solution containing  $\text{MgCl}_2$  and  $\text{CaCl}_2$  in a ratio of  $0.5$ – $0.1$  is added to a mother-liquor saturated with  $\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  and the mixture is evaporated at  $152$ – $177^\circ$ , whereby a mixture of

$\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  is obtained as a non-caking crystal meal. (Cf. U.S.P. 1,738,492; B., 1930, 373.)

A. R. POWELL.

**Preparation of a dehydrating agent [barium magnesium perchlorate].** G. F. SMITH (U.S.P. 1,798,175, 31.3.31. Appl., 13.9.26).—A solution containing  $\text{Mg}(\text{ClO}_4)_2$  and a larger quantity of  $\text{Ba}(\text{ClO}_4)_2$  is evaporated and the salt mixture dried at  $300^\circ$  to form a granular, porous dehydrating agent. Alternatively,  $\text{Ba}(\text{ClO}_4)_2$  sprayed with a solution of  $\text{Mg}(\text{ClO}_4)_2$ , or a mixture of  $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$  and  $\text{Mg}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$  crystals, is heated at  $250$ – $300^\circ$ .

A. R. POWELL.

**Manufacture of chlorides of the alkaline-earths from their sulphates.** T. LICHTENBERGER and L. KAISER (U.S.P. 1,798,091, 24.3.31. Appl., 9.2.27. Ger., 27.10.25).—The alkaline-earth sulphate is introduced together with powdered coke into a fused bath of the corresponding chloride, and a current of  $\text{Cl}_2$  is passed through the mixture, followed at the end of the reaction by HCl gas to convert any oxide into chloride. The  $\text{S}_2\text{Cl}_2$  produced in the reaction may be hydrolysed by introducing steam into the reaction vessels.

A. R. POWELL.

**Manufacture of cyanamides of the alkaline-earth metals.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 358,276, 6.10.30).—An alkaline-earth or Mg oxide or carbonate is treated at  $300$ – $900^\circ$  (preferably  $350$ – $600^\circ$ ) with  $(\text{CN})_2$  or a mixture of it with, e.g.,  $\text{N}_2$ ,  $\text{H}_2$ , CO, or  $\text{NH}_3$ , to effect the reaction:  $\text{XO} + (\text{CN})_2 = \text{XCN}_2 + \text{CO}$ , or  $\text{XCO}_3 + (\text{CN})_2 = \text{XCN}_2 + \text{CO} + \text{CO}_2$ .

L. A. COLES.

**Stable alkaline ferric [hydroxide] solution.** W. J. HUFF, D. L. JACOBSON, and W. H. HILL, Assrs. to KOPPERS CO (U.S.P. 1,786,490, 30.12.30. Appl., 17.7.24).—A solution of invert sugar or of dextrose is heated with  $\text{Na}_2\text{CO}_3$  to boiling for a few min. and is then cooled rapidly, treated with a solution of  $\text{FeSO}_4$ , and oxidised with a current of air until the  $\text{Fe}(\text{OH})_2$  first precipitated redissolves as colloidal  $\text{Fe}(\text{OH})_3$ .

A. R. POWELL.

**Precipitation of aluminium fluoride.** J. E. MORROW, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,797,994, 24.3.31. Appl., 20.9.28).—Dil.  $\text{AlF}_3$  solutions, obtained by decomp. of kaolin, bauxite, or clay with  $\text{Hf}$  or  $\text{H}_2\text{SiF}_6$ , are heated at  $90$ – $95^\circ$  with a small quantity (e.g., 1%) of finely-divided dry  $\text{AlF}_3$  for several hr., whereby 80–90% of the contained  $\text{AlF}_3$  is precipitated in a granular form.

A. R. POWELL.

**Treatment of greensand.** A. LAMBERT, Assr. to COSMIC ARTS, INC. (U.S.P. 1,797,002, 17.3.31. Appl., 28.11.24).—Greensand is decomposed with HCl,  $\text{H}_2\text{SO}_4$ , or  $\text{HNO}_3$  and, after removal of the  $\text{SiO}_2$ , the solution is neutralised with  $\text{Na}_2\text{CO}_3$ , the  $(\text{Fe}, \text{Al})(\text{OH})_3$  removed, and the Na precipitated with  $\text{NH}_3$  and  $\text{CO}_2$ , leaving a solution from which a mixed  $\text{NH}_3$ -K fertiliser is obtained by evaporation.

A. R. POWELL.

**Production of substantially pure chemical compounds [oxides and anhydrous chlorides] or metals.** A. S. BURMAN and I. RENNERFELT (B.P. 358,000, 20.6.30. Swed., 25.6.29).—Solutions containing metal chlorides obtained by dissolving crude material in aq. HCl are evaporated to dryness in the presence of

a sol. org. compound, *e.g.*, a sugar, and the residual mass is calcined to expel  $\text{HCl}$  and to yield a porous mixture of C and a metal oxide. *E.g.*, in the prep. of pure  $\text{Al}_2\text{O}_3$  from bauxite or clay, the porous mixture is heated to redness in a reducing atm., Fe etc. being removed by extraction with aq.  $\text{HCl}$  or by treatment with  $\text{Cl}_2$ .

L. A. COLES.

**Treatment of [recovery of light silica from] siliceous materials.** P. SPENCE & SONS, LTD., and A. KIRKHAM (B.P. 357,993, 28.3.30).—Siliceous residues obtained, *e.g.*, in the extraction of clays, bauxite, etc. with acids are dissolved at 90–100° in  $\text{Na}_2\text{CO}_3$  solution, *d* 1.20–1.28, in quantities such that  $\text{SiO}_2 : \text{Na}_2\text{CO}_3 = 1 : 5$ ; after removal of insol. material, which contains  $\text{TiO}_2$ , the solution is cooled rapidly to yield a flocculent ppt. of hydrated  $\text{SiO}_2$  which is washed with  $\text{H}_2\text{O}$  and dil. mineral acids, dried, and sieved.

L. A. COLES.

**Production of titanium dioxide.** J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (U.S.P. 1,795,467, 10.3.31. Appl., 2.2.28).—Ilmenite is heated with conc.  $\text{H}_2\text{SO}_4$ , the sulphates are dissolved in  $\text{H}_2\text{O}$ , and the solution is reduced with scrap Fe. The reduced solution is then evaporated to remove the greater part of the Fe as  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , leaving a solution containing about 200 g. of  $\text{TiO}_2$ , 20–25 g. of Fe, and 500–550 g. of  $\text{H}_2\text{SO}_4$  per litre. The boiling solution is then added slowly with vigorous agitation to an equal vol. of boiling  $\text{H}_2\text{O}$  and boiling is continued for some hr. until 90–95% of the  $\text{TiO}_2$  is precipitated in a dense flocculent form.

A. R. POWELL.

**Production of titanium compositions [colloidal titanium dioxide].** C. DE ROHDEN, Assr. to COMMERCIAL PIGMENTS CORP. (U.S.P. 1,797,760, 24.3.31. Appl., 10.2.28).— $\text{Ti}(\text{OH})_4$ , produced by hydrolysis of  $\text{Ti}(\text{SO}_4)_2$  solutions, is washed and treated with aq.  $\text{NH}_3$  to neutralise adsorbed acid, then thoroughly washed to remove salts. The resulting  $\text{Ti}(\text{OH})_4$  is peptised by treatment with 35% aq.  $\text{NH}_3$ . A colloidal  $\text{TiO}_2$  may also be prepared by grinding calcined  $\text{TiO}_2$  with 1–2% aq.  $\text{NH}_3$ .

A. R. POWELL.

**Recovery of titanium from its ores.** O. T. COFFELT (U.S.P. 1,795,361, 10.3.31. Appl., 5.7.28).—Finely-ground ilmenite is digested with 75–90%  $\text{H}_2\text{SO}_4$  and the resulting Ti and Fe sulphates are dissolved in  $\text{H}_2\text{O}$ . The solution is reduced with Fe or Zn until part of the  $\text{Ti}^{IV}$  is reduced to violet  $\text{Ti}^{III}$ , diluted with  $\text{H}_2\text{O}$ , and boiled with  $\text{NH}_4\text{Cl}$  equal in wt. to the Ti present. The resulting ppt. of hydrated  $\text{TiO}_2$  settles well and is readily freed from Fe by washing with hot  $\text{H}_2\text{O}$ .

A. R. POWELL.

**Extraction of zirconia from ores.** C. O. TERWILIGER, Assr. to WARD LEONARD ELECTRIC CO. (U.S.P. 1,796,170, 10.3.31. Appl., 18.5.29).—Baddeleyite is ground at 120° with  $\text{H}_2\text{SO}_4$  (*d* 1.45) until it passes 350-mesh, whereby most of the sol. basic impurities are dissolved. The residual  $\text{ZrO}_2$  is washed by decantation, boiled with  $\text{HCl}$  (*d* 1.14), again washed, boiled with  $\text{HCl}$  and  $\text{Cl}_2$ , washed, and digested with 48%  $\text{HF}$  and a little  $\text{H}_2\text{SO}_4$  at 110° until the  $\text{SiO}_2$  is expelled; the insol.  $\text{ZrO}_2$  is washed free from acid and ignited to expel  $\text{SO}_3$ .

A. R. POWELL.

**Non-corroding composition [antifreeze solution].** J. W. ORELUP and O. I. LEE (U.S.P. 1,795,883, 10.3.31.

Appl., 29.10.25).—An antifreeze solution for the radiators of internal-combustion engines comprises a 38% solution of  $\text{CaCl}_2$  in which is dispersed 0.04–0.05% of a mixture of 50%  $\text{V}_2\text{O}_5$ , 30% U Th hydroxides, and 20%  $\text{ZrO}_2$ .

A. R. POWELL.

**Manufacture of monoxanthogens [monosulphur-dicarbothionates].** G. S. WHITBY, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,796,972, 17.3.31. Appl., 17.5.28).—By the process already described by Whitby and Greenburg (A., 1930, 320), secondary alkyl monosulphurdicarbothionates (especially the  $\text{Pr}^s_2$  compound) are claimed.

A. R. POWELL.

**Preparation of lead tetra-alkyl.** H. W. DAUDT, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,798,593, 31.3.31. Appl., 20.1.27).—A suspension of  $\text{PbCl}_2$  and Mg turnings in anhyd.  $\text{Et}_2\text{O}$  with MeI or I as a catalyst is treated with an excess of  $\text{EtCl}$  for 12 hr. at 28–40° under reflux. The product is treated with  $\text{H}_2\text{O}$  and neutralised with  $\text{NaOH}$ ; the  $\text{Et}_2\text{O}$  is removed by distillation and the  $\text{PbEt}_4$  recovered by steam-distillation.

A. R. POWELL.

**Production of hydrogen.** J. S. BEEKLEY, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,799,452, 7.4.31. Appl., 27.7.28).—A mixture of steam and  $\text{CH}_4$  in the ratio 11 : 1–29 : 1 is passed at 350–400° under 20 atm. over a catalyst comprising  $\text{MgO}$  impregnated with  $\text{NiO}$  or a mixture of  $\text{MgO}$  and pumice impregnated with  $\text{NiO}$ ,  $\text{Ce}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3$ . Since the  $\text{CO}_2$  produced in the reaction is absorbed by the  $\text{MgO}$ , the catalyst must be regenerated occasionally by calcining it to expel  $\text{CO}_2$ .

A. R. POWELL.

**Manufacture of a sulphur product.** C. ELLIS, Assr. to ELLIS-FOSTER CO. (U.S.P. 1,795,364, 10.3.31. Appl., 24.11.24).—A colloidal dispersion of S is obtained by grinding 90–95 pts. of S with 10–5 pts. of bentonite and 1 pt. of  $\text{Ca}(\text{OH})_2$  in 600 pts. of  $\text{H}_2\text{O}$ . The product may be dried at a temp. below that at which S sinters and is then suitable for incorporation into rubber mixes, for the manufacture of moulded articles, and for waterproofing felt or paper.

A. R. POWELL.

**Containers for acids. Separating mixed gases. Catalysing gaseous reactions.**—See I. Grinding org. peroxides. Trialkyl phosphates.—See III. Zn-bearing material. Salt bath for Ag. Cu extraction.—See X. Electrolysis of  $\text{H}_2\text{O}$ . Carrying out chemical reactions.—See XI. Fertilisers.—See XVI. Explosives containing  $\text{Ca}(\text{NO}_3)_2$ .—See XXII.

## VIII.—GLASS; CERAMICS.

**Chemical-resisting glass for laboratory vessels.** I. J. KITAIGORODSKI and S. M. KUROVSKI (Trans. State Inst. Test. Building Mat., Moscow, 1930, No. 31, 3–43).—Glasses having the composition  $\text{SiO}_2$  74, 74;  $\text{B}_2\text{O}_3$  5.5, 3;  $\text{Al}_2\text{O}_3$  3, 3;  $\text{CaO-MgO}$  10, 10;  $\text{Na}_2\text{O}$  7.5, 10, or sand 71.3, 71.3; kaolin 7.7, 7.7; dolomite 19.65, 19.65;  $\text{H}_3\text{BO}_3$  9.8, 5.35; soda 13.12, 18.7; salt-petre 4.0, 4.0 (temp. 1350°) were resistant to chemical attack and change of temp.

CHEMICAL ABSTRACTS.

**Solubility of glasses in water in relation to their composition.** S. MORIYASU (J. Soc. Chem. Ind., Japan, 1931, 34, 314–317 B).—Knapp's formula (B.,



1927, 12) for the solubility of Pb glass disagrees with Tabata's experimental results. It was found that, using glass grains of 0.25–0.40 mm. diam. heated in  $H_2O$  for 1 hr. at 99° followed by conductometric titration, fairly concordant results were easily obtained. A series of tests by the same method were made in  $Na_2O$ – $CaO$  glasses containing 3–17%  $CaO$  and 65–75%  $SiO_2$ , and the results are expressed as "isokals" (composition curves of glasses of equal solubility). A relationship between composition and  $d$  is also worked out.

C. IRWIN.

**Silica grinding.** R. L. CAWOOD (Trans. Ceram. Soc., 1931, 30, 295–300).—A grinding unit, consisting of a Patterson 8ft. by 10 ft. continuous-charge and -discharge ball-mill in conjunction with a 14-ft. centrifugal air separator, as installed at the Potters' Mining & Milling Co., U.S.A., is described. The capacity is 40 tons per day (99% passing 200-mesh) at a power cost of 3s. 4d. per ton compared with 8s. per ton in the old plant. The labour and maintenance costs are reduced by half, and there is complete elimination of dust. J. A. SUGDEN.

**Effect of smelter atmospheres on the quality of dry-process enamels for cast iron.** A. I. ANDREWS and H. W. ALEXANDER (Univ. Ill. Eng. Exp. Sta. Bull. 1931, No. 227, 16 pp.).—Reducing atm. were deleterious to all enamels except leadless Sn enamel, causing formation of metallic beads and loss of opacity. Reducing atm. with high  $SO_2$  content were deleterious to all enamels.

CHEMICAL ABSTRACTS.

**Preparation of "brilliant gold" and "silver" [lustres].** V. E. TISCHTSCHENKO and S. A. SMIRNOV (J. Appl. Chem., Russia, 1930, 3, 1041–1054).—"Brilliant gold" for ceramic use is prepared by a modification of the Chemnitz method. The prep. of "brilliant silver" (Pd, not Ag) is described.

CHEMICAL ABSTRACTS.

**Influence of iron oxide on the sintering and some technically important properties of steel-works' dolomite.** O. KRAUSE and H. GUHR (Feuerfest, 1931, 7, 129–136).—The porosity of dolomite bricks with <5%  $FeO$  begins to decrease after firing above 1200°, whereas that of bricks made from dolomite with 9%  $FeO$  commences to decrease above 1000°. The  $d$  rises almost linearly with the burning temp., except for a pronounced break at 600–800°, up to 1000°, after which it remains const. The compression strength of dolomite bricks is considerably increased by addition of  $FeCO_3$  to the mixture and the burning temp. at which max. strength is reached is appreciably lowered; thus for dolomite with 1%  $FeO$  firing at 1450° produces max. strength, whereas a greater strength is obtained at 1050° with dolomite containing 9%  $FeO$ . The softening point under load decreases almost linearly with increase in  $FeO$  from 1210° with 4% to 1050° with 25%  $FeO$ . Irrespective of the Fe content, all dolomite bricks have a large plastic range. Heating curves of dolomite show two breaks, viz., at 630–660° and 740–820°, whereas lines in the X-ray spectra corresponding with  $MgO$  and  $CaO$  appear only above 800° and 1000° respectively, although pure  $MgCO_3$  shows lines of  $MgO$  above 500°. At above 1000° burnt dolomite containing  $FeO$  gives a spectrogram containing lines due to a ferrite which would seem to be formed by a reaction in the solid state;

as  $Mg$  and  $Ca$  ferrites melt at about 1200°, the formation of these compounds explains the lowering of the sintering temp. produced by  $FeO$ .

A. R. POWELL.

**Suitability of various refractory materials for lead-refining furnaces.** E. R. THEWS (Feuerfest, 1931, 7, 84–86).—The special requirements of Pb-refining furnaces, in particular the action of the molten Pb and hot Sb slags on the refractories, are briefly outlined. Short descriptions are given of the properties of a number of refractories, the use of which, however, is limited by their high price. A comparison is made between high-grade fireclay and magnesite bricks, and preference is given to the former on account both of their technological properties and low cost. F. SALT.

**Electrical resistivity of refractory materials at elevated temperatures.** H. M. KRANER (Ind. Eng. Chem., 1931, 23, 1098–1102).—Discordant results in reported values for the resistances of ceramic furnace linings are due to variations in chemical composition, fire treatment, grain size, etc. All ceramic materials contain a glass bond, the conductivity of which is determined by the mobility of the ions and the viscosity of the melt. The resistance therefore increases with ionic size, and a K glass is more resistant than a Na glass. Al increases viscosity and resistance. The variations in composition of the systems  $Al_2O_3$ – $SiO_2$  and  $MgO$ – $Al_2O_3$ – $SiO_2$  with variations in heat-treatment are discussed.  $MgO$  is used in preference to alkalis as a flux for high-temp. ceramic materials. Resistances are given for porcelain, clay-quartz mixtures, etc. over the range 0–600° and for an unfired mixture of clay, corundum, and zircon. These indicate the very serious effect of the presence of combined  $H_2O$ . The effect of a 5% bentonite bond in reducing the resistance of zircon is shown.

C. IRWIN.

**Heat losses through furnace walls. Pyrometer-tube walls.**—See I. Analysis of aluminosilicates.—See VII.

#### PATENTS.

**Manufacture of laminated glass.** F. B. DEHN. From LIBBEY-OWENS-FORD GLASS Co. (B.P. 358,149, 11.7.30).—A solution of dry gelatin in a polyglycol (ethylene glycol) and a polyhydric alcohol (glycerol), containing, if desired, a little  $CaCl_2$ ,  $BaCl_2$ ,  $MnCl_2$ , or  $CeCl_3$ , prepared by agitation of the constituents at 90–100°, is used as adhesive for combining glass and cellulose sheets at raised temp. and pressure.

L. A. COLES.

**Preventing the condensation of moisture on glass windows and the like.** G. SCHWARTZ (B.P. 357,892, 30.12.20. Switz., 24.11.30).—The surface of the glass is covered with a transparent layer of regenerated cellulose.

H. ROYAL-DAWSON.

**Tunnel kiln and process of heating ceramic articles.** I. HARTER, A. M. KOHLER, and F. H. NORTON, Assrs. to BABCOCK & WILCOX Co. (U.S.P. 1,799,296, 7.4.31. Appl., 31.12.26).—The kiln is operated in three zones for cooling, firing, and preheating, respectively, and a gradual drop in pressure is maintained from the first to the last zone, i.e., countercurrent to the goods. The fuel is admitted to a raised combustion and reverberatory chamber above the middle zone. The trucks

for the goods are not provided with wheels, but slide on metallic rails, and the goods are placed on them in such a manner that there is a gas passage on two sides of each article. In the preheating zone the gases are caused to flow with sufficient speed to prevent laminar flow, *i.e.*, above an average of 15 ft./sec. A temp. of 1538° is exceeded in the firing zone. B. M. VENABLES.

**Manufacture of refractory articles [graphite crucibles].** CARBORUNDUM CO., LTD. From F. J. TONE (B.P. 357,611, 27.5.30).—A mix containing flake graphite or similar material is shaped under hydraulic pressure in a mould inverted to impart max. strength to the upper portions, and the mould is jolted to cause the particles to lie in planes at right angles to the side walls, thereby increasing the thermal conductivity in that direction. L. A. COLES.

[Sealing-in] Ni alloys [for glass].—See X.

### IX.—BUILDING MATERIALS.

**Colloidal hardening and the hardening of cement.** H. KÜHL [with F. THILO, C. GOTTFRIED, and W. BÜSSEM] (Z. physikal. Chem., Bodenstein Festb., 1931, 126—130).—Various specimens of cement, after curing for 4 weeks, showed no trace of cryst. structure when examined microscopically or by means of X-rays. The hardening of cement must therefore be a colloidal process.

H. F. GILLBE.

**Chemical examination of damp brickwork.** G. SCHMIDT (Chem.-Ztg., 1931, 55, 558—559).—Samples of brickwork from a damp building were examined for H<sub>2</sub>O content, H<sub>2</sub>O absorption, chemical composition, etc. Bricks from damp portions of the walls contained Cl', N<sub>2</sub>O<sub>5</sub>, and CaO, whereas the dry bricks contained only traces of these. The H<sub>2</sub>O absorption was proportional to the Cl' content. Bricks from old stables had been used in building the house in question.

F. SALT.

**Distribution of ether extractive in slash pine [*Pinus caribæa*].** E. F. KURTH and E. C. SHERRARD (Ind. Eng. Chem., 1931, 23, 1156—1159).—In all cases the percentage of oil and extract increases from the bark to the centre of the tree. It also decreases with increasing height at the centre of the trunk, but at the outside this decrease is small. The acid val. increases with the age of the wood and is highest for the centre of the butt log, whilst the reverse obtains for sap. val. The amount of unsaponifiable matter varies with the acid val.; it is greatest at the centre of the trunk and decreases with increasing height. Trees of rapid growth have more extractable matter than those of slow growth. The extract from the outer rings is a yellow liquid, whilst that from the centre is solid. The difference between the extracts from the sap- and heart-woods, respectively, may be caused by a greater dilution of the resin acids in the former by esters. B. P. RIDGE.

**Manufacture of wallboard and [heat]-insulating sheets from wood and pulp wastes.** K. WEILER (Papier-Fabr., 1931, 29, 549—554).—A detailed account is given of current American practice in the manufacture of such products in continuous lengths. Diagrams of machinery and details of physical properties of the boards are given. T. T. POTTS.

### PATENTS.

**Production of material more particularly for building and street-construction purposes.** ALUMINIUMERZ BERGBAU U. IND., A.-G. (B.P. 357,293, 2.7.30. Addn. to B.P. 356,797; B., 1931, 1052).—The bauxite may be pretreated by drying, calcining, sintering, or fusing, and further substances, *e.g.*, bauxite cement, clay, limestone, trass, SiO<sub>2</sub>, dolomite, may be added to the mixture. L. A. COLES.

**Bituminous concrete.** E. R. TAYLOR (B.P. 357,282, 1.7.30).—A mineral aggregate is mixed with a fine filler containing sufficient calcined gypsum or plaster of Paris to combine with H<sub>2</sub>O in the aggregate, and a suitable quantity of "cut-back" bitumen is then stirred in. L. A. COLES.

**Material for covering bituminous road surfaces.** O. DÖNIG (B.P. 357,699, 26.7.30. Ger., 27.7.29).—Fragments of stone, blast-furnace slag, or gravel are impregnated with 4—5 pts. per 10<sup>3</sup> of bituminous material. L. A. COLES.

**Heat-insulating walls.** F. FÖRSTER and R. KRAFFT (B.P. 357,356, 2.9.30).—A wooden framework supports outer and inner metallic plates, also a number of intermediate plates which may also be of metal.

B. M. VENABLES.

**Manufacture of road-building materials.** S. S. SADTLER (B.P. 357,603, 22.5.30. U.S., 28.5.29).—See U.S.P. 1,758,913; B., 1931, 679.

**Manufacture of artificial stone or concrete blocks, slabs, panels, etc. for use in building.** F. T. WALKER (B.P. 358,047, 1.7.30).

**Rotary-kiln cooler.**—See I. **Fibrous slabs.**—See V. **Rubber floor-covering.**—See XIV.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Reduction characteristics of some iron ores.** J. KLÄRDING (Arch. Eisenhüttenw., 1931—2, 5, 129—138).—Various finely-powdered Fe ores were heated at 900° in vac., definite quantities of CO then admitted into the apparatus, and the compositions of the solid and gaseous phases determined and compared graphically with the results obtained with pure Fe<sub>2</sub>O<sub>3</sub>. The rate of reduction of the Fe oxides in the ores was appreciably retarded by the presence of impurities which combined with the FeO formed in the first stage of the reduction; microscopical examination of partly reduced products revealed the presence of several Fe silicates. The reducibility of Fe ores is a function of the proportion of normally reducible Fe present to the total Fe content of the ore.

A. R. POWELL.

**Reduction of iron ore.** III. B. STÅLHANE and T. MALMBERG (Jernkontorets Ann., 1930, 609—622; Chem. Zentr., 1931, i, 2800; cf. B., 1931, 203).—Reduction with H<sub>2</sub> or H<sub>2</sub>-CO mixtures is more rapid than that with CO. The rate is markedly reduced by the presence of CO<sub>2</sub> or H<sub>2</sub>O in the gas; the reduction in the rate falls with increasing H<sub>2</sub> content. A. A. ELDRIDGE.

**Problems in the development of furnace construction and operation, with especial reference to**

**heat-treatment furnaces.** A. SCHACK (Arch. Eisenhüttenw., 1931—2, 5, 193—208).—The sources of heat loss in steel-treating furnaces are discussed and methods are indicated for the more economical operation of these furnaces and for improvements in their construction.

A. R. POWELL.

**Mathematical treatment of the mechanism of cooling and solidification of liquid metals.** I, II. C. SCHWARZ (Arch. Eisenhüttenw., 1931—2, 5, 139—148, 177—186).—I. The effects of overheating, casting temp., mould temp., m.p. of metal, and thermal properties of mould material on the rate of solidification and cooling of cast metal, especially Fe and steel, are shown graphically and mathematical expressions are derived by the use of which these effects can be calc. for certain conditions.

II. Mathematical expressions are deduced for the rate of cooling of large Fe and steel castings and the effects thereon of mould dressings and of the air space which forms when the casting shrinks away from the sides of the mould. The temp. distribution during cooling in various types of mould is shown graphically.

A. R. POWELL.

**Heat-treatment fundamentals of plain and alloy cast iron.** F. B. COYLE (Metals and Alloys, 1931, 2, 120—131).—Prolonged annealing of cast Fe at 450—650° causes decomp. of eutectoid cementite into ferrite and graphite, a process which is favoured by a high Si and retarded by a high Cr content. Si raises the Ac and Ar crit. ranges by 30° for each 1% Si, whereas each 1% Ni lowers these ranges by 22°, and each 1% Cr raises them by 66°. The effect of Ni is augmented by V, but Mo has the reverse effect. Low-Si cast Fe responds to heat treatment better than that with a high Si content, but Ni-Cr alloys with low Si and C contents develop max. hardness when quenched in oil from 30° above the crit. point. Recent work on the heat-treatment of cast Fe is reviewed in detail. A. R. POWELL.

**Oils for use in the heat-treatment [of steel].** K. KREKELER and F. RAPATZ (Arch. Eisenhüttenw., 1931—2, 5, 173—176).—A satisfactory oil for quenching steel in the hardening process must be capable of cooling the metal at a rate greater than the crit. velocity, must have a high flash point, must drain well from the articles when they are removed from the bath, and must be resistant to the chemical action of the hot Fe scale, i.e., resistant to oxidation. Mineral oils appear to be superior in these respects to vegetable oils. For tempering baths the oil should have a flash point above 300°, a low asphalt content, and only a small tendency to form asphaltic compounds during use.

A. R. POWELL.

**Oil as a carburising medium [for steels].** A. J. LINDBERG (Metals and Alloys, 1931, 2, 106—109).—An electrically heated retort for carburising small steel articles comprises a heating chamber with its elements divided into several control zones each independently connected to a magnetic switch and automatic control pyrometer so that just the correct amount of heat is generated in each zone to hold the temp. of the retort adjacent to it at the desired value. The retort is closed with an insulated cover sealed with an asbestos gasket and supporting a motor which operates a fan

in the top of the retort. This fan sucks the vaporised oil up the centre of the retort and discharges it horizontally through a jet in the top of the retort where it is burned. The oil is placed in a lubricating cup and dripped into the fan discharge, which throws it against the side of the retort, where it is vaporised; the rate of drip is so regulated that the flame at the jet is of a greenish-golden colour free from a yellow tip and smokiness. After carbonising for the desired time, the oil drip is stopped and the furnace held at a const. temp. for 1—2 hr. to permit diffusion of the hyper-eutectoid case. The basket containing the charge can then be quenched directly from the furnace to obtain grain refinement, or the whole retort may be removed from the furnace and allowed to cool slowly so as to obtain a bright surface on the articles.

A. R. POWELL.

**Microstructure of pearlite.** H. E. PULOW and C. HEATH (Metals and Alloys, 1931, 2, 155—157).—Evidence based on the microstructure of variously heat-treated steels is adduced to show that pearlite may consist of small grains of ferrite and cementite rather than of a large grain of ferrite with cementite particles embedded in it.

A. R. POWELL.

**Growth of cast iron under tension.** E. PIVOVARSKY and O. BORNHOFFEN (Arch. Eisenhüttenw., 1931—2, 5, 163—166).—As a result of numerous tests on different types of cast Fe it is recommended that, in carrying out accelerated tests on the rate of growth, the specimen should be heated twice for 10 hr. at 650° under a load of 1 kg./sq. mm. with fairly free access of air. When the test is made in vac. with cast Fe, annealed to a ferritic structure, the vol. changes which occur are practically independent of the composition and are of the same order of magnitude as those observed with steel. In tests made below 500° cast Fe undergoes a static elongation which is quite independent of the tendency of the metal to grow, but the vol. changes which occur above 500° are characteristic of this tendency.

A. R. POWELL.

**Classification of [iron] castings.** J. NAVARRO (Anal. Fís. Quím., 1931, 29, 443—452).—The difficulties of applying a quant. treatment to the mechanical properties of cast Fe are discussed. Since the ratio of flexure,  $f$ , to breaking strength,  $s$ , bears some relation to the fineness of the segregated graphite it has been determined as a function of the hardness for a number of different sized cast-Fe bars. The results show that for an average casting the hardness ( $\Delta$ ) =  $2.65(S/f^{0.4})$ . The merits of a given casting may be estimated by the extent to which its properties deviate from this equation. The variation of  $S/f$  with the nature of the segregated graphite has been studied experimentally.

H. F. GILLBE.

**Comparative tests on natural and artificial (Aston) wrought iron.** W. VON GUTMANN and H. ESSER (Stahl u. Eisen, 1931, 51, 1193—1197).—The composition and structure of Aston Fe are similar to those of natural wrought Fe except that the former has the slightly higher S content; both metals behave similarly in the red-shortness and weldability tests, but Aston Fe gives the higher notched-bar impact figures and is less sensitive to ageing than wrought Fe, Armco

Fe, and Krupp's soft Fe, although it is not quite so resistant to corrosion and has a slightly lower tensile strength.

A. R. POWELL.

**Aqueous solutions of ethylene glycol, glycerin, and sodium silicate as quenching media for steels.** T. E. HAMILL (Bur. Stand. J. Res., 1931, 7, 555—571).—Surface- and centre-cooling curves have been obtained and hardness determinations made with cylinders of 1% C steel quenched from 875° in aq. solutions of the above-named compounds. It is not possible to achieve satisfactory cooling at rates intermediate between the rate for H<sub>2</sub>O and that for oil by means of aq. glycerin or glycol. Na silicate solutions may, however, be used, and have the advantage that moderate variations in the temp. of the quenching medium do not greatly affect the properties of the quenched steel. It may be necessary to add NaOH to Na silicate solutions to prevent deposition of solid on keeping. Quenching experiments were also made with "stepped" steel bars in order to investigate the relation between the hardness and microstructures produced by various quenching media. R. CUTHILL.

**Thermomagnetic investigation of tempering of quenched 0.75% carbon steel.** G. A. ELLINGER (Bur. Stand. J. Res., 1931, 7, 441—451).—On heating a quenched 0.75% C steel an increase in magnetisation at once resulted, probably in consequence of stress release caused by the separation of C from supersaturated solid solution in  $\alpha$ -Fe. When the temp. was maintained at 200° until the magnetisation had become const., on raising the temp. again an increase in magnetisation occurred at about 235°, this apparently corresponding with the decomp. of austenite. As the temp. rose to 300°, however, the magnetisation diminished. After the specimen had been kept at 300° until the magnetisation had become const., raising the temp. to 700° produced no further change. It is probable that between 200° and 300° the C which had separated was forming Fe<sub>3</sub>C, combination being complete when the magnetisation at 300° had become steady, for, on cooling, the A0 transformation, which was absent on the heating curve, appeared.

R. CUTHILL.

**Detection of internal stresses [in steel].** F. WEVER and H. MÖLLER (Arch. Eisenhüttenw., 1931—2, 5, 215—218).—A method for measuring the internal stresses in worked steels with an accuracy of  $\pm 5$  kg./sq. mm. by means of X-ray examination and comparison with an Au standard is described.

A. R. POWELL.

**Effect of surface decarburisation on fatigue properties of steel.** C. R. AUSTIN (Metals and Alloys, 1931, 2, 117—119).—The endurance limit of mild steel is reduced by about 20% by annealing in H<sub>2</sub> for 40 min. at 760° to decarburise the metal to a depth of 0.01 in.

A. R. POWELL.

**Influence of corrosion on fatigue of notched [nickel-steel] specimens.** T. S. FULLER (Min. and Met., 1931, 12, 446).—The endurance limit of a heat-treated 3.5% Ni steel without a notch was 58,000 lb./sq. in., and with a 45° notch, 0.035 in. deep with a 0.01 in. radius at the bottom, 32,000 lb./sq. in. Exposure of both specimens to an atm. of wet steam and air for 1 week reduced these vals. to 31,000 and

27,000 lb./sq. in., respectively, whereas the endurance limit of a Cr-plated notched specimen after similar exposure remained at 32,000 lb./sq. in. These results illustrate the deleterious effects of corrosion in conjunction with localised stress and the value of Cr-plating as a protection for steel members subject to alternating stresses under corrosive conditions.

A. R. POWELL.

**Causes and avoidance of tool-steel failures.** W. H. WILLS (Metals and Alloys, 1931, 2, 112—116).—Examples of failures of various tools made from hardened steels are illustrated and discussed; most of the failures were caused by rapid heating resulting in non-uniformity of the parts at the quenching temp., overheating or underheating in the heat-treatment, incorrect furnace atm. leading to decarburisation and the formation of soft spots, or incorrect tempering temp.

A. R. POWELL.

**Effect of nitrogen on steel.** F. W. SCOTT (Ind. Eng. Chem., 1931, 23, 1036—1051).—N increases the tensile strength, elastic limit, and Brinell hardness, but lowers the elongation and reduction in area, of mild steel; annealing of nitrified sheets causes no loss of N, does not produce blisters, and removes the deleterious effect of N on the drawing properties of the steel. The quality of galvanised steel sheets is not affected by the presence of up to 0.024% N, but the physical properties of the metal are affected in a similar manner to the effect produced by P, the effect of N being equal to that of four times as much P. The effect of N on the ductility of steel increases with increase in the C content; and as the tendency for N to segregate is greater than that of P, even a small amount of N in steel may produce very undesirable results. The N content of finished steel is fairly const., varying between 0.0032 and 0.0060% (average 0.0045%); low-C steels retain less N than do high-C steels which have been recarburised and deoxidised before casting. In refining a steel in the furnace the N is removed simultaneously with, and at about the same rate as, the P. The N content of pig Fe varies from 0.0032 to 0.0129%, and is the greater the higher is the temp. at which the blast furnace is operated.

A. R. POWELL.

**Resistance to wear of carbon steels.** S. J. ROSENBERG (Bur. Stand. J. Res., 1931, 7, 419—428).—The wear-resistance of annealed steels, as measured by the Amsler machine, was considerably less than that of hardened steels and unaffected by variations in the C content. The wear-resistance of hardened steels, on the other hand, increased rapidly with increase in the C content up to about 0.6%, further increase in the amount of C having relatively little effect. Tempering a hardened steel reduced its wear-resistance. The wear of low-C normalised steels was greater than that of annealed steels of similar C content, but with increasing C content the wear-resistance rapidly increased, ultimately approximating to that of a hardened steel. Wearing was accompanied by a considerable distortion in structure near the worn surface. A martensitic-troostitic matrix resisted wear best, and lamellar pearlite was also very resistant. The wear-resistance was reduced by the presence of granular pearlite, or spheroids of free ferrite or free Fe<sub>3</sub>C. R. CUTHILL.

**Reduction of non-metallic inclusions in steel by the addition of zirconium.** W. ZIELER (Arch. Eisenhüttenw., 1931—2, 5, 167—172).—Owing to the high affinity of Zr for  $O_2$ ,  $N_2$ , and S, the addition of a small quantity of Zr, *e.g.*, 0.15%, to molten steel in the ladle results in a considerable decrease in the non-metallic inclusions present. The deoxidising action of Zr is stronger than that of Si and its desulphurising action is greater than that of Mn.  $ZrS_2$  in steel does not cause brittleness, since it usually occurs as oval spots which are quite plastic in the rolling process. The S content of steels containing Zr cannot be determined by the evolution method since  $ZrS_2$  is not decomposed by 1:1 HCl. A. R. POWELL.

#### Corrosion of metals: theory and experiments.

**II. Dissolution of metals.** W. PALMER [with H. SUNDBERG, B. HANNERZ, O. PERSSON, G. KARLSSON, T. WILNER, E. BERG, and H. FANT] (Handl. Ing. Vetenskaps-Akad., Stockholm, 1931, no. 108, 198 pp.; cf. B., 1929, 921).—The dissolution of malleable cast Fe in acids is preceded by a long period of induction, after which it follows the course previously described for pig Fe, but at a slower rate owing to the smaller number of graphite inclusions; corrosion is most pronounced around the graphite inclusions, thus confirming the local-element theory of corrosion. The behaviour of soft Fe with about 0.1% C in acids at 25—50° is similar as regards effect of temp. and of concentration of acid to that of pig Fe; this is explained on the assumption that cementite acts as a cathode to anodic ferrite. The effect of cold-rolling in increasing the velocity of dissolution of Fe is attributed to the presence of fissures, which offer a greater surface to attack, and to further increase of this surface by enlargement of the fissures due to release of internal stresses along the corroded surfaces. Annealing after cold-working reduces the rate of dissolution to one fifth that of the hard wire; annealing in  $H_2$  still further reduces this rate owing to removal of the C, wire with 0.01—0.02% C dissolving in *N*-HCl at only one fiftieth the rate of hard-drawn wire with 0.1% C. Electrolytic Fe wire dissolves at one twelfth the rate of soft Fe wire. Experiments with Al and Zn of varying degrees of purity show that the nearer the metal approaches to abs. purity the slower is the rate of dissolution in acids, hence it appears that an absolutely chemically pure metal with a perfectly homogeneous surface should not be dissolved at all by acids, as the e.m.f. at all points of the surface is the same. Measurements of the electrical conductivity of mixtures of  $ZnSO_4$  and  $H_2SO_4$  show that isohydric solutions of two electrolytes with a common ion can be obtained only if the no. of the non-common ions in the mol. of each of the electrolytes is unity, and that the condition that two solutions of such electrolytes may be isohydric is that the concentration of the common ion in each solution is identical. Mathematical treatment of the effect of size and distribution of the impurities in metals on their rate of dissolution in acids indicates that the more finely distributed are these cathodic particles over the surface of the metal the smaller is the total resistance capacity of the local elements and the greater is the rate of dissolution of the metal; the local currents are concentrated at the actual boundaries between the

main metal and the particles of impurities, and this accounts for the severe pitting which occurs at areas surrounding the impurities. The method of determining the relative resistance to corrosion of various types of Fe by measuring the loss in wt. in acids during a definite time is adversely criticised, as the rate varies from time to time according to the physical conditions and chemical composition, increasing with lapse of time with some types of Fe and decreasing with others. When a metal is dissolved in the presence of a depolariser the action proceeds according to the diffusion theory only when the test pieces have been previously etched; this implies the occurrence of a period of induction, and hence that the activity of local elements is the primary factor in this case also. From theoretical considerations it is pointed out that the dissolution potential of a metal dissolved in Hg must be identical with that of the metal in a pure solid form, and hence that no period of induction should occur, dissolution being due to the activity of at. or mol. galvanic elements the resistance capacity of which must be small. From this it follows, firstly, that with const. e.m.f. and const. conductivity of the solution the rate of dissolution of a metal from a homogeneous liquid amalgam is proportional to its concentration: this is true for dil. Ca amalgams; and secondly, that if the amalgam contains an excess of metal or intermetallic compound over that sol. in the Hg to a homogeneous phase the rate of dissolution remains const. until the solid phase disappears, and then decreases proportionately to the decrease in concentration of the amalgam: this applies to saturated Ba amalgam. The theories of Centnerszwer and his collaborators on the mechanism of dissolution of metals are adversely criticised and his results are shown to be capable of a simpler explanation based on the theory of local galvanic elements.

A. R. POWELL.

**Corrosion rates of steel and composition of corrosion products formed in oxygenated water as affected by velocity.** B. E. ROETHELI and R. H. BROWN (Ind. Eng. Chem., 1931, 23, 1010—1012).—The rate of corrosion of rotated specimens of steel in oxygenated  $H_2O$  is dependent on the velocity of rotation, which affects the thickness of the liquid film and the type of corrosion product formed. At low speeds of rotation for concentrations of  $O_2$  usually met with, the steel becomes coated with a granular non-resistant film of a magnetic oxide of Fe, whereas at higher speeds the metal becomes covered with a protective film of gelatinous  $Fe(OH)_3$ , and hence the rate of corrosion decreases. At low velocities the rate of corrosion is governed by the resistance offered by the liquid film to the transfer of  $O_2$ , but at high velocities it is controlled by the resistance of the corrosion product. As the rotational velocity is increased, the rate of corrosion increases to a max. because of a reduction in the thickness of the liquid film, then decreases to a very low val. owing to the resistance of the  $Fe(OH)_3$  to the transfer of  $O_2$ , and finally rises again to a high val. at high velocities owing to the breakdown of the oxide film by mechanical erosion.

A. R. POWELL.

**Effect of oxygen concentration on corrosion rates of steel and composition of corrosion products**

**formed in oxygenated water.** G. L. COX and B. E. ROETHELI (Ind. Eng. Chem., 1931, 2, 1012—1016).—The rate of corrosion of steel in  $H_2O$  containing  $O_2$  is a linear function of the  $O_2$  concentration up to 5.5 c.c./litre, but with higher concentrations the rate gradually decreases with increasing  $O_2$  owing to an increase in the resistance of the film of corrosion product to the transfer of  $O_2$  to the metal surface. At low  $O_2$  concentrations the surface is coated with a film of a porous, black, magnetic oxide, whereas at higher concentrations red, gelatinous  $Fe(OH)_3$  is formed owing to the rapid rate of oxidation of  $Fe^{2+}$  ions, which prevents the precipitation of  $Fe(OH)_2$  (cf. preceding abstract).

A. R. POWELL.

**Methods of formation of [iron] rust.** G. CHAUDRON (Chim. et Ind., 1931, 26, 273—279).—The modern electrochemical theory of the rusting of Fe is discussed with reference to recent work of Evans, Maass and Herzog, and Chaudron, and its use in indicating suitable methods of protecting Fe from rusting is pointed out.

A. R. POWELL.

**Prevention of corrosion of metals by sodium dichromate as affected by salt concentrations and temperature.** B. E. ROETHELI and G. L. COX (Ind. Eng. Chem., 1931, 23, 1084—1090).—The protective action of chromates on metals exposed to corrosive salts is used in refrigerating plants, but no definite information has been available as to the proportion of chromate required to be added to the brine. Corrosion rates on different metals of NaCl solutions with additions of chromate were determined, the specimen being rotated and the solution aerated. With Fe a min. chromate concentration is necessary for protection, which min. increases with temp. and NaCl concentration. With Zn pitting is very prevalent and protection effective only with dil. NaCl solutions and at 20°. Cu and brass are easily protected, but in the case of Al the presence of chromate seems to promote pitting. The protection of Pb is slight and variable. These differences are, no doubt, due to variations in the structure of the protective film.

C. IRWIN.

**Soil-corrosion studies. Non-ferrous metals and alloys, metallic coatings, and specially prepared ferrous pipes removed in 1930.** K. H. LOGAN (Bur. Stand. J. Res., 1931, 7, 585—605).—The results of the examination of specimens of a variety of non-ferrous materials and metal-protective coatings after having been buried in various types of soils for 4—6 years are described. To secure the max. resistance to corrosion the material used must be chosen to suit the soil with which it is to be in contact; no one material is best for all soil conditions. In nearly all soils Cu and alloys high in Cu exhibit a high degree of resistance to corrosion, and Pb is attacked less readily than Fe or steel. All the metallic coatings examined afforded protection to the material beneath.

R. CUTHILL.

**Age-hardening of copper-titanium alloys.** E. E. SCHUMACHER and W. C. ELLIS (Metals and Alloys, 1931, 2, 111).—The Rockwell B hardness of Cu with 2.03% Ti after quenching from 875° is 37; on ageing at 500° it rises steeply to 89 in 2 hr., then slowly falls to 82 in a further 8 hr. At 400° the hardness rises to 79 in 1 hr.,

then slowly increases to 84 in the following 8 hr.; ageing at 200° produces only a very slow increase in hardness. The alloy with max. age-hardness has a tensile strength of 92,000 lb./sq. in. with an elongation of 10% and an electrical resistivity of 10.5 microhms/cm. at 20°. Alloys with 0.67 and 0.93% Ti also harden on ageing at 400° after quenching from 875°, but the max. hardness is only 15 and 36 on the Rockwell B scale, respectively.

A. R. POWELL.

**Causes of fire and explosion in aluminium-bronze factories.** RITTER, BOLLÉ, FINGER, FRICKE, VOM BERG, SCHNEIDER, and GLIWITZKY (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 207—214; Chem. Zentr., 1931, i, 2792).—The danger is diminished by retarding oxidation by control of the  $O_2$  supply or cooling by the use of a large excess of air; working in an atmosphere of  $N_2$  produces an explosively oxidisable bronze. The ignitability depends largely on the extent of the surface.

A. A. ELDRIDGE.

**Quantitative spectral analysis of alloys.** H. TÖPFELMANN and W. SCHUHNECHT (Z. anal. Chem., 1931, 86, 160—174).—Experimental details are given.

E. S. HEDGES.

**Electrolytic refining of copper, using complex salts of cuprous chloride. VII. Behaviour of antimony.** S. MAKISHIMA and N. KAMEYAMA (J. Soc. Chem. Ind., Japan, 1931, 34, 324—325 B; cf. B., 1930, 772).—To an electrolyte of Cu alkali-metal chloride 100 mg. of Sb per 1 kg. of Cu anode was added and the current passed for 30 hr. at about 0.3 volt. The cathode was found to contain 0.00024% Sb only, or in presence of K H tartrate 0.00002% Sb. It is therefore probable that in this process contamination of Cu with Sb, if present in the anode, is practically prevented.

C. IRWIN.

**Influence of acidity of electrolyte on structure and hardness of electrodeposited nickel.** D. J. MACNAUGHTAN and R. A. F. HAMMOND (Trans. Faraday Soc., 1931, 27, 633—648).—In the electrodeposition of Ni from buffered aq.  $NiSO_4$ , the hardness of the deposit increases as the  $p_H$  is increased, the rate of increase becoming much more rapid, however, above a certain crit.  $p_H$ , which depends on the buffer present. For the production of a hard deposit, free from cracks, a solution buffered with  $(NH_4)_2SO_4$  is most suitable (cf. B., 1928, 644), whereas by using  $H_3BO_3$ , or  $H_2BO_3$  and NaF as buffer and working below the crit.  $p_H$  satisfactory soft deposits are obtained. The structure of the deposit has no direct relation to the strength of the electric field at the cathode face, and is only slightly influenced by the amount of  $H_2$  discharged. It is probable, however, that the co-deposition of  $Ni(OH)_2$  or a basic salt may vitally influence the character of the Ni deposit.

R. CUTHILL.

**Throwing power of plating solutions, with particular reference to certain zinc-plating solutions.** B. K. BRAUND (Trans. Faraday Soc., 1931, 27, 661—674).—Their throwing power has been determined by Haring and Blum's method (B., 1923, 556 A), a correction for variations in conductivity due to uneven temp. in the bath being found, in general, to be necessary. The throwing power varies with the material of

the cathode, and with a mild-steel cathode depends to a considerable extent on the method of prep.

R. CUTHILL.

**Protection of metals by metal films.** E. S. HEDGES (Chem. & Ind., 1931, 768—772).—A review.

**Industrial steels and alloys.** Recent developments. I. Steels for strength, corrosion, and machining. II. Steels, alloys, and cast irons for heat-resistance. J. W. DONALDSON (Chem. & Ind., 1931, 787—793, 838—845).

**[Historical review of] development of the cyanide process.** G. H. CLEVINGER (Trans. Amer. Electrochem. Soc., 1931, 60, 79—87).

**Pyrometer-tube walls.**—See I. **Blast-furnace coke.**—See II. **Pt catalyst for  $\text{NH}_3$  oxidation.**—See VII. **Enamels for cast Fe.** **Au and "Ag" lustres.** **Steelworks' dolomite.** **Refractories for Pb-refining furnaces.**—See VIII.

#### PATENTS.

**Ore classifier.** A. L. MOND. From COLORADO IRON WORKS Co. (B.P. 357,159, 11.6.30).—In the type in which ore pulp is fed through an inclined trough which is fitted with a rotary spiral agitator and conveyor, one end of which is submerged partly in the liquid, means for raising or lowering this end are provided so as to obviate digging out the tank when operations are interrupted.

C. A. KING.

**Concentration of [sulphide and phosphate] ores.** R. B. MARTIN (U.S.P. 1,797,356, 24.3.31. Appl., 23.9.29).—The moist ore is tumbled in a revolving cylinder with selective agents to coat the mineral particles and is then subjected to gravity or flotation concentration. *E.g.*, a phosphate ore is ground to pass 48-mesh and the slime removed by classification; the moist sandy portion is agitated with a sol. soap and  $\text{Na}_2\text{SiO}_3$ , crude oil is added, and the mixture passed over Wilsley tables, whereby the gangue passes off in the usual concentrate launder and the phosphate collects in the usual tailings launder in the form of oily flocculent aggregates.

A. R. POWELL.

**Treatment of iron ores.** COMSTOCK & WESCOTT, Inc.; Assecs. of E. W. WESCOTT (B.P. 355,235, 17.2.30. U.S., 24.10.29. Addn. to B.P. 306,107; B., 1930, 106).—Sulphide Fe ores are heated in the vapours of  $\text{FeCl}_2$  obtained from a later stage of the process, whereby the Fe is converted into  $\text{FeCl}_2$  with elimination of S. The  $\text{FeCl}_2$  is chlorinated to  $\text{FeCl}_3$ , part of which is returned as vapour to the first stage of the process and the remainder burned with air to yield  $\text{Fe}_2\text{O}_3$  and dil.  $\text{Cl}_2$  for the chlorination stage.

A. R. POWELL.

**[Metal-casting] sand moulds.** B. F. WALLACE (B.P. 357,126, 12.5.30).—A pulverised carbonaceous base, *e.g.*, graphite impregnated with a pitch-like residual product of b.p.  $> 250^\circ$ , from the distillation of a hydrocarbon, is dusted on to a pattern which is then rammed round with sand to form a mould.

C. A. KING.

**Heat-treatment of metal articles.** W. BECK, Assr. to DEUTS. GOLD- U. SILBER-SCHNEIDESTALT VORM. ROESSLER (U.S.P. 1,799,945, 7.4.31. Appl., 19.5.28. Ger., 20.5.27).—The articles are heated at  $500^\circ$  in a

bath of fused KOH or NaOH to which is added sufficient finely-divided C to prevent surface oxidation.

A. R. POWELL.

**[Heat]-treatment of chromium-nickel-iron-carbon alloys.** F. KRUPP A.-G. (B.P. 357,254, 21.6.30. Ger., 24.6.29).—The endurance limit for reversed bending stresses and the yield point of steels with 6—40% Cr, 40—4% Ni, and  $< 1\%$  C are improved by quenching from  $500\text{--}950^\circ$ , preferably from  $850\text{--}900^\circ$ .

A. R. POWELL.

**[Copper] coating of [iron or steel] wire.** R. M. HUGHES. From JOHNSON STEEL & WIRE Co., Inc. (B.P. 355,463, 17.7.30).—Fe wire is passed continuously through a solution of  $\text{CuSO}_4$  at  $5\text{--}10^\circ$ , the liquid being agitated by a stream of finely-divided air bubbles, thence through a washing tank, and finally through a bed of hot sand which dries and burnishes the Cu deposit.

A. R. POWELL.

**Coating of metals [iron with chromium].** BRIT. THOMSON-HOUSTON Co., LTD., Assecs. of G. H. HOWE (B.P. 354,887, 27.5.30. U.S., 28.5.29).—Fe articles are coated with a layer of Cr-Fe alloy by heating in a closed electric-resistance furnace in contact with a powdered mixture of 85% Cr, 10% Zn, and 5% Al at a temp. above the volatilisation point of Zn, *e.g.*, at  $925^\circ$ .

A. R. POWELL.

**Metallising articles of organic origin.** K. RICHTER (B.P. 355,848, 6.6.30).—The articles are treated in vac. with the vapour of Zn or Cd.

A. R. POWELL.

**Case-hardening composition.** E. C. MOFFETT, Assr. to AMER. CYANAMID Co. (U.S.P. 1,796,800, 17.3.31. Appl., 30.7.27).—A case-hardening bath comprises a fused 2 : 1 mixture of  $\text{CaCl}_2$  and NaCl to which is added from time to time as required a small proportion of  $\text{Ca}(\text{CN})_2$ .

A. R. POWELL.

**[Refining agent for] manufacture of metals and alloys.** MAY & BAKER, LTD., and G. S. HIGGINSON (B.P. 357,539 and 357,540, 19.6.30).—In casting metals and alloys the structure is refined and gases are eliminated by treating the molten metal with (A) KI or  $\text{BaI}_2$  either in the ladle or as a mould dressing, or (B) a cartridge containing KI or  $\text{BaI}_2$  with or without a flux.

A. R. POWELL.

**Manufacture of steel.** F. F. McINTOSH, Assr. to CRUCIBLE STEEL Co. of AMERICA (U.S.P. 1,797,728, 24.3.31. Appl., 6.5.29).—A small quantity of  $\text{MoS}_2$  is added to the finished steel in the ladle.

A. R. POWELL.

**Steel alloy insensible to hot gases and vapours.** B. STRAUSS, Assr. to F. KRUPP A.-G. (U.S.P. 1,787,403, 30.12.30. Appl., 5.4.29. Ger., 21.9.28).—Vessels for cracking petroleum are made of a steel alloy containing 0.1—0.2 (0.15)% C, 5—12 (6)% Cr, 0.2—5.5 (0.3)% V, and 0.5—2.0 (2)% Ni, which has been heated above  $\text{Ac}_3$ , quenched, and tempered at  $600\text{--}650^\circ$ .

A. R. POWELL.

**Alloy steel containing nickel, silicon, and copper.** P. A. E. ARMSTRONG and R. P. DE VRIES, Assrs. to LUDLEM STEEL Co. (U.S.P. 1,799,273, 7.4.31. Appl., 5.7.28).—Steel resistant to corrosion and to scaling at high temp. contains 5—40 (37)% Ni, 2—7 (4)% Si, 0.5—15 (12)% Cu, and 0.05—1.5 (0.3)% C.

A. R. POWELL.



**[Nickel-cobalt steel] alloys.** ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Asses. of H. SCOTT (B.P. 357,665, 7.7.30. U.S., 5.7.29).—A leading-in wire for sealing into glass is made from an Fe alloy containing 15–30 (23.7)% Ni, 20–40 (21.9)% Co, <0.5 (0.2)% Mn, <0.3% C, and 0.1% Si. A. R. POWELL.

**Roasting of ores containing iron and copper sulphides.** H. K. A. LASSEN (B.P. 355,664, 19.5.30).—Sulphide ores containing Cu pyrites in association with Ag minerals and/or molybdenite are roasted with agitation with a regulated supply of air at 430–470°, whereby the Fe sulphides are converted into  $\text{Fe}_3\text{O}_4$  with which the Cu is associated. Magnetic separation of the roasted material affords a magnetic Fe–Cu product and a non-magnetic Ag–Mo product. A. R. POWELL.

**Refining of copper.** J. LEEMANS, and SOC. GÉN. MÉTALLURG. DE HOBOKEN (B.P. 356,077, 8.1.31).—Black Cu is refined by melting it with refinery slag containing 30–40% Cu as oxide and silicate sufficient to remove the Fe and S from the Cu. Very impure Cu is smelted in a cupola with the slag and the resulting Cu is further purified by bessemerisation. A. R. POWELL.

**Recovery of metallic elements and alloys. [Treatment of scrap brass etc.]** H. M. BURKEY and D. L. OGDEN, Assrs. to AMER. METAL CO., LTD. (U.S.P. 1,785,247, 16.12.30. Appl., 15.8.28).—The scrap is first heated at a low temp. to remove most of the Sn–Pb solder by liquation, then smelted in a cupola under such conditions that the greater part of the Pb, Zn, and Sn is removed as fume and a crude Cu is obtained. The latter is blown in a converter to eliminate Fe and to volatilise the remainder of the Pb, Zn, and Sn. The Cu is finally purified by electrolysis and the fume extracted with aq.  $\text{NH}_3$  to remove Zn, which is recovered as oxide. The residual fume is smelted to obtain a Sn–Pb alloy which, together with the solder runnings, is refined by electrolysis. A. R. POWELL.

**Treatment of zinc-bearing material.** (A) W. C. HOOEY, (B) W. O. BORCHERT, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,799,166 and 1,799,278, [A, B] 7.4.31. Appl., [A] 26.12.28, [B] 8.12.28).—(A)  $\text{CaCO}_3$  and  $\text{MgCO}_3$  are removed from ZnS concentrates by agitating the material in a series of tanks with dil.  $\text{H}_2\text{SO}_4$  on the countercurrent principle. The  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  formed is separated from the blende by means of classifiers. (B) Blende contaminated with  $\text{CaCO}_3$  and/or  $\text{MgCO}_3$  is treated with sufficient  $\text{H}_2\text{SO}_4$  to convert the carbonates into sulphates (in a finely-divided form) which are readily washed away from the Zn mineral. A. R. POWELL.

**Treatment of zinciferous materials for removal of cadmium or cadmium together with lead.** NEW JERSEY ZINC CO., Asses. of H. M. CYR and R. K. WARING (B.P. 355,840, 4.6.30. U.S., 15.10.29).—Roasted Zn ore is mixed with about 5% of anthracite powder and a solution of  $\text{ZnCl}_2$  sufficient to chloridise all the Cd and Pb, and the moist mixture is sintered in a Dwight–Lloyd machine, whereby the Cd and Pb are volatilised as chlorides and a sinter containing <0.0025% Cd and 0.1% Pb is obtained from which a high-grade Zn is produced by the usual smelting operation. A. R. POWELL.

**Treatment of magnesium.** E. C. BURDICK, Assr. to DOW CHEM. CO. (U.S.P. 1,795,473, 10.3.31. Appl., 10.11.26).—Mg articles are cleaned and given a bright, lustrous surface by immersion in 10–30 (25)%  $\text{H}_2\text{SO}_4$ . A. R. POWELL.

**Production of magnesium alloys.** C. ARNOLD. From DOW CHEM. CO. (B.P. 354,744, 11.3.30).—Mg alloys containing Mn, Ce, Be, V, Mo, or Ni are obtained by addition of the corresponding chloride to the fused  $\text{MgCl}_2$  bath, from which the Mg is produced by electrolysis. A. R. POWELL.

**Protection of magnesium and its alloys from attack by hot baths of saline melts.** I. G. FARBENIND. A.-G. (B.P. 357,428, 26.11.30. Ger., 11.12.29).—In the heat-treatment of Mg or its light alloys in fused nitrate baths, addition of 1–2% of an alkali chromate, dichromate, or fluoride to the fused salt prevents corrosion of the metal. A. R. POWELL.

**Increasing the resistance to corrosion of objects made from alloys of magnesium with manganese.** I. G. FARBENIND. A.-G. (B.P. 355,920, 5.8.30. Ger., 23.8.29).—Mg alloys containing 0.5–2.5% Mn are hot-worked above 410–480°, preferably at 470–500°, maintained at this temp. for 2–48 hr., and allowed to cool in the air. A. R. POWELL.

**Manufacture of aluminium and its alloys.** T. R. HAGLUND (B.P. 355,792, 22.5.30).—A mixture of a W alloy or compound with  $\text{Al}_2\text{O}_3$  or bauxite which has been subjected to an electrothermal smelting to remove Fe and part or all of the Si and Ti is smelted in an electric furnace with carbonaceous reducing agents to give an alloy containing 50–90% Al and >10% W. Liquation of this alloy affords Al containing <0.07% W and a W–Al alloy which may be returned to the smelting operation. Al may also be recovered from the alloy by electrolytic refining in a molten fluoride bath or by distillation in vac. A. R. POWELL.

**Aluminium alloys.** H. W. CLARKE and L. AITCHISON (B.P. 357,800 and 357,805, [A] 8.10.30, [B] 13.10.30).—The alloys comprise Al with (A) 1.5–3 (2)% Cu, 0.5–2.5 (1.5)% Ni, 0.5–2.5 (1.4)% Fe, 0.5–2 (0.75)% Mg, and 0.25–1 (0.5)% Si, or (B) 0.5–5 (0.83)% Mn, 0.5–1.5 (0.94)% Si, 0.3–1.25 (0.78)% Mg, and 0.3–2 (1.03)% Sb, heat-treated by quenching from 510° and ageing at 150–175°. A. R. POWELL.

**Aluminium-base alloy.** T. W. BOSSERT and J. A. NOCK, JUN., Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,797,851, 24.3.31. Appl., 5.6.30).—An alloy containing at least 95% Al with 1–1.5% Mn and 0.5–1.5% Mg is claimed. A. R. POWELL.

**Plastic heat-treated aluminium alloy.** N. V. HYBINETTE, Assr. to HYBINETTE PATENTS CORP. (U.S.P. 1,785,774, 23.12.30. Appl., 18.5.29).—The alloy comprises Al with 0.5–1 (0.5)% Zn, 0.5–1 (0.75)% Mg, 0.05–0.15 (0.1)% Cd, and <0.25% Cu, Ni, Co, Mn, Cr, Mo, or W (0.2% Cu). After suitable heat-treatment the alloy is not subject to intercryst. corrosion. A. R. POWELL.

**Recovery of tungsten from ores.** C. V. IREDELL, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,796,026, 10.3.31. Appl., 28.5.29).—The ore is digested with

NaOH solution and the insol. material removed by filtration. The filtrate is neutralised with HCl until it contains 0.3–0.7% of free alkalinity, then diluted with warm H<sub>2</sub>O to 150 g. of Na<sub>2</sub>WO<sub>4</sub> per litre, and treated with CaCl<sub>2</sub> solution (*d* 1.175) in a fine stream, whereby CaWO<sub>4</sub>·H<sub>2</sub>O is precipitated. This is converted into WO<sub>3</sub> in the usual way by boiling with HCl.

A. R. POWELL.

**Manufacture of refractory metals, e.g., tungsten.** GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (B.P. 354,785, 10.5.30).—A bar of W sintered in H<sub>2</sub> in the usual way is heated in A at 3000° for 3–5 min. with a boat containing Na disposed about 1 cm. away from the bar so as to provide an envelope of Na vapour around the bar, which is thereby effectively deoxidised.

A. R. POWELL.

**Improvement of cobalt-tungsten alloys.** VEREIN. STAHLWERKE A.-G. (B.P. 356,089, 28.2.31. Ger., 4.7.30).—Co alloys with 5–50% W are quenched from above 1000° and aged at 500–900°; e.g., the 60:40 Co-W alloy, quenched from 1300° and aged at 800°, has a Brinell hardness of 660.

A. R. POWELL.

**Production of hard [cobalt-chromium-tungsten] alloys.** VEREIN. STAHLWERKE A.-G. (B.P. 355,782, 25.3.30. Ger., 26.3.29).—Alloys of Co and Cr with 10–40% W and 2–5 (3)% C are cooled slowly after casting, then reheated at 700° until 10–60% of the total C is liberated as finely-dispersed graphite, and again allowed to cool slowly. Part of the Co may be replaced by Ni and part of the W by Mo.

A. R. POWELL.

**Recovery of cobalt or its compounds [from mattes].** E. A. A. GRÖNWALL (B.P. 357,366, 16.9.30. Swed., 17.9.29).—Cu matte obtained in the usual way from cobaltiferous Cu pyrites is bessemerised until all or most of the Fe has been slagged and removed. Blowing is then continued with addition of SiO<sub>2</sub> to slag off the Co and the resulting Co silicate is crushed and leached with H<sub>2</sub>SO<sub>4</sub> to extract Co, smelted with more Cu pyrites to obtain a Co-rich matte, or roasted with pyrites and NaCl to obtain sol. Co salts. Reduction of the slag in an electric furnace affords a crude metal from which pure Co may be recovered by wet methods.

A. R. POWELL.

**Salt bath for annealing metal [silver] articles.** E. FAURLÄNDER (B.P. 357,538, 19.6.30).—The bath comprises a mixture of NaCl, K<sub>2</sub>SO<sub>4</sub>, and K<sub>2</sub>CO<sub>3</sub> and/or Na<sub>2</sub>CO<sub>3</sub>, with or without small quantities of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, MgCO<sub>3</sub>, CaCO<sub>3</sub>, and NaNO<sub>3</sub>, the mixture having a m.p. < 600°.

A. R. POWELL.

**[Palladium] alloy.** H. KLAUSMANN and H. R. KIEFE, Assrs. to BAKER & Co., INC. (U.S.P. 1,797,236, 24.3.31. Appl., 3.5.30).—The alloy contains 75–98 (95)% Pd, 24–1 (3)% Rh, and the rest (2%) Ru.

A. R. POWELL.

**Welding composition.** J. A. HEALY (U.S.P. 1,796,329, 17.3.31. Appl., 16.9.29).—WC tips are brazed on to steel shanks by the use of a powdered mixture of 30% of fused Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, 60% of ferromanganese, and 10% of ferro-silicon, to which is added 20–100% of high-speed steel filings.

A. R. POWELL.

**[Inhibitor for] the pickling of metals and like processes.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P.

357,387, 8.10.30. U.S., 9.10.29).—Thioglycollic acid, or its salts, esters, or other derivatives in combination with a protective colloid or foaming agent is claimed.

A. R. POWELL.

**Copper extraction process.** W. E. GREENAWALT (U.S.P. 1,798,255, 31.3.31. Appl., 6.1.27. Renewed 18.8.30).—Cu sulphide concentrates are separated by gravity or flotation into a rich product containing 50–60% Cu and a poor product containing < 10% Cu. The former is smelted to produce a rich matte, part of which is converted into crude Cu for anodes and the remainder roasted and extracted with H<sub>2</sub>SO<sub>4</sub> to give a CuSO<sub>4</sub> solution for electrolysis. The spent electrolyte from the Cu refining is returned to the leaching tanks. The poor product from flotation is roasted and leached with H<sub>2</sub>SO<sub>4</sub> separately, and the leach liquor is purified by digestion with part of the CuO obtained in roasting the matte. The purified liquor is passed to the electrolytic tanks. The insol. residues from the process are smelted for the recovery of Au and Ag.

A. R. POWELL.

**Production of metallic tantalum.** C. W. BALKE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,799,403, 7.4.31. Appl., 20.8.23).—A fused mixture of K<sub>2</sub>TaF<sub>7</sub> and Ta<sub>2</sub>O<sub>5</sub> is electrolysed in an Fe pot with a graphite anode. When the mass contains 15% Ta it is cooled, crushed, and the Ta recovered by concentration on Wilfley tables.

A. R. POWELL.

**Detinning [of scrap tinplate].** E. KARDOS (U.S.P. 1,798,607, 31.3.31. Appl., 30.8.28).—The scrap is leached with NaOH into which are passed nitrous gases produced by the catalytic oxidation of NH<sub>3</sub>. When the solution contains 5–6% Sn and the nitrites have all been decomposed it is electrolysed to recover Sn and the spent electrolyte is returned to the leaching tanks.

A. R. POWELL.

**Plating of metal articles.** F. B. DEHN, From MAJOR ENG. CORP. (B.P. 354,706, 17.2.30).—The anode comprises pulverulent metal at the bottom of the tank, and the cathodes are arranged so as to be movable through the tank, during which movement part of the anode metal is disseminated throughout the electrolyte so as to effect depolarisation and burnishing of the cathodes.

A. R. POWELL.

**[Nickel-]plating process.** W. J. WRIGHTON and E. D. TILLYER, Assrs. to AMER. OPTICAL CO. (U.S.P. 1,797,254, 24.3.31. Appl., 2.4.28).—Articles made of Ni wire, e.g., spectacle frames, are finished with a heavy coating of Ni from any of the ordinary plating baths.

A. R. POWELL.

**Chromium plating.** L. R. WESTBROOK, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,795,459, 10.3.31. Appl., 23.2.28).—An alkali molybdate or vanadate is added to the usual Cr-plating bath.

A. R. POWELL.

**Treating [finishing] cadmium-plated goods.** L. MELLERSH-JACKSON, From C. T. NEAL (B.P. 357,887, 23.12.30).—The articles are dipped into dil. HNO<sub>3</sub> (1 oz. per gal.) immediately after removal from the plating bath, rinsed thoroughly, and dried, to produce a lustrous finish.

A. R. POWELL.

**Coating and colouring metals.** A. PACZ (U.S.P. 1,798,218, 31.3.31. Appl., 23.3.25).—Articles of steel, Fe, Sn, Pb, Al, Zn, Cd, or Mg are covered with a lustrous,

black, protective coating by immersion in a solution containing  $(\text{NH}_4)_2\text{MoO}_4$  and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ , with or without contact with a Zn anode. A. R. POWELL.

**Purification of zinc ore.** O. GERLACH (B.P. 358,039, 27.5.30).—See U.S.P. 1,773,991; B., 1931, 448.

**Sintering of material. Deflocculation of colloids. Gas cleaner.**—See I. **Non-corroding solution. Ti from ores.**—See VII. **Metal-melting furnace. Brazing furnace. Electroplating anodes. Welding rod.**—See XI. **Rubber[metal] adhesive. Moulds for rubber.**—See XIV.

## XI.—ELECTROTECHNICS.

**Electrical insulation with special reference to the applications of synthetic resins.** W. D. OWEN and A. M. THOMAS (J. Oil Col. Chem. Assoc., 1931, 14, 290—305).—Chemical aspects of electrical insulation problems are discussed, the nature of true and pseudo-dielectrics being indicated.  $\text{PhOH-CH}_2\text{O}$  resins and other synthetic resins used in the electrical industry are summarised, the nature, sources of raw materials, and theories of resinification receiving attention. The electrical, chemical, and physical properties required for electrical insulation and standard methods of testing for these are detailed. S. S. WOOLF.

**Evaporation of pulp-mill waste liquors.**—See V. **NaCl industry of Rio.  $\text{MnO}_2$ . Decomposition of  $\text{H}_2\text{S}$ .**—See VII. **Resistivity of refractories.**—See VIII. **Carburising steels. Cu. Ni-plate. Zn-plate.**—See X. **Fe oxide pigments.**—See XIII. **Electric currents and fermentation.**—See XVIII. **Determining  $\text{H}_2\text{O}$  in wheat.**—See XIX.

### PATENTS.

**[Electric metal-melting] furnace.** M. UNGER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,800,034, 7.4.31. Appl., 18.4.30).—Powdered or granular material, e.g.,  $\text{MgO}$ , is packed round the heated crucible, and is agitated when being packed. J. S. G. THOMAS.

**[Electric] furnace [for high-temperature brazing].** F. C. KELLEY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,799,102, 31.3.31. Appl., 13.8.29).—The furnace is heated to a temp. just short of the softening point of the materials of construction by known means, e.g., an electric resistance coil. The charge is first heated to nearly that temp. by heat from the furnace, and then the junction alone (e.g., a W filament and terminal rod) is further heated by a high-frequency field emanating from a coil round the outside of the furnace.

B. M. VENABLES.

**[Preventing the formation of crust in] electric batteries having an ammonium chloride electrolyte.** SOC. ANON. LE CARBONE (B.P. 357,890, 29.12.30. Fr., 19.11.30).—Wicks are arranged to cause continuous circulation of the electrolyte. J. S. G. THOMAS.

**Electrolytic cells.** J. W. THOMPSON, J. W. HINCHLEY, and L. H. BAMTON (B.P. 356,241, 2.6.30).—A counterpoised vessel containing electrolyte can be raised or lowered within a frame supporting stationary C or graphite electrodes. J. S. G. THOMAS.

**Electrolysers [for decomposing water etc. under pressure].** J. E. NOEGGERATH (B.P. 358,191, 11.8.30).—

Radially extending tension members are arranged within the electrolyser to take up stresses due to pressure on the walls of the vessel. J. S. G. THOMAS.

**[Soluble electroplating] anodes.** UDYLYTE PROCESS Co., Asses. of J. O. WAGNER and A. S. ASCHER (B.P. 357,977, 30.6.30. U.S., 20.2.30).—Large balls of the metal to be deposited fill substantially the cross-section of a long, narrow, hollow cage of foraminous material and descend under gravity as they dissolve.

J. S. G. THOMAS.

**Manufacture of electrodes for positive-column, luminous, gaseous conduction tubes.** W. W. TRIGGS. From Q. R. S. DE VRY CORP. (B.P. 357,516, 19.5.30).—A small-bored, tubular, cylindrical body, e.g., of Ni, is coated internally with  $\text{BaC}_2$  [made from a paste of  $\text{Ba}(\text{OH})_2$  and C] and externally with a mixture of  $\text{NiO}_2$  (1 pt.), sealing glass (1 pt.), and powdered  $\text{SiO}_2$  (4 pts.) in EtOH and heated slowly.

J. S. G. THOMAS.

**Low-temperature emissive cathodes for electric-discharge devices.** STANDARD TELEPHONES & CABLES, LTD., and W. E. BENHAM (B.P. 357,632, 26.6.30).—A core consisting of an alloy containing equal parts of W and Mo is coated with oxide by heating.

J. S. G. THOMAS.

**[Support of cathode in] electron-discharge tubes or thermionic valves.** N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 357,444, 18.12.30. Holl., 19.7.30).—The cathode is soldered, preferably with  $\text{Ag}_2\text{O}$  mixed with  $\text{H}_2\text{O}$ , to supporting members which do not conduct current to the cathode.

J. S. G. THOMAS.

**Introduction of mercury into [electric] discharge tubes.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 358,200, 14.8.30. Ger., 21.8.29).—A mixture of Hg sulphide and a reducing agent, e.g., Fe, Ni, and, if desired, Mg, is heated inside the tube.

J. S. G. THOMAS.

**Dielectric materials.** STANDARD TELEPHONES & CABLES, LTD., M. C. FIELD, W. E. HUGH, and L. G. B. PARSONS (B.P. 357,624, 20.6.30).—A dielectric composed of rubber, preferably Hevea pale crêpe rubber freed from protein and resinous matters and  $\text{H}_2\text{O}$ -sol. constituents, 40—80% of a synthetic hydrocarbon polymeride, preferably polystyrene, and up to 20% of a hydrocarbon wax is claimed. J. S. G. THOMAS.

**Electrical insulators.** S. JUST (B.P. 357,510, 14.4.30. Hung., 16.9.29).—A compressed mixture of colloidal BN with a small proportion of binder, e.g., hydrated silicic acid, is heated slowly and then baked at a very high temp. Various other inorg. or org. materials, e.g., glass, asbestos, bakelite, paraffin, resin-lac, may be added to the mixture. J. S. G. THOMAS.

**[Inorganic] electrical insulation.** BRIT. THOMSON-HOUSTON Co., LTD., Asses. of C. DANTSZEN (B.P. 358,285, 10.10.30. U.S., 11.10.29).—A coating of  $\text{ZnF}_2$  is deposited electrolytically on a conductor of Zn or coated with Zn. J. S. G. THOMAS.

**Weld-rod for electric-arc welding.** A. F. BURGESS. From A. O. SMITH CORP. (B.P. 357,512, 22.4.30).—A steel, alloy steel, or low-C Fe welding rod (diam.  $> 0.25$  in.) covered with carbohydrate material, e.g., a mixture of wood flour (15%), Na silicate (70.6%),

kaolin (9.4%), and  $\text{SiO}_2$  flour (4.7%), is supplied with a current of 500–2000 amp. at 35–55 volts.

J. S. G. THOMAS.

**Carrying out chemical reactions.** INST. F. PHYSIKAL. GRUNDLAGEN DER MEDIZIN (B.P. 357,534, 17.6.30. Ger., 26.6.29).—The reactions are effected by d.c. glow discharges between unequally curved electrodes arranged in a chamber so that the cathode-fall space is approx. equal to the space between the electrodes. Examples are: the synthesis of  $\text{NH}_3$ , and manufacture of  $\text{C}_2\text{H}_2$  from  $\text{CH}_4$  or from a hydrocarbon mixture containing  $\text{CH}_4$ , with or without additional  $\text{H}_2$ . Diacetylene is among the final products in this latter case.

J. S. G. THOMAS.

**Kerr and similar electro-optical cells.** MARCONI'S WIRELESS TELEGRAPH CO., LTD., ASSEES. OF S. BLOOMENTHAL (B.P. 358,368, 12.12.30. U.S., 12.12.29).

**[Miniature dry-cell] electric batteries.** BURGESS BATTERY CO., ASSEES. OF W. B. SCHULTE and J. S. ZOOK (B.P. 357,561, 20.5.30. U.S., 30.7.29).

**[Plate, separator, container, and venting system for] electric storage cells and batteries.** S. A. POLLOCK (B.P. 357,504—7, 18.3.30).

**Drying of storage-battery plates.**—See I. **Liquid hydrocarbons.**—See II.  $\text{CH}_2\text{O}$  from  $\text{CH}_4$  and  $\text{CO}_2$ . **Hexamethylenetetramine.**—See III. **Purifying acids.**—See VII. **Ni-Co [sealing-in] alloys.** **Roasting of sulphide ores.** **Scrap brass.** **Mg alloys.** **Al and its alloys.** **Cu extraction.** **Ta.** **Sn-plate.** **Plating metal articles.** **Ni-, Cr-, and Cd-plate.** **Coating etc. metals.**—See X.

## XII.—FATS; OILS; WAXES.

**Characterisation and determination of oils and fats.** J. K. CHOWDHURY and S. M. DAS-GUPTA (J. Indian Chem. Soc., 1931, 8, 423–432).—Oils and fats can be characterised by treatment with  $\text{KMnO}_4$  in dry pyridine; the temp. ( $T$ ) at which  $\text{CO}_2$  evolution begins and the amount of  $\text{CO}_2$  evolved (g. per 100 g. of oil =  $\text{CO}_2$  val.) are consts. (within narrow limits) for individual oils. The value of  $T$  for a particular oil is unaffected by the presence of other oils, but free acid lowers  $T$  and increases the  $\text{CO}_2$  val. Adulterants can be detected and determined from the  $\text{CO}_2$  and acid vals. and  $T$ . Mixtures of 2 oils are determined from the  $\text{CO}_2$  val. and  $T$ , whilst mixtures of 3 oils can be determined similarly or, if the constituent oils are known, from the  $\text{CO}_2$  and I vals. Numerous examples are given. Treatment of drying oils with  $\text{KMnO}_4$  in pyridine gives a very viscous product; semi-drying oils become partly viscous, whilst non-drying oils remain mobile.

H. BURTON.

**Analysis of oils and fats, particularly butter.** S. FACHINI and G. DORTA (Atti III Cong. Naz. Chim. pura Appl., 1929, 683–687; Chem. Zentr., 1931, i, 2815).—By precipitation of the K salts in  $\text{COMe}_2$  of suitable concentration, octoic, decoic, oleic, and linoleic acids are separated from lauric, myristic, palmitic, and stearic acids. The separation may be used for the analysis of olive oil and butter. A. A. ELDRIDGE.

**Hydrogenation standardises quality of lard.** M. H. GWYNN and A. P. LEE (Oil and Fat Ind.,

1931, 8, 385, 387).—A small degree of hydrogenation (with deacidification and deodorisation) improves the consistency and keeping properties of lard; the Bolton-Lush continuous process is suitable and shows considerable selective hydrogenation of the linoleic acid present.

E. LEWKOWITSCH.

**Kreis [rancidity] reaction [for rancid fats].** K. TÄUFEL, P. SADLER, and F. K. RUSSOW (Z. angew. Chem., 1931, 44, 873–875).—Since the epihydrinaldehyde is present in rancid fat in the free state to only a small extent, but is liberated by acid, the method of carrying out the phloroglucinol reaction has been modified. The fat is mixed in a test-tube with conc.  $\text{HCl}$  and a plug of white cotton wool, moistened with 1% ethereal phloroglucinol and 2%  $\text{HCl}$ , is pushed a little way down the tube. On careful shaking, without wetting the cotton wool, and if necessary warming at  $60^\circ$ , the characteristic red colour appears on the cotton wool. The limit of sensitivity is about 0.9 microg. of epihydrinaldehyde.

H. F. GILLBE.

**Effect of ultra-violet irradiation on the free sterols of lanoline.** A. BERNHARD and I. J. DREKTER (J. Biol. Chem., 1931, 93, 1–3).—The free sterols precipitable by digitonin and expressed as cholesterol in a sample of anhyd. lanoline increased from 0.93% to 5.37% on irradiation, the max. being attained in about 1 hr.

F. O. HOWITT.

**Chemical composition of certain oils.** W. HUMNICKI (Rocz. Chem., 1931, 11, 678–682).—The sp. gr., sap. val. and I val., and N, P, and lecithin contents of plum-seed oil, rye-embryo oil, refined and crude cod-liver oil, and calf and swine-liver oils are given.  $\text{FeCl}_3$  may be used in place of  $\text{HgCl}_2$  in determining the I val.

R. TRUSZKOWSKI.

**Composition of Philippine peanut oil.** A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1931, 46, 199–207).—Philippine peanuts (Valencia variety) yielded 40% of an oil (purified) which was similar to American peanut oil (cf. B., 1921, 740 A), but contains rather more linoleic and palmitic acids, having: I val. (Hanus) 101.3, saturated acids (corr.) 17.12%, unsaturated acids (corr.) 77.89% (I val. 125). The composition of the oil was determined as: acids (%): palmitic 8.14, stearic 3.43, arachidic 3.25, lignoceric 2.3, oleic 51.54, linoleic 25.88, and unsaponifiable matter 0.29%.

E. LEWKOWITSCH.

**Forming and pressing cakes in the oil mill.** J. G. GIBSON (Oil and Fat Ind., 1931, 8, 383–394, 397).—Analyses of badly prepared press-cakes illustrate the importance of care and uniformity in forming and wrapping the meal.

E. LEWKOWITSCH.

**Relation of the effect of rubber anti-ageing agents on the oxidation of drying oil, and of the anti-oxidising properties of organic compounds to their structure.** V. TANAKA and S. NAKAMURA (J. Rubber Soc. Japan, 1930, 2, 176–186).—The comparative rates of oxidation of linseed oil in presence of various org. antioxidising agents are recorded. The titration acidity was nearly parallel with the increase in wt. The antioxidising agent does not act by formation of a protective film. The action is due to the presence of (1) antioxidant groups  $\text{Ph}$ ,  $\text{C}_{10}\text{H}_7$ ,  $\text{N}\cdot\text{NH}_2$ ,

CN, (2) auxoantioxygenophores  $\text{NH}_2$ , OH, Alk, OMe,  $\text{CO}_2\text{H}$ , (3) oxygenophores Cl,  $\text{NO}_2$ .

## CHEMICAL ABSTRACTS.

**Rancidity and porous packings.**—See XIX. **Grease and oils from sewage. Sapocresols.**—See XXIII.

## PATENTS.

**Production of emulsifying agents and of emulsions prepared therewith.** M. BURAK (B.P. 357,128, 5.6.30. Ger., 7.6.29).—Olein is added to a mixture of soap (or substitutes, e.g., salts of sulpho- or hydro-aromatic acids) and cyclohexanol or its homologues (wt. more than 8% of the org. components). Clear emulsions in  $\text{H}_2\text{O}$  with hydrocarbons etc. are obtained.

E. LEWKOWITSCH.

**Manufacture of sulphonated fatty acid derivatives.** IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, and R. P. MCGLYNN (B.P. 357,670, 10.7.30).—An acylated, unsaturated hydroxy-fatty acid (oil), e.g., acetylated castor oil, is sulphonated, preferably with oleum. The products are wetting-out and emulsifying agents.

E. LEWKOWITSCH.

**Esterification of fatty acids and mixtures containing fatty acids.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,935, 5.4.30).—To prevent preferential esterification of the "normal" portion of a "pathological" fatty acid mixture, such as sanza olive oil, sulphur olive oil, train oil, etc., excess of alcohol (glycerol) is first used and excess of "normal" fatty acid is then added to esterify the remaining OH groups. The unesterified acid is recovered by extraction and added to the next batch.

C. HOLLINS.

**Extractor for sol. substances. Continuous extraction and filtration.**—See I. **Wetting etc. agents.**—See III. **Detergents.**—See VII. **Treatment of [oily] fumes.**—See XIII.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Polish turpentine.** S. KRAUZE (Diss., Univ. Warsaw, 1931, 51 pp.).—Bialowieza forest turpentine was fractionated. Purification is effected with  $\text{H}_3\text{PO}_4$ , not  $\text{H}_2\text{SO}_4$ , and by boiling with Na for 1 hr. The I val. of the crude turpentine is 315–355. The low-boiling fractions contain *d*- and *dl*- $\alpha$ -pinene;  $\beta$ -pinene was not found, but *isopinene* is probably present, together with  $\Delta^3$ - and  $\Delta^4$ -carene.

## CHEMICAL ABSTRACTS.

**Composition of Spanish turpentine from *Pinus pinaster*. Derivatives of  $\alpha$ -pinene.** M. S. ROMERO (Anal. Fis. Quím., 1931, 29, 431–442).—Spanish turpentine from *P. pinaster* has been fractionally distilled under 14–22 mm., and  $[\alpha]_{5780}^{15}$ ,  $[\alpha]_{5461}^{15}$ , and  $d^{15}$  were determined for each of 23 fractions. The composition deduced from the results is: active  $\alpha$ -pinene 56.1%, inactive  $\alpha$ -pinene 8.9%,  $\beta$ -pinene 18.9%, tail fraction 16.0%. Modifications in the prep. of  $\alpha$ -pinene nitroso-chloride and of terpenyl acetate are described.

R. K. CALLOW.

**Precipitated iron oxide [pigments].** A. FOULON (Farbe u. Lack, 1931, 450, 460).—The development of artificial Fe oxide pigments is traced from earlier unsuccessful products prepared from  $\text{FeCl}_3$  and  $\text{Ca}(\text{OH})_2$

to the modern Mars yellow type, e.g., "Neutralin," prepared from  $\text{FeCl}_2$  or  $\text{FeSO}_4$  and  $\text{CaCO}_3$  with subsequent oxidation in the presence, if desired, of a catalyst, e.g.,  $\text{ZnSO}_4$ , and the iron-reds prepared from basic  $\text{FeSO}_4$ . The control of the tone of these products is briefly discussed. Other pigments of this class are the "Veralu reds," by-products of the manufacture of  $\text{Al}_2\text{O}_3$  from bauxite, a typical pigment consisting of 10% of  $\text{TiO}_2$ , 60–65% of  $\text{Fe}_2\text{O}_3$ , and the remainder Ca Al silicates, and Fe oxide blacks obtained by action of aq.  $\text{NH}_3$  on  $\text{FeSO}_4$ – $\text{Fe}_2(\text{SO}_4)_3$  mixtures. The most recent development in this field is the electrolytic method based on anodic oxidation of Fe; patents are reviewed.

S. S. WOOLF.

**Colorimetric determination of iron in titanium pigments.** K. HEISE (Farbe u. Lack, 1931, 464).—The amyl alcohol– $\text{Et}_2\text{O}$  (5:1) mixture used normally in the colorimetric determination of Fe with KCNS is unsuitable for use in determining traces of Fe in  $\text{TiO}_2$  pigments, since such compounds as  $\text{K}_2\text{Ti}(\text{CNS})_4 \cdot \text{H}_2\text{O}$  and  $\text{K}_3\text{Ti}(\text{CNS})_6 \cdot 6\text{H}_2\text{O}$  are formed which give an intense yellow coloration in amyl alcohol, masking the iron-red colour to be measured. The use of pure,  $\text{EtOH}$ -free  $\text{Et}_2\text{O}$  gives satisfactory results.

S. S. WOOLF.

**Dispersion of pigments.** H. HEBBERLING (Farbe u. Lack, 1931, 441–442).—The behaviour of a mixed pigment is a function of the degree of dispersion of each component; thus commercial "zinc greens," which show appreciable variations among themselves, are mixtures of a relatively coarse Zn chromate with a colloidal iron-blue (partly adsorbed) and  $\text{BaSO}_4$  as extender, the packing of such a system greatly influencing its opacity, tone, etc. as a paint pigment. The "extending" of white-lead with  $\text{BaSO}_4$  does not necessarily involve poorer durability, as a structure may be set up improving the hardening-through of the film. The use of  $\text{CaCO}_3$  is also mentioned in this connexion, but its use is limited regionally. The diminution of the staining power of lithopone with falling ZnS content is also discussed and illustrated.

S. S. WOOLF.

**Gum resin.**—See II.  **$\text{Et}_2\text{O}$  extractive of slash pine.**—See IX. **Electrical insulation.**—See XI. **Extracts from conifers.**—See XX.

## PATENTS.

**Removal of paint, varnish, and the like.** J. LEWY (B.P. 357,184, 18.6.30. Ger., 20.6.29).—Compositions of  $\text{CH}_2\text{Cl}_2$  (90 pts.), acetylcellulose (5 pts.), stearin or a fat of similar effect (5 pts.), in the absence of an alcohol or an ether, are claimed.

S. S. WOOLF.

**Producing and clearing printing inks.** H. ZIMMER (B.P. 357,150, 16.6.30. Ger., 27.7.29).— $\text{H}_2\text{O}$ -sol. coal-tar dyes are introduced into the oil base of printing inks, which have first been partly saponified by alkali. In addition to  $\text{H}_2\text{O}$ -sol. dyes, ppts. of these in paste form, with or without a substratum, and oil-sol. dyes may be incorporated.

S. S. WOOLF.

**Production of solutions of nitrocelluloses.** U.S. INDUSTRIAL ALCOHOL CO. (B.P. 357,227, 18.6.30. U.S., 23.7.29).—Mixtures of acetals (30–70%), obtained by condensing  $\text{MeCHO}$  with an aliphatic monohydric

alcohol, *e.g.*, diethyl acetal, and one or more (preferably anhyd.) alcohols (70–30%), *e.g.*, EtOH, are used as nitrocellulose solvents. S. S. WOOLF.

**Reducing the viscosity of nitrocellulose solutions.** W. C. WILSON, Assr. to J. S. STOKES (U.S.P. 1,795,918, 10.3.31. Appl., 11.9.26).—PhOH condensation resins, such as Durite resins (PhOH–furfuraldehyde, preferably including an active methylene compound), are added to viscous solutions of ordinary nitrocellulose; the  $\eta$  is further reduced on keeping or by heating under reflux. E. LEWKOWITSCH.

**Coating compositions.** IMPERIAL CHEM. INDUSTRIES, LTD., D. TRAILL, and A. S. LEVESLEY (B.P. 357,123, 16.4.30).—A high-viscosity cellulose ether (Et or  $\text{CH}_2\text{Ph}$  ether) is converted, *e.g.*, by heating under pressure with dil. acid, into an undegraded ether of low viscosity, *e.g.*, 0.1–5.0 c.g.s. units in 10% solution in PhMe–industrial spirit (4:1). This is incorporated with plasticiser and solvent and, if desired, diluent, resin, and pigment. [Stat. ref.] S. S. WOOLF.

**Marking or decorating articles of thermoplastic materials.** CELLULOID CORP. (B.P. 357,457, 12.1.31. U.S., 11.1.30).—Decorative “characters,” *e.g.*, of cellulose acetate, are applied to thermoplastic articles of the same material, the characters having lower plasticity (according to the amount of plasticiser used) than the articles and thus welding in place under heat and pressure. S. S. WOOLF.

**Pigmented lacquer base.** H. J. HEMINGWAY and W. A. WEIDLICH (U.S.P. 1,798,840, 31.3.31. Appl., 13.10.30).—(EtOH) damp nitrocellulose is kneaded with a portion of EtOH or other non-aq. liquid until the latter is soaked up; the pigment is then dispersed by kneading (not grinding) in, and the remainder of the diluents and solvents are gradually mixed in completely to dissolve the nitrocellulose, and finally to reduce it to suitable fluidity. E. LEWKOWITSCH.

**[Synthetic resin] varnishes.** H. WADE. From BAKELITE CORP. (B.P. 357,140, 14.6.30).—Compositions of non-reactive resins from  $\text{CH}_2\text{O}$  and thiourea (or mixtures thereof with urea) and methylene-containing hardening agents in solid or partly polymerised state, *e.g.*, paraformaldehyde, in proportion to impart to the resin potentially reactive character, and in a common solvent for both, *e.g.*, Et lactate, ethylene glycol, etc., are claimed. S. S. WOOLF.

**Control and treatment of fumes [produced in varnish manufacture].** R. S. PERRY (U.S.P. 1,799,177, 7.4.31. Appl., 10.7.26).—Alkaline sprays directed along the open-ended conduit provide the necessary draught to draw off the fumes and serve to condense and absorb them. The condensates are collected in a gravity separator, whence the alkaline liquid is returned to the system. E. LEWKOWITSCH.

**Extraction of resin from coal.** H. N. SKERRETT. From COMBINED METALS REDUCTION Co. (B.P. 357,733, 22.8.30).—Crude resin is separated from aq. pulp of the powdered coal by froth-flotation, using amyl alcohol (0.6 lb. per ton of coal), turpentine, cresol, etc.; the resin froth is purified by refootation in the presence of K alum (0.4 lb. per ton of original coal). E. LEWKOWITSCH.

**Manufacture of artificial masses from vinyl compounds.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 357,173, 12.6.30).—Viscous plastic or solid compounds of varying solubility are formed by polymerisation (in the absence of a solvent) of a preferably equimolar mixture of an org. vinyl ester and a vinyl ether, or of either with a polymerisable compound which contains the  $\text{CH}_2\text{:C:}$  group but is not a vinyl ester, ether, or halide. E. LEWKOWITSCH.

**[Glass-like resinous] urea condensation product.** O. A. CHERRY, Assr. to ECONOMY FUSE & MANUFACT. CO. (U.S.P. 1,799,954, 7.4.31. Appl., 20.4.29).—The resin obtained by adding sucrose to a urea– $\text{CH}_2\text{O}$  condensation product at any stage prior to gelatinisation may be hardened by heat without risk of cracking. E. LEWKOWITSCH.

**Manufacture of transparent phenol-formaldehyde condensation products.** KUNSTHAARZFABR. DR. F. POLLAK G.M.B.H. (B.P. 357,276, 28.6.30. Austr., 20.3.30).— $1\frac{1}{2}$  mol. pts. of  $\text{CH}_2\text{O}$  are condensed with 1 mol. pt. of PhOH (etc.) in a solution sufficiently alkaline to maintain the resin in solution (cf. B.P. 267,901; B., 1928, 720). The product is then acidified (*e.g.*, with lactic acid), bases and acids being chosen which yield gel-sol. salts (cf. B.P. 290,963; B., 1929, 404), and a further small amount of  $\text{CH}_2\text{O}$  (or its solid polymerides) is added before, during, or after distilling off the  $\text{H}_2\text{O}$  in order to clarify the product before hardening at 60–100°. E. LEWKOWITSCH.

**Compositions of matter and [resinous] moulding mixtures prepared therefrom.** H. WADE. From BAKELITE CORP. (B.P. 357,259, 24.5.30).—A non-reactive fusible phenol-aldehyde resin is treated at about 175° with sufficient of a solid basic oxide, hydroxide, or carbonate of Ca, Mg, Na, etc. to neutralise or combine with the free OH groups of any free phenol and, preferably, also of the resin itself. The product needs no mechanical plasticising and may be readily hot-moulded after comminuting and mixing with fillers and a methylene-containing hardening agent. E. LEWKOWITSCH.

**Manufacture of synthetic resins [from polyhydric alcohols and polybasic acids].** BRIT. THOMSON-HOUSTON Co., LTD., Assecs. of W. C. ARSEM (B.P. 353,249, 23.7.30. U.S., 27.7.29).—The production of resins of the “Glyptal” type is accelerated by addition of 1–5% of an org. sulphonic acid, *e.g.*, *p*-toluidine-*o*-sulphonic acid, Laurent acid,  $\text{PhSO}_3\text{H}$ , ethanesulphonic acid. C. HOLLINS.

**Production of [emulsifiable] paracoumarone resins.** E. H. ELLMS, Assr. to BARRETT Co. (U.S.P. 1,797,260, 24.3.31. Appl., 14.8.28).—Such resins, m.p. above 30°, are prepared by controlling the washing treatments so that the resins may contain total ash within the limits 0.3–1.9%, whilst the ratio amount of  $\text{Na}_2\text{SO}_4$  (ash) corresponding to the org. sulphonic acids of the resin/ $\text{Na}_2\text{SO}_4$  actually present as such is  $\leq 10$  in the case of low-ash resins and  $\leq 1.5$  in the high-ash material. These proportions may also be attained by blending an ordinary paracoumarone resin with paracoumarone sludges from the acid-settling tanks, or soaps from the first washes. E. LEWKOWITSCH.

Containers for liquids. Decolorising agents [for rosin].—See I. [Resin etc. from] ligneous material. Panels for oil paintings.—See V. Colloidal  $\text{TiO}_2$ .—See VII. Moulds for rubber.—See XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Active filling materials [for rubber].** W. BACHMANN, J. BEHRE, and P. BLANKENSTEIN (Kolloid-Z., 1931, 57, 64—80; cf. B., 1931, 1020).—The plasticity of non-vulcanised rubber is affected by the filling material and the treatment of the mixture, but the particle size of the filler has no important influence and plasticity alone is no measure of the value of a filler. The effect of the filler on "nerve" is doubtful. Surface tension and particle size of caoutchouc depend on the degree of kneading. Ultrafiltration experiments show caoutchouc solutions to be polydisperse, the approx. mol. wt. being 300,000 for weakly rolled material and 37,000 for very strongly rolled material. Rolled caoutchouc swells less strongly than the untreated material and the velocity of swelling is reduced by fillers. The adsorptive capacity of certain  $\text{SiO}_2$  preparations for  $\text{C}_6\text{H}_6$  vapour is better than that of lampblack. Vulcanised rubber with certain  $\text{SiO}_2$  preparations as fillers is equal in tensile strength, hardness, elasticity, and wear to that containing lamp-black.

E. S. HEDGES.

**Value of zinc sulphide as a rubber-compounding ingredient.** K. NAMITA (J. Rubber Soc. Japan, 1930, 2, 157—159).—Tensile strength, electrical insulating power, and whiteness are greater if  $\text{ZnS}$  is substituted for  $\text{ZnO}$ . Vulcanisation acceleration is greater with  $\text{ZnS}$ .

CHEMICAL ABSTRACTS.

**Effect of gas-black on rubber.** P. STAMBERGER (Kautschuk, 1931, 7, 182—186).—Mixtures of rubber with C black exhibit marked resistance to ordinary rubber solvents. The decrease in solubility has no parallelism with the reduction in plasticity, and flocculation of the C black in the rubber is probably not responsible, because the effect develops in the masticated mixture only after storage. No adsorption of rubber from solution by C black could be detected, and the heat of wetting of the black, and the quantity of gas concurrently liberated, were the same whether wetting was effected with  $\text{PhMe}$  or with a 5% solution of rubber in  $\text{PhMe}$ .

D. F. TWISS.

**Effect of selenium in rubber mixings.** W. ESCH (Kautschuk, 1931, 7, 190—192). H. RIMPEL (*Ibid.*, 192—194).—Mainly polemical (cf. B., 1931, 643).

D. F. TWISS.

**Comparison of stearic and sebacic acids as rubber-softening agents.** K. ISHIGURO (J. Rubber Soc. Japan, 1930, 2, 238—242).—Sebacic acid, when used in excess with  $\text{ZnO}$ , is a better softening agent than stearic acid. When thus used, sebacic acid improved the quality of the rubber and did not retard the action of vulcanisation accelerators. CHEMICAL ABSTRACTS.

**Effect of rubber compounding on the sp. gr. of vulcanised rubber.** K. NAMITA, T. NAKAJIMA, and K. TSUTSUNI (J. Rubber Soc. Japan, 1931, 2, 408—413).—The formulæ  $y = 0.95 + mx$  ( $y$  = sp. gr. of compounded rubber,  $x$  = vol.-% of compounding substance,  $m$  = const.) and  $y = 0.95(x + 1)^b$  ( $x$  =

wt.-% of compounding substance,  $b$  = const.) give better results than those usually employed.

CHEMICAL ABSTRACTS.

**Determination of carbon black in vulcanised rubber.** K. NAMITA (J. Rubber Soc. Japan, 1930, 2, 255—260).— $B = [F - \Sigma(1 - F_u)D_u]/(1 - K)$ , where  $B$  = % C,  $D_u$  = % of each ingredient,  $F_u$  = const. for each ingredient,  $F$  = wt. of residue after combustion in N by Ooyaji's method, and  $K$  = const. for C. Values of  $F_u$  are given. Low results are obtained when  $\text{CaCO}_3$  is present, owing to the reaction  $\text{CO}_2 + \text{C} = 2\text{CO}$ .

CHEMICAL ABSTRACTS.

**Determination of sulphur in rubber by the calorimetric bomb.** B. SALADINI (Giorn. Chim. Ind. Appl., 1931, 13, 409—411).—The methods of determining S in rubber by oxidation in an open vessel with  $\text{Na}_2\text{O}_2$ ,  $\text{HClO}_4$ , or  $\text{K}_2\text{CO}_3$ - $\text{KNO}_3$  mixture are long and subject to losses by projection or by incomplete oxidation. A more rapid and accurate method consists in combusting the rubber (0.1—0.3 g.) mixed with Decalin (about 1 c.c.) (to ensure complete combustion) in a Kroecker calorimetric bomb charged with  $\text{O}_2$  at 30 atm. and containing about 10 c.c. of  $\text{H}_2\text{O}$  to condense the  $\text{SO}_3$  formed; this is weighed as  $\text{BaSO}_4$  in the usual way.

T. H. POPE.

**Fluorescence of organic vulcanisation accelerators.** K. KOJIMA and I. NAGAI (J. Rubber Soc. Japan, 1930, 2, 260—262).—Fluorescence after exposure to ultra-violet light is in some cases useful for identification.

CHEMICAL ABSTRACTS.

**Combination of organic accelerators for rubber vulcanisation.** II. K. KOJIMA and I. NAGAI (J. Soc. Chem. Ind., Japan, 1931, 34, 308—309 B; cf. B., 1931, 643).—Mercaptobenzthiazole and methylenedianiline form a eutectic mixture, but no mol. compound. A mixture of the two accelerators is more active than either constituent in expediting vulcanisation, and the activity attains a max. at the eutectic. The scorching tendency, however, increases with the proportion of mercaptobenzthiazole, and the eutectic mixture exhibits no exaltation of this characteristic at 70° or 100°.

D. F. TWISS.

**Vulcanisation experiments with the accelerator-activator "Barak."** E. A. HAUSER and H. WOLF (Kautschuk, 1931, 7, 186—188).—The use of the org. activator for org. accelerators enables satisfactory vulcanisation results to be obtained with a reduced proportion of org. accelerator or of S; the "plateau effect" is improved and subsequent blooming of S is lessened. With mixings made from conc. latex (revertex) good results could be obtained when the proportions of the vulcanising agents were still further reduced.

D. F. TWISS.

**Thermochemistry of rubber.** I. Heat of vulcanisation. K. NAMITA, K. FUKAYA, and T. NAKAJIMA (J. Rubber Soc. Japan, 1931, 2, 389—397).—The vulcanisation of soft rubber up to 4% combined S is endothermic; above 4% it is exothermic (max. at 12%), decreasing to 22%, when hard rubber is formed. Above 22% the reaction is endothermic.

CHEMICAL ABSTRACTS.

**Heat of reaction during vulcanisation of rubber.** Y. TOYABE (J. Rubber Soc. Japan, 1930, 2, 251—254).—



The heat of vulcanisation becomes max. in 1 hr., but in presence of accelerators the max. is reached immediately. At the point of max. heat of vulcanisation about 50% of S is combined with the rubber. Addition of accelerators appears to cause a great change in the state of aggregation of the rubber mol.

## CHEMICAL ABSTRACTS.

**Chemical risks in the rubber industry and their prevention.** R. DITMAR (Chem.-Ztg., 1931, 55, 770—772).—Reference is made to the possibility of mouldiness developing with latex or rubber and to the harmful physiological action of certain compounding ingredients and solvents.

D. F. TWISS.

**Rubber anti-ageing agents.**—See XII.

## PATENTS.

**Manufacture of adhesive materials from rubber latex.** IMPERIAL CHEM. INDUSTRIES, LTD., R. B. F. CLARKE, E. B. ROBINSON, and A. SHEPHERDSON (B.P. 357,238, 19.6.30).—Latex is creamed by the addition of a weak org. acid without coagulation; a strong acid, e.g.,  $\text{H}_2\text{SO}_4$  or benzenesulphonic acid, or an acid-forming substance is added, and after evaporation of the volatile matter, the residuum is baked, e.g., at  $150^\circ$ , until chemical reaction occurs between the non-aq. constituents of the latex and the acidic material. The dark-coloured product is useful as an adhesive for attaching rubber to metal surfaces.

D. F. TWISS.

**Manufacture of rubber or the like material for covering floors and the like.** DUNLOP RUBBER CO., LTD., and H. C. YOUNG (B.P. 357,212, 14.6.30 and 3.3.31).—Threads, strips, or cords of semi-vulcanised rubber are woven alone or in combination with textile material; vulcanisation is then effected under pressure so as to effect consolidation and impart a smooth surface (cf. B.P. 319,448; B., 1929, 991).

D. F. TWISS.

**Metal moulds [for shaping rubber articles].** BAKELITE CORP., ASSEES. of G. W. CROSBY (B.P. 354,770, 6.3.30. U.S., 7.3.29).—The part forming the pattern, e.g., the tread of tyres, is made of moulded bakelite. (Cf. B.P. 352,519; B., 1931, 897.)

A. R. POWELL.

**S product.**—See VII. **Dielectric material.**—See XI. **Chewing gum.**—See XIX.

## XV.—LEATHER; GLUE.

**Swelling.** II. **Effect of acids on hide powder.** A. LOTTERMOSER and F. TACHECI (Kolloid-Z., 1931, 57, 56—63; cf. A., 1931, 1232).—Strong acids are taken up to a saturation limit, which is approx. the same for each acid. Weak acids do not attain a saturation limit, the amount taken up increasing continuously with the concentration, probably because the hydrolysis of the compound formed between the weak base collagen and the weak acid is repressed by higher concentrations of acid. The swelling curves in the same acids are similar to the curves representing the acid-binding power, and there is no reason to doubt the existence of a true chemical combination between hide powder and acids.

E. S. HEDGES.

**Some organic constituents (non-tans) of vegetable tanning extracts with special reference to chestnut wood extracts.** I. **Pentose and uronic**

**acid-containing constituents.** II. PHILLIPS (J. Soc. Leather Trades' Chem., 1931, 15, 465—479).—Encrusting substances consisting of hexoses, pentoses, and their uronic acids are present in tannin extracts, especially those derived from woods. Determinations were made of the amounts of pentoses and uronic acid in some tanning extracts and in a series of commercial chestnut extracts; the latter were the richer in pentoses. The amount of encrusting substances extracted from tanning woods was increased by raising the temp. of extraction. The amount of pentoses and uronic acids in chestnut liquors used in sole-leather tannages was not materially increased on prolonged usage, and only small quantities were absorbed by the hides during the tannage. They were not present in tan-pit sludge, but tend to accumulate in the suspender liquors in tanneries where chestnut extract is used. They constitute 20% of the org. non-tans in such liquors and the uronic acids act as buffers.

D. WOODROFFE.

**Deterioration of vegetable-tanned [book-binding] leather on storage.** IV. **Conditions of storage and the rotting of leather by artificial means.** R. F. INNES (J. Soc. Leather Trades' Chem., 1931, 15, 480—494; cf. B., 1931, 130).—Pyrocatechol-tanned book-binding leathers, dyed in the presence of  $\text{H}_2\text{SO}_4$ , remained sound after long storage in the dark in a polluted atm. restricted in circulation. Rotting is chiefly caused by oxidation in the presence of  $\text{H}_2\text{SO}_4$ . Leathers of different tannages were not rotted by exposure to acid gas fumes for 88 days, nor by exposure to ultra-violet light, nor by  $\text{H}_2\text{O}_2$ , but were rotted by treatment with  $\text{H}_2\text{SO}_4$  and after-treatment with  $\text{H}_2\text{O}_2$ , the leather appearing burnt and blackened. No red powdery rotting was obtained.

D. WOODROFFE.

**Water-permeability of leather.** R. S. EDWARDS (J. Soc. Leather Trades' Chem., 1931, 15, 495—514).—An apparatus for determining the water-permeability of leather is described. The vol. of  $\text{H}_2\text{O}$ ,  $V$ , entering the leather and the time,  $t$ , required were measured; the relationship  $V = At^n$  where  $A$  and  $n$  are consts. was established. The amount of  $\text{H}_2\text{O}$  which permeated the leather and the rate of evaporation of the  $\text{H}_2\text{O}$  from the air side of the leather were greater as the R.H. was diminished on the dry side of the leather. There was a sudden increase in the rates of absorption and evaporation of the  $\text{H}_2\text{O}$  at the time when the leather was completely permeated, so that this inflexion in the curve might be used to determine when the leather was completely penetrated.

D. WOODROFFE.

**Examination of dyed leather in cases of alleged dermatitis.** T. CALLAN and N. STRAFFORD (Analyst, 1931, 56, 625—635).—10 g. of leather are extracted for 48 hr. with 40 c.c. of 1% AcOH in the cold, and tests are carried out with 0.5 c.c. of the extract together with control portions of the extract to which small quantities of *m*- and *p*-phenylenediamine have been added. The most sensitive reagent for the *p*-diamine is a 1% EtOH solution of *p*-dimethylaminobenzaldehyde in the presence of a trace of HCl; this will detect 0.01% (calc. on the leather) in a coloured extract containing tannins. 0.5 c.c. of 1%  $\text{NH}_2\text{Ph}\cdot\text{HCl}$  and a drop of 2%  $\text{K}_2\text{Cr}_2\text{O}_7$

is equally delicate, when  $\text{FeCl}_3$  indicates the presence of more than traces of tannins. 0.02% of the *m*-diamine is indicated by the following reagents: (1) 0.2 c.c. of 2*N*-HCl and 0.05 c.c. of 0.5*N*- $\text{NaNO}_2$  mixed with the extract and added to 4 c.c. of 0.05%  $\beta$ -naphthol and 1 c.c. of NaOH; (2) *p*-dimethylaminobenzaldehyde reagent as above; (3) 1 g. of NaOAc and 1 c.c. of diazobenzene-*p*-sulphonic acid. Single tests are insufficient; several reagents must be used and the results interpreted accordingly. T. McLACHLAN.

Extracts from ethereal oils.—See XX. Waste waters from leather factories.—See XXIII.

## XVI.—AGRICULTURE.

**Report of the Imperial Agricultural Bacteriologist [Pusa].** J. H. WALTON (Agric. Res. Inst., Pusa, Sci. Rep., 1929—30, 49—57).—(a) Addition of 1—2% of superphosphate to cow manure greatly diminishes loss of N during storage. (b) In soils of  $p_H$  7.0 or higher, urea was not formed from  $\text{CaCN}_2$ , which persisted for 3 days, no nitrification occurring after incubation for many weeks. In soils of  $p_H$  5.0—6.9 urea was formed. (c) The varying effects of addition of  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{SO}_4$ , or NaCl on nitrification were studied. CHEMICAL ABSTRACTS.

**Report of the Imperial Agricultural Chemist [Pusa].** J. SEN (Agric. Res. Inst., Pusa, Sci. Rep., 1929—30, 33—48).—(a) During curing tobacco loses 18—35% of the nicotine. Ground-cured contains more nicotine than rack-cured tobacco, especially that cured in the sun. Of the starch, 50—80% was lost, the greatest losses occurring in ground-cured samples. The sugar content was increased by rack-curing, but decreased by ground-curing. The  $\text{NH}_2\text{-N}$  was lowest in ground-cured samples. The upper leaves were generally richer in  $\text{NH}_2\text{-N}$  and nicotine than the lower. (b) Unfertilised plots yielded sugar-cane juice richest in sucrose and poorest in dextrose. The largest crop was obtained by the use of mustard cake, superphosphate, and  $\text{K}_2\text{SO}_4$ . (c) In potatoes stored at 33—38° the total org. N remained const., but the dextrin and  $\text{NH}_3\text{-N}$  increased. (d) The effect of treatment of Pusa soil with aq. NaCl on the results of mechanical analysis is described. CHEMICAL ABSTRACTS.

**Soils of the Nile and Gash. IV.** P. VAGELER and F. ALTEN (Z. Pflanz. Düng., 1931, 22A, 191—267; cf. B., 1931, 1022).—The water relationships of these soils are examined in reference to base contents and physical properties. The application of various methods of determining soil nutrient vals. is considered with special reference to the *Aspergillus* method. A. G. POLLARD.

**New methods for examination of organic matter in soil and their application to soil types and humus material. I. Methods.** U. SPRINGER (Z. Pflanz. Düng., 1931, 22A, 135—152).—Methods for separating the constituents of humus are critically examined. Special attention is given to the AcBr method and to methods involving differential oxidation of the separated material. (Cf. B., 1928, 619.)

A. G. POLLARD.

**Determination of the buffer capacity of the soil by the Tovborg-Jensen method.** N. N. SOLOVEVA (Udobr. Urozhai, 1930, 2, 784—789).—A sandy podsol requires more CaO than a loam podsol. A certain

correlation exists between the buffer surface activity and the hydrolytic acidity. CHEMICAL ABSTRACTS.

**Laws of alkali-soil formation in reference to soil amelioration.** A. A. J. SIGMOND (Mezőg. Katalások, 1929, 2, [6]; Bied. Zentr., 1931, [ii], 1A, 361—362).—The factors essential to alkali-soil formation are a dry climate, an impermeable subsoil, and occasional excessive moisture. The initial phase is the accumulation of NaCl,  $\text{Na}_2\text{SO}_4$ ,  $\text{NaHCO}_3$ , and  $\text{Na}_2\text{CO}_3$ . In this stage simple leaching suffices for amelioration. The second phase, "alkalination," consists of the exchange of the Na with the adsorbed bases of the soil. For soils in this phase replacement of exchangeable Na by Ca is essential for soil improvement. In the third phase  $\text{H}_2\text{O}$ -sol. salts are leached out and either are removed entirely or accumulate in the subsoil. Such soils have a weakly acid reaction due to the hydrolysis and partial replacement of exchangeable Na by H. Reclamation of this type of soil must include applications of Ca and of org. matter. Alkali soils containing  $\text{Na}_2\text{CO}_3$  are not effectively treated by means of  $\text{CaCO}_3$  since acid conditions are necessary for the dissolution and subsequent action of Ca. This may be ensured by secondary treatment with  $\text{H}_2\text{SO}_4$ , S, or Ca, Al, or  $\text{Fe}^{+++}$  sulphates.

A. G. POLLARD.

**Effect of certain simple non-nitrogenous salts on the growth of bacteria in soil.** H. J. CONN (J. Bact., 1931, 21, 35).—The growth of certain non-sporing organisms in soils in which they do not grow naturally may be secured by the addition of glucose and salts of  $\text{NH}_4$  or  $\text{NH}_2$ -acids. Similar growth may be obtained by the substitution for these Na compounds of certain non-nitrogenous compounds, notably K salts or KOH, certain Na and Ca salts, or NaOH. Sulphates, carbonates, phosphates, and hydroxides are very effective, but chlorides appear to be toxic. The above compounds are assumed to act by rendering available the soil N, either by chemical decomp. or by liberation from colloidal complexes in which it is adsorbed.

A. G. POLLARD.

**Adsorptive power of soils.** K. K. GEDROIZ (Kolloidchem. Beih., 1931, 33, 317—448).—A summary of the results obtained from 20 years' work on the mechanical, physical, chemical, and biological binding power of soils, most of which has already been published. The principal features discussed are the mechanical structure of the soil, adsorption and surface tension, capillary electric properties and coagulating effect of electrolytes, influence of acidity or alkalinity, coagulation by oppositely-charged colloids, exchange adsorption between soils and electrolytes in solution, physico-chemical nature of the adsorbing soil complexes and the effect of  $\text{H}_2\text{O}$ , determination of adsorbed substances, chemical binding of  $\text{PO}_4$ ,  $\text{CO}_3$ , and  $\text{SO}_4$  ions by soils, and biological relations.

E. S. HEDGES.

**Water supply as a factor in the distribution of plants.** F. SEKERA (Z. Pflanz. Düng., 1931, 22A, 152—190).—Relationships between the RK val. of soils ("Regen-Kapazität," i.e., mm. of rain which a cropped soil can store) and plant growth are examined. The use of RK vals. in the classification of soils and in the calculation of crop yields is demonstrated. A. G. POLLARD.

**Drainage waters from loams and moor soils.** O. BRAADLIE (Tidsskr. norske Landbruk, 1930, 341; Bied. Zentr., 1931, [ii], 1A, 363—364).—In leaching experiments, the removal of Ca, P,  $\text{NO}_3'$ ,  $\text{NH}_3$ , and total N was greater, and that of K, total org. matter, and org. N was less, from a loam than from a moor soil. Changes in  $p_{\text{H}}$  due to leaching were small in both soils.

A. G. POLLARD.

**Hydrolytic acidity and lime requirement.** K. K. GEDROIZ (Udobr. Urozhai, 1930, 2, 781—784).— $\text{CO}_2$  from the air and from the soil produces  $\text{HCO}_3'$  and hence an acid reaction to phenolphthalein when a carbonate soil is treated with  $\text{Ca}(\text{OAc})_2$ .

CHEMICAL ABSTRACTS.

**Phosphate content of soils.** E. G. DOERELL (Superphosphat, 1931, 7, 59—60; Bied. Zentr., 1931, [ii], 1A, 355—356).—Comparison of field results with various laboratory methods shows the practical value of Neubauer and Dirks' methods for determining the available P of soils.

A. G. POLLARD.

**How reliable are existing chemical methods for determining soil deficiencies in ash constituents of plants?** G. S. FRAPS (J. Amer. Soc. Agron., 1931, 23, 337—351).—Factors other than the capacity of the soil to supply plant food are concerned.

CHEMICAL ABSTRACTS.

**Effect of roasting of phosphorites on their assimilability [by plants].** N. D. SMIRNOV (Düng. u. Ernte, 1929, 359—363; Bied. Zentr., 1931, [ii], 1A, 365).—Of numerous samples examined, the majority of phosphorites were less assimilable after roasting. Crop increases were greater from samples heated at  $300^\circ$  than from those heated at  $800^\circ$ . The response of black-earth soils and of podzols to phosphorites were definitely different.

A. G. POLLARD.

**Comparative effect of superphosphate and precipitated phosphate.** D. V. DRUZHININ and Z. I. STROGANOVA (Udobr. Urozhai, 1930, 2, 895—903).—With soils of various zones slight differences were observed. Soils which do not respond well to  $\text{P}_2\text{O}_5$  fertilisation give better results with superphosphate.

CHEMICAL ABSTRACTS.

**Phosphoric acid and potash from manure under various conditions of storage.** I. F. ROMASHEVICH (Udobr. Urozhai, 1930, 2, 769—773).—After four months the amount of  $\text{P}_2\text{O}_5$  sol. in HCl increased threefold for peat moss- and straw-manure (88% cow, 12% horse); the  $\text{H}_2\text{O}$ -sol.  $\text{P}_2\text{O}_5$  also increased. In uncompacted manure the loss of  $\text{P}_2\text{O}_5$  was 21.3%, 1% of this being lost with the liquid. Fertilisation experiments with oats and compacted manure showed that the  $\text{P}_2\text{O}_5$  from peat moss- and straw-manures became available to the extent of 74% and 59.2%, respectively, corresponding vals. for  $\text{K}_2\text{O}$  being 82.2% and 84.6%.

CHEMICAL ABSTRACTS.

**Rate of intake of the  $\text{PO}_4$  ion by various agricultural plants.** F. PEIPERS (Diss., Hohenheim, 1930; Bied. Zentr., 1931, [ii], 1A, 370—371).—The intake of P by maize was more rapid from nutrient solutions containing 0.1—10.0 mg./litre than from those with <0.1 mg./litre, whether considered as % or in abs. units. With more conc. solutions (10.0—210 mg./

litre), the abs. intake reached a max., but the % removed from the solution declined rapidly. The absorption of P by the plants was not influenced by the intensity of illumination or by temp. except in extreme cases. Older plants absorbed more P from solutions than young ones, although the % P in the roots was always greater in the younger plants.

A. G. POLLARD.

**Fertilising limed soils.** S. S. YARUSOV (Udobr. Urozhai, 1931, 3, 44—53).—Limed medium podsolised soils gave an increased yield of oats. They were low in N; K fertilisers were effective, but phosphates were ineffective.

CHEMICAL ABSTRACTS.

**Effect of mineral nutrition on the reaction of wheat varieties to leaf rust.** K. D. DOAK (Phytopath., 1931, 21, 108—109).—In artificial cultures N increased the susceptibility of wheat to leaf rust, whilst K and P reduced this. Excess N favoured the production of uredinia and decreased chlorosis. Excess P increased chlorosis and retarded the development of secondary uredinia. Excess of K increased chlorosis and decreased the size of primary uredinia.

A. G. POLLARD.

**Mottling of the leaves of cereals as a phenomenon of magnesium deficiency.** W. JESSEN (Z. Pflanz. Düng., 1931, 22A, 129—135).—The "mottling" or "marbling" of cereal leaves is not a manifestation of acid soil conditions, but is the result of Mg deficiency.

A. G. POLLARD.

**Expressed sap of maize plants as an indicator of nutrient needs.** N. A. PETTINGER (J. Agric. Res., 1931, 43, 95—119).—The brown coloration of the expressed sap of maize which remains after clarification with charcoal is greatest in plants from least productive soils. There was a close inverse relationship between the intensity of the colour and the available K content of the soil. The  $\text{NO}_3'$  content of the sap was closely correlated with that of the soil carrying the plant, and the P content of the sap with the amount of P fertiliser applied. Applications of superphosphate and of farm-yard manure increased the P content of the sap 2—5 times. Rock phosphates were less effective in this respect. The P content of the sap was high where grain production was subnormal and *vice versa*. After the development of the ear there is a rapid accumulation of P in the sap, irrespective of the fertiliser applied; this is accompanied by a similar but smaller accumulation of K. A scale of limiting concentrations of N, P, and K in the sap as indicative of the nutrient contents of the soil is given. The  $p_{\text{H}}$  of the sap is closely related to the amounts of K fertiliser applied (fertilised plots showing vals. > 5.45), but not with the soil productivity. The depth of green colour in the leaves is a good indicator of N deficiency and is in agreement with the results of the Hofferstalk test and the  $\text{NO}_3'$  concentration in the sap. K deficiency is shown by the presence of dead tissue round the margins and between the veins of the leaves. The K requirement of soils is closely in agreement with the amount of lodging of the crop.

A. G. POLLARD.

**Fertiliser in the northern portion of the deep chernozem.** A. G. IVANOV (Udobr. Urozhai, 1930, 2, 759—764).—N is not the limiting factor; the soil does

not respond to N fertilisation. P is minimal. The sugar content of beet decreases when  $(\text{NH}_4)_2\text{SO}_4$  is used.

## CHEMICAL ABSTRACTS.

**Influence of nitrogen fertilisers on composition of soil solution.** E. A. ZHORIKOV (Udobr. Urozhai, 1930, 2, 774—781).— $\text{NO}_3^-$  accumulates; little  $\text{NH}_3$  is retained, except with  $\text{CaCN}_2$ , which gave high amounts of  $\text{NH}_3$ , depressing the  $\text{NO}_3^-$ . Mineral N gave the highest accumulation of  $\text{NO}_3^-$ , oil meal less, and manure and green manure least. Increase in  $\text{NO}_3^-$  increases the Ca, Mg, total sol. substances, and yield of cotton, but decreases the total alkalinity and  $\text{P}_2\text{O}_5$ .

## CHEMICAL ABSTRACTS.

**Influence of nitrogen, phosphoric acid, and potash on the number, shape, and weight of potato tubers.** W. H. MARTIN, B. E. BROWN, and H. B. SPRAGUE (J. Agric. Res., 1931, 43, 231—260).—Fertilisers of different N : P : K ratios tended to produce tubers of varying predominant shape, the major effects being attributable to the proportion of K used. Wide potatoes are associated with fertilisers of low P content; thick potatoes with low P and medium to high K content; thin and narrow tubers with low N and K and high P; long tubers with medium to high N and P and low K; and short tubers with low N, low to medium P, and medium to high K. In general, the largest potatoes result from high N and low P, the largest numbers of tubers from low N and high P, and the fewest potatoes with high N and low K. The largest total yields were obtained with medium to high N and K and low P. Additional details of the proportions of different grades of potatoes in the various fertiliser trials are recorded.

A. G. POLLARD.

**Effect of different pressures and of different types of lime in potato spraying.** P. E. TILFORD (Phytopath., 1931, 21, 105).—Potato spraying with Bordeaux mixture was more successful at 400 lb. pressure than at 200 or 600 lb. Slaked limes of high Ca content gave as good results as quicklimes and better than slaked limes of high Mg content in the prep. of Bordeaux mixture.

A. G. POLLARD.

**Use of fertilisers in reducing losses from pea-root rot caused by *Aphanomyces euteiches*.** C. M. HAENSELER (Phytopath., 1931, 21, 116—117).—The use of complete fertiliser mixtures decreased the root rot. In this respect  $\text{NaNO}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ , and KCl were more effective than was superphosphate.

A. G. POLLARD.

**Modification of soil-nitrogen and -organic matter by Austrian winter peas.** M. M. OVESON and W. L. POWERS (J. Amer. Soc. Agron., 1931, 23, 372—387).—The growth of the peas as a green manure crop increased the soil-N and -org. matter. The soil total N remained unchanged when the crop was harvested, when the total N balance was greater than when the crop was turned under. Changes in org. C content tended to vary with those of N content. Addition of lucerne tops to the soil decreased the amount of N fixed by the pea crop, but helped to maintain a more nearly normal N : C ratio.

## CHEMICAL ABSTRACTS.

**Seed treatment for damping-off of tomatoes.** J. G. HORSFALL (Phytopath., 1931, 21, 105).—In cases where the steaming of tomato soils is impracticable,

good results are obtained by soaking in 5%  $\text{CuSO}_4$  solution for 1 hr. or dusting with  $\text{CuSO}_4 \cdot \text{H}_2\text{O}$  or  $\text{CuCO}_3$ .  $\text{Cu}^{++}$  oxalate, tartrate, sulphide, silicate, arsenate, oxide, and oxychloride and  $\text{Cu}^+$  chloride, bromide, sulphide, thiocyanate, and also malachite reduced the disease. The germination of the seeds was not reduced and in many cases was considerably improved. Hg compounds reduced the disease, but also impaired germination.

A. G. POLLARD.

**Manuring of sugar beet in Kiev. I. Collective results for 1927.** P. BRASCHNIK and W. FANSTIL. **II. Results for 1928.** P. BRASCHNIK (Kiew. Landw. Versuchs-stat., 1929, 35, 30, and 1930, 57, 38; Bied. Zentr., 1931, [ii], 1A, 380—381).—Fertiliser trials are recorded and discussed.

A. G. POLLARD.

**Effect of seed treatments on seed longevity.** E. E. CLAYTON (Phytopath., 1931, 21, 105—106).—Commonly used wet treatments for seeds, e.g.,  $\text{HgCl}_2$ , org. Hg. compounds, hot  $\text{H}_2\text{O}$ , etc., whilst causing no apparent injury immediately, reduce germination after storage. Dust treatments have no ill after-effects. Seed treated with 0.25%  $\text{ZnSO}_4$  solution for 25 min. at  $50^\circ$  produced beneficial results to seed, even when stored for 18 months after treatment.

A. G. POLLARD.

**Corm treatments for gladiolus and calla lily.** P. E. TILFORD (Phytopath., 1931, 21, 121—122).—Standard treatments were unsatisfactory. Good results were obtained by dipping in a suspension of  $\text{Hg}_2\text{Cl}_2$  (1 lb. per 2½ gals.) and in Semesan solution.

A. G. POLLARD.

**Effect of some inorganic and organic mercurials on growth of *Lupinus albus*.** D. J. MACHT (Amer. J. Bot., 1931, 18, 598—602).—Inorg. Hg compounds were more toxic than org. preps. to lupin seedlings. Adulteration of org. Hg compounds, notably mercuriochrome, with  $\text{HgCl}_2$  may be detected by the phytotoxic test with lupins.

A. G. POLLARD.

**Control of *Botrytis* rot of pears with chemically treated wrappers.** J. S. COOLEY (U.S. Dept. Agric. Circ., 1931, No. 177, 9 pp.).—Impregnation of wrapping papers with 2.5%  $\text{CuSO}_4$  solution prevented the spread of *Botrytis* in packed pears.  $\text{ZnSO}_4$  was slightly, and  $\text{FeSO}_4$  considerably, less effective. Satisfactory results from oiled paper treated with  $\text{CuSO}_4$  are indicated. No injury from  $\text{CuSO}_4$  was observed unless the fruit was packed in a damp condition.

A. G. POLLARD.

**Breeding of nicotine-free tobaccos and those poor in nicotine.** R. VON SENGEBUSCH (Der Züchter, 1931, 3, 33; Bied. Zentr., 1931, [ii], 1A, 375).—From normal strains of tobacco plants it is possible to breed strains of reduced nicotine content. Methods for the determination of nicotine are discussed.

A. G. POLLARD.

**Burning quality of tobacco.** M. B. SMITS (Int. landw. Runds., Agrik.-wiss. Runds., 1930, 21, 485; Bied. Zentr., 1931, [ii], 1A, 375—376).—In wet seasons tobacco plants contain less Cl, Ca, Mg, N, P, S, and Mn and more K,  $\text{SiO}_2$ , Fe, and Al than in dry years; the former also have superior burning qualities. Good burning qualities are associated with an alkaline reaction and with relatively high K contents. Soils of high Cl

content tend to produce poor-burning tobacco. The general effects of climatic conditions on the quality of tobacco are discussed. A. G. POLLARD.

**Plant development under Vitaglass.** W. E. TOTTINGHAM and J. G. MOORE (J. Agric. Res., 1931, 43, 133—163).—In a comparison of the growth of various plants under Vitaglass and under ordinary glass, certain species showed the response to Vitaglass which might be anticipated from the slightly increased temp. caused by the additional infra-red transmission. In many cases there was an increased lipin-N content in the dry matter of the plants. A. G. POLLARD.

**Guano.** M. POPP and J. MARXEN (Landw. Versuchsstat., 1931, 112, 261—312).—The N of guano exists in the form of urea (50%),  $\text{NH}_3$  (30%), purines (10%), with smaller proportions of keratin and frequently of nitrate. A portion of the  $\text{NH}_3$ -N is easily sol., the sparingly sol. portion being probably  $\text{NH}_4$  urate. Present-day guano contains 6% of  $\text{H}_2\text{C}_2\text{O}_4$  and 1% of guanine. In soil the org. matter of guano is rapidly decomposed to form  $\text{CO}_2$ , the yield of which is approx. 35 times that from ordinary soil org. matter. A considerable portion of the N-free org. matter of guano has a C content intermediate between that of urea and humus. In older guano deposits these compounds predominate. The P and N contents of guano are inversely related. The ageing of guano results in an increased proportion of P and decreased N. A high proportion of guano-P is sol. in 2% citric acid and a smaller amount in  $\text{NH}_4$  citrate. In Seychelles guano of low N content  $\text{NH}_4$  citrate-sol. P is absent, and the citric acid-sol. fraction is approx. 50%. In Peruvian guano a portion of the P exists as Mg phosphates. A. G. POLLARD.

**Chemical processes involved in the decomposition of manure by *Agaricus campestris*.** S. A. WAKSMAN and J. M. McGRATH (Amer. J. Bot., 1931, 18, 572—581).—During the composting of horse manure for mushroom beds, the  $\text{Et}_2\text{O}$ -sol. constituents and hemi-cellulose decreased rapidly. The  $\text{H}_2\text{O}$ -sol. org. matter increased slightly during composting and rapidly as the mushroom mycelium developed. The cellulose content did not change appreciably during composting, but decreased rapidly with the growth of mycelium. There was a steady rise in the lignin and protein contents during composting, and early development of mycelium, followed by a rapid decline during the period of most active growth. Lignins, proteins, and celluloses are probably directly concerned in the development of mycelium. A. G. POLLARD.

**Relation of pentathionic acid and its constituents to the toxicity of sulphur fungicides.** O. N. LIMING (Phytopath., 1931, 21, 130—131).—At summer temp. >10% of the S dusted on foliage may be vaporised within 14 days. S vapour is not toxic to fungus spores and the condensation products are toxic only after several hours' contact.  $\text{SO}_2$  occurs only in traces in ground S and is not toxic at these concentrations.  $\text{H}_2\text{S}$  does not occur in ground S, but is present in non-toxic amounts when S is in contact with higher plants and fungi.  $\text{H}_2\text{S}_5\text{O}_6$  is associated with ground S in toxic quantities. The  $\text{S}_5\text{O}_6^{--}$  ion is stable in acid and weakly alkaline solutions, but is toxic in acid solutions

only. Its toxic action is governed by the condition of the fungus. A. G. POLLARD.

**Arsenical and other fruit injuries of apples resulting from washing operations.** D. F. FISHER and E. L. REEVES (U.S. Dept. Agric. Tech. Bull., 1931, No. 245, 12 pp.).—Calyx burn of apples caused by sol. As may occur on the tree or in picked apples which have become wet, but is most common in washed fruit where HCl or alkali solvents are used. Direct injury by HCl or alkalis is differentiated from injury by As. The latter is minimised in the washing process by the use of adequate quantities of rinsing water or by the addition of CaO during rinsing. A. G. POLLARD.

**New sulphur dusts for apple scab control.** A. I. PIERSTORFF and H. C. YOUNG (Phytopath., 1931, 21, 131).—The failure of many S dusts is attributed to uneven adherence and low toxicity. Improved dusts contain 85 pts. of S with either 15 pts. of dry lime-S or with 10 pts. of Manganar and 5 pts. of  $\text{Al}(\text{OH})_3$ . A. G. POLLARD.

**Use of disinfectants in fertilisers for the control of potato scab and *Rhizoctinia*.** W. H. MARTIN (Phytopath., 1931, 21, 104).—Results of the application of various  $\text{Hg}$  preps. in conjunction with fertilisers were satisfactory. A. G. POLLARD.

**Penetration and toxicities of petroleum oil sprays.** P. A. YOUNG (Phytopath., 1931, 21, 130).—Apple and potato leaves partly soaked with oils having <6% of sulphonatable residue lived for 30—80 days. Oils having >13% of such residue killed the leaves in 7—14 days and were toxic if applied to the leaves as 4—8% emulsions. The penetration of oil within the plant tissues is examined. A. G. POLLARD.

**Control of San José scale with lubricating oil emulsions in the Pacific Northwest.** E. J. NEWCOMER and M. A. YOTHERS (U.S. Dept. Agric. Circ., 1931, No. 175, 12 pp.).—The use of 4% oil emulsions is recommended. Grades of oil having a sulphonation val. of 50—70% may safely be used. Addition of casein to the standard caseinate emulsion does not increase its efficiency. Admixture with dil. CaO-S produces marked improvement, but the mixture is unsafe on any but dormant wood. Coconut fatty acids increase the toxicity of oil emulsions. A. G. POLLARD.

**Diagnostic value of plant symptoms in determining nutrient deficiencies of soils.** J. P. JONES (J. Amer. Soc. Agron., 1931, 23, 352—356).

**Melassigenic nitrogen in sugar beet after various fertilisers and on different soils.** P. A. VLASSJUK (Arb. Versuchs-stat. Uman, 1930, 12, 22; Bied. Zentr., 1931, [ii], 1A, 379).

**Phosphate rock. Silico-superphosphate.**—See VII. **Soil-corrosion of metals etc.**—See X.

#### PATENTS.

**Production of fertilisers.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 357,461, 16.1.31. U.S., 17.1.30).—A mixture of 33—55% of urea and 60—30% of  $\text{NH}_4$  carbamate, prepared together with a little  $\text{H}_2\text{O}$  by the interaction of  $\text{CO}_2$  and  $\text{NH}_3$  under raised temp. and pressure, is mixed with a solid superphosphate in

proportion such that there is not less than 0.5 mol. of  $\text{NH}_4$  carbamate per mol. of  $\text{P}_2\text{O}_5$ . L. A. COLES.

**Manufacture of fertilisers.** SOC. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTRI., ASSEES. of E. VOITURON (B.P. 356,624, 16.1.31. Ger., 6.2.30. Addn., to B.P. 342,931; B., 1931, 509).—The mother-liquor from the crystallisation of  $\text{NaNH}_2\text{HPO}_4 \cdot 4\text{H}_2\text{O}$  is acidified with about 15% of the total  $\text{H}_3\text{PO}_4$  used in the cycle and evaporated for the recovery of  $\text{KCl}$  and  $\text{NH}_4\text{Cl}$ , the mother-liquor being returned to the phosphate stage of the process, in which it is treated with sylvinit, more  $\text{H}_3\text{PO}_4$ , and  $\text{NH}_3$  to yield microcosmic salt.

A. R. POWELL.

**Fertiliser material.** H. J. KRASE, H. C. HETHERINGTON, and L. A. PINCK (U.S.P. 1,797,095, 17.3.31. Appl., 2.3.26).— $\text{NH}_3$ ,  $\text{CO}_2$ , and  $\text{H}_2\text{O}$  are heated in an autoclave until 40% of the  $\text{NH}_3$  is converted into urea and the solution is then treated with  $\text{H}_3\text{PO}_4$  or  $\text{HNO}_3$  until the remaining 60% of  $\text{NH}_3$  is converted into  $\text{NH}_4\text{H}_2\text{PO}_4$  or  $\text{NH}_4\text{NO}_3$ , respectively. On evaporation a crystal meal with a high N content is obtained.

A. R. POWELL.

**Preparation, and treatment, of phosphatic fertilising materials.** W. W. TRIGGS. From G. OBER & SONS CO. (B.P. 375,508—9, 7.4.30).—(A) Predetermined proportions of hot  $\text{H}_2\text{SO}_4$  and aerated crude phosphate dust are passed under pressure through a mixing device into a rotating autoclave provided with a jacket for regulation of temp., with means for withdrawing and conveying to a condenser gases liberated during the process, and for discharging the contents when the reaction is complete. (B) Nitrogenous material or K compounds may be added to the crude phosphate dust used in (A), and aq.  $\text{NH}_3$  is added to the reaction products in the autoclave after the mixture has been cooled and the pressure reduced to <1 atm.

L. A. COLES.

**Production of a fertiliser mixture.** KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 357,473, 17.2.31. Ger., 21.2.30).—Crude phosphate is decomposed with  $\text{HNO}_3$  (e.g., 50%) in the presence of  $\text{K}_2\text{SO}_4$ , and the product is treated with gaseous  $\text{NH}_3$  as long as any  $\text{H}_2\text{O}$  escapes; a dry, granulated product stable in air is obtained.

L. A. COLES.

**Insecticide and fungicide.** F. W. SULLIVAN, JUN., and E. W. ADAMS, ASSES. to STANDARD OIL CO. (U.S.P. 1,800,114, 7.4.31. Appl., 13.9.26).—The material comprises a thick emulsion of 5% of liquid glue, 8% of Cu rosin soap, 9% of  $\text{Pb}_3\text{As}_2\text{O}_8$ , 1.5% of the Na salt of a mineral oil sulphonic acid, 43% of white oil, and 33.5% of  $\text{H}_2\text{O}$ .

A. R. POWELL.

[Fertiliser from] greensand.—See VII. Insecticide.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Manufacture of sugar from dried beets.** J. B. MINTZ, A. K. KARATSHOV, B. E. KRASILSHCHIKOV, and P. K. GROSHEV (Nauk. Zapiski Tzuk. Prom., 1931, 11, 187—191).—A temp. above  $75^\circ$  at the diffusion battery increases the solubility of pectin substances, and the acidity and invert sugar in diffusion juice; a lower temp. increases losses of sugar in pulp and  $\text{H}_2\text{O}$ . The  $p_{\text{H}}$  of the juice is about 5.0; when  $\text{CaO}$  is added the colour

markedly increases. The invert sugar in diffusion juice is about 0.5—0.6% of the wt. of juice. The temp. of defecation should not be above  $85^\circ$ . Addition of 3% of  $\text{CaO}$  decreases the colour. Sulphitation improves the quality of syrups and massecuites.

CHEMICAL ABSTRACTS.

**Chains in the cells of the diffusion battery [for sugar].** V. S. VUIROVUI and A. I. SHAPIRO (Sovet. Sakhar, 1929, 174—175).—Fe chains (diam. 15—22 mm.) decrease the loss of sugar 0.07—0.08%. The yield of molasses is decreased.

CHEMICAL ABSTRACTS.

**Purification of sugar juices.** J. ZAMARON (Bull. Assoc. Chim. Sucr., 1931, 48, 277—287).—On the basis of laboratory tests on beet and cane juices, it is proposed to treat raw juice with 0.25—0.3% of  $\text{CaO}$  and 1% of  $\text{Al}(\text{OH})_3$  paste containing 94%  $\text{H}_2\text{O}$ , and then to heat to  $83$ — $85^\circ$  and filter. The filtered juice is treated with 0.4—0.5% of  $\text{CaO}$  and 0.1—0.15% of  $\text{Ba}(\text{OH})_2$ , carbonated once, filtered, sulphited, filtered, and evaporated. The  $\text{Al}(\text{OH})_3$  is prepared by precipitation of alum solutions with aq.  $\text{NH}_3$  and washed in filter-presses. The  $\text{Ba}(\text{OH})_2$  is added to the juice to remove sulphates derived from the alumina paste. With beet juices remarkable purification is claimed, besides a saving of 2/3 of the amount of  $\text{CaO}$  usually employed.

J. H. LANE.

**Isoelectric point in the Teatini process [of beet-juice purification] and modern physico-chemical theories.** D. TEATINI (Bull. Assoc. Chim. Sucr., 1931, 48, 166—182, 241—261).—Mainly an exposition of electrochemical theory leading up to the properties of amphoteric electrolytes and the nature of the isoelectric point. Treatment of raw beet juice at  $80^\circ$  with 2.5—3 g. of  $\text{CaO}$  per litre raises the  $p_{\text{H}}$  val. from 6.4 to about 10.6, which is the isoelectric point of the precipitable colloids, and larger amounts of  $\text{CaO}$  produce practically no further displacement of the  $p_{\text{H}}$ . The precise amount of  $\text{CaO}$  required to attain the isoelectric point may vary somewhat in different cases, depending on the quality of the  $\text{CaO}$  and the beets, but mainly on the temp. conditions during diffusion. In the Teatini process, 2.5 g. of  $\text{CaO}$  per litre may be regarded as a min., but a small excess is better than a deficiency, and even 3.5 g. per litre would not displace the  $p_{\text{H}}$  appreciably beyond the isoelectric point, especially as the  $\text{SO}_2$  added subsequently combines with some of the  $\text{CaO}$ . (Cf. B., 1931, 736.)

J. H. LANE.

**Carbonatation [of sugar-beet juice] under pressure, and economy in lime.** G. DURET (Bull. Assoc. Chim. Sucr., 1931, 48, 183—188).—Continuous carbonatation under pressure has given good results in large-scale plant throughout two campaigns. Limed juice and gas were delivered separately, but continuously, under pressure to the bottom of a closed carbonatation vessel, and from the upper part of this the partly carbonated juice passed to the bottom of a second carbonatation vessel to which also the excess of gas from the first vessel was led. The second vessel was open, but the column of juice in it, 4.5 m. high, produced a back-pressure of 0.5 kg./sq. cm. in the first. The best results were obtained when the gas entered the first vessel at 1 kg./sq. cm. No froth was produced in the first vessel. The ppt. formed deposited very rapidly, and gave filter-press

cakes which could be easily sweetened off. In the second campaign the amount of CaO employed was reduced from 2.3 to 1.5% for the first carbonatation and from 0.3 to 0.1% for the second, without any disadvantage. Automatic apparatus was used for liming the juice.

J. H. LANE.

**Sulphi-hydrosulphitation process [for sugar-beet juices].** R. MESTRE and J. DUTILLOY (Bull. Assoc. Chim. Suer., 1931, 48, 262—268).—It is proposed to treat raw diffusion juice with a colloidal hyposulphite, "Redoid ZN,"  $(\text{NH}_4)_2\text{S}_2\text{O}_4 \cdot (\text{CH}_2\text{O})_2 \cdot 3\text{ZnS}_2\text{O}_4 \cdot (\text{CH}_2\text{O})_2$ , at the rate of 50 g. per ton of beets, followed almost immediately by 50 g. of  $\text{SO}_2$  per ton. Liming and double carbonatation follow as usual, except that only 1.2—1.4 kg. of CaO per hectolitre are employed before the first carbonatation and none before the second. Improved purification (cf. B., 1927, 711) as well as saving in CaO are claimed.

J. H. LANE.

**A degree Brix-total solid relationship [for cane molasses].** R. H. KING (Ind. Eng. Chem. [Anal.], 1931, 3, 230—232).—Data for 1870 samples of [Philippine] molasses from 31 manufacturing units showed, for average group values, an approx. proportionality between the degrees Brix, taken at 6-fold dilution and multiplied by 6, and the difference between the degrees Brix and the true solid content determined by drying. This difference ("theoretical ash") was identical with the actual carbonate ash at very low and very high vals. (8% and 14%), but at intermediate vals. it exceeded the carbonate ash by amounts up to 1.4%; it was proportional to the logarithm of the carbonate ash. The possibility of applying these relations to the calculation of the true solid contents of molasses from the degrees Brix and the ash content is suggested.

J. H. LANE.

**Injection of sugar powder into vacuum pan for spontaneous crystallisation.** A. VOITZEKHOVSKI (Sovet. Sakhar, 1929, 84—85).—Sugar powder (1—2 g. for 92—95° purity, 3—5 g. for 89—92° purity) is preferably injected when the massecuite is at 90—95° and a thread 5—6 cm. long does not break. CHEMICAL ABSTRACTS.

**Bleaching of the massecuite of the second crystallisation (skip) with greens and molasses.** DERT. STAND. SUGAR TRUST (RUSSIA) (Sovet. Sakhar, 1929, 223—224).—Washing with diluted molasses (54° Brix at 50°) is preferred. CHEMICAL ABSTRACTS.

**Speed of crystallisation of lactose, galactose, glucose [dextrose], and sucrose from pure solution.** E. O. WHITTIER and S. P. GOULD (Ind. Eng. Chem., 1931, 23, 670—673).—Conc. solutions of these 4 sugars, prepared at boiling temp., were cooled and maintained, with continuous stirring, at various const. temp. between 0° and 30°, and the rates of crystallisation were determined refractometrically. With the first three sugars the rate of crystallisation is at first determined mainly by the initial supersaturation of the separating form, but later by the rate at which this form is produced from its more sol. isomeride by mutarotation. In the case of lactose, and to a smaller extent in that of galactose, mutarotation is so retarded by fall in temp. that crystallisation is slower at 20° than at 30° in spite of the greater solubility of the sugars at the higher temp. The most rapid crystallisation of lactose takes place if the solution is

maintained at or slightly above 30° for 3 hr. and is then allowed to cool to about 20°.

J. H. LANE.

**Effect of rate of boiling on the residual sulphur dioxide content in mixtures of sugar and corn syrup; also effect of bleaches containing sulphur dioxide.** R. H. MORGAN (Analyst, 1931, 56, 638—646; cf. B., 1930, 962).—Additional time taken over the boiling process reduces the amount of residual  $\text{SO}_2$  to a const., which is soon reached. The residual  $\text{SO}_2$  contents in boilings from different samples of corn syrup bear no relation to each other, taking into account the initial  $\text{SO}_2$  present, and are probably influenced considerably by viscosity. The addition of bleaches, even in small amounts, considerably raises the residual  $\text{SO}_2$  content of boilings, as compared with controls to which no bleach has been added, but no definite relationship has been established.

T. McLACHLAN.

**Determination of dextrose and laevulose in honey by use of the iodine-oxidation method.** R. E. LOTHROP and R. L. HOLMES (Ind. Eng. Chem. [Anal.], 1931, 3, 334—339).—The methods of Auerbach and Bodländer (B., 1924, 107, 567) and of Slater and Acree (A., 1930, 1165) for the determination of dextrose are unsatisfactory, but that of Hinton and Macara (B., 1924, 189) gives accurate results. Mannose can be accurately determined under similar conditions, but only if the alkali is added slowly, e.g., over a period of 2 min., so as to minimise the formation of iodate. The iodine absorption of laevulose varies with temp. and duration of action, and is probably the result of a slow Lobry de Bruyn transformation into aldoses. For the analyses of honey 20 c.c. of a solution containing 0.2 g. of honey should be treated with 40 c.c. of 0.05N-I solution and 25 c.c. of 0.1N-NaOH, allowed to remain stoppered for 10 min. at 20°, and then treated with 5 c.c. of 2N- $\text{H}_2\text{SO}_4$  and titrated at once with 0.05N- $\text{Na}_2\text{S}_2\text{O}_3$ . 1 c.c. of 0.05N-I solution absorbed  $\equiv$  4.502 mg. of dextrose, but the % dextrose content of the honey calc. on this basis must be corrected by subtracting 0.012 times the approx. laevulose content. The exact laevulose content is found by subtracting the corrected dextrose content from the total reducing sugar content (calc. as dextrose) and dividing the difference by 0.925; vals. thus obtained are usually 1—2% higher than those determined by polarisation at low and high temp. The ratio of laevulose to dextrose in 33 samples of fresh American honeys from different predominant floral sources was found to range from 1.02 to 1.70.

J. H. LANE.

**Determination of sugar losses in water from the barometric condenser.** AFANASENKO (Sovet. Sakhar, 1929, 176).—The  $\text{H}_2\text{O}$  is treated with  $\alpha$ -naphthol and  $\text{H}_2\text{SO}_4$  and the colour compared with standards prepared from 0.001—0.01% of sugar; if the quantity is above 0.1% the  $\text{H}_2\text{O}$  is polarised. Sampling apparatus is described. CHEMICAL ABSTRACTS.

**Impurities in white sugars. II. Determination of sulphates, sulphites, and aldehyde-sulphites.** J. A. AMBLER, J. B. SNIDER, and S. BYALL. **III. Determination of labile organic sulphur.** J. A. AMBLER (Ind. Eng. Chem. [Anal.], 1931, 3, 339—340, 341—343; cf. B., 1931, 648).—II. Sulphates in white sugars can



be accurately determined by direct precipitation of acidified solutions with  $\text{BaCl}_2$ .  $\text{SO}_2$  in 20 factory white beet sugars ranged from 0 to 1188 p.p.m., and in 4 factory white cane sugars from 19 to 295 p.p.m., whilst 5 refined sugars contained none. Iodometric titration of solutions of factory white sugars indicated amounts of  $\text{SO}_2$  up to 56 p.p.m. in inorg. combination, and amounts up to 6 p.p.m. combined with aldehydes and liberated by pretreatment with  $\text{KOH}$ . Apparent  $\text{SO}_2$  contents of 0.4–0.6 p.p.m. in refined sugars were attributed to traces of reducing polyphenols.

III. On boiling solutions of white sugars with alkaline plumbite, minute quantities of  $\text{PbS}$  are formed, due to compounds, such as cystine, containing labile S. A spectrophotometric method of determining such S, based on the formation of methylen-blue, is described. Results, calc. as cystine, for 14 factory white and refined sugars ranged from 3.4 p.p.m. downwards to traces.

J. H. LANE.

**Turbidity in sugar products. I. Relation between intensity of Tyndall beam and depth and concentration of solution.** F. W. ZERBAN and L. SATTLER (Ind. Eng. Chem. [Anal.], 1931, 3, 326–330).—The Zeiss-Pulfrich spectrophotometer, which can be used for measuring the Tyndall-beam intensities of turbid liquids as well as transmittancies, was employed in the present study. With coloured turbid liquids, the Tyndall-beam intensity cannot be satisfactorily measured without a correction for colour absorption. Using a raw sugar solution of 60° Brix diluted with pure sucrose solutions of the same density, the ratio,  $R$ , between the Tyndall-beam intensity and the transmittancy could be related to the depth,  $b$ , of the layer of solution at const. concentration, or to the concentration,  $c$ , at const. depth, by formulæ of the type  $R = R_1 b^n$  or  $R_1 c^n$ , which are being further studied.

J. H. LANE.

**Influence of size of granules on the value of starch.** E. SZÉGGÖ (Bull. Assoc. Chim. Sucr., 1931, 48, 268–274).—Potato starch of first quality commonly contains 1.5–6 wt.-% of granules <20  $\mu$  in diam. and 43–44% of granules of >40  $\mu$  in diam., and Sprockhoff has shown that the small granules yield pastes of higher viscosity and lower acidity than pastes from the larger ones (B., 1930, 436; 1931, 648). Deterioration in the gelatinising power of the small granules in after-product starch, due to prolonged factory treatment, could be minimised by use of centrifugal methods of deposition.

J. H. LANE.

**Starch gels.** S. WOODRUFF and L. NICOLI (Cereal Chem., 1931, 8, 243–251).—Starch- $\text{H}_2\text{O}$  pastes containing 5% of maize, wheat, rice, potato, arrowroot, or cassava starches require to be heated to temp. above those at which sudden change in translucency occurs, in order to produce gels strong enough to retain the shape of a mould on cooling. Max. strength is obtained by heating to 90° or above. Cereal starches give much stronger gels than root starches. Addition of sucrose reduces the strength in a greater degree than dilution with  $\text{H}_2\text{O}$  to the same vol. Addition of 60 wt.-% of sucrose to a 5% starch- $\text{H}_2\text{O}$  paste completely prevents gelation in all cases, but 50% added to the cereal starch pastes did not prevent the formation of well-formed gels.

W. J. BOXD.

**Pulp dryers.**—See I. Report [on sugar cane].  
**Manuring of sugar beet.**—See XVI. Yeast from molasses.—See XVIII. Sugar in apples. Yeasts in fermenting honey.—See XIX.

PATENT.

**Higher fatty acid esters.**—See V.

## XVIII.—FERMENTATION INDUSTRIES.

**Preservative principles of hops. XI. Effects produced by certain constituents of hop resin during the boiling of hopped worts.** T. K. WALKER and J. J. H. HASTINGS. XII. Quantitative comparison of very small degrees of antiseptic activity and of very small differences between more pronounced degrees of activity. T. K. WALKER, J. J. H. HASTINGS, and E. J. FARRAR (J. Inst. Brew., 1931, 37, 509–512, 512–533; cf. B., 1929, 533).—XI. The bitterness which the  $\alpha$ -resin imparts to the boiling wort increases to a max. and is greater in amount than that yielded by the  $\beta$ -resin. The time required to reach the max. depends on the concentration and physical condition of the resin, and on the rate and temp. of boiling. On prolonged boiling both  $\alpha$ - and  $\beta$ -resins give rise to unpleasant flavours. The  $\alpha$ -resin produces no aroma in the wort, whereas a pleasant aroma is imparted by the  $\beta$ -resin.

XII. The method is based on a given dose of antiseptic causing the max. restriction of acid formation if it is added immediately after the commencement of the logarithmic phase of growth of *B. bulgaricus* seeded into sterile wort. Under these conditions the ratio of the % restriction of acid formation to % concentration of antiseptic is const. The culture medium used throughout is sterile malt wort of  $d$  1.05 and  $p_H$  5.0, and the temp. of incubation is 30°. 10 c.c. of wort are inoculated by two loops of a 24-hr. old culture of *B. bulgaricus* and incubated for 18 hr. 1 c.c. of this culture is added to 99 c.c. of wort at 30°, and the whole (100 c.c.), which contains  $2 \times 10^6$  organisms per c.c., is the standard inoculum for the flasks of wort on which the actual determination is conducted. Flasks containing 200 c.c. of wort at 30° are incubated for 6 hr. with 1 c.c. of the standard inoculum. The addition of 1 c.c. of a 0.5% hop decoction is then made and, after incubation for 17 hr., 50 or 100 c.c. are withdrawn and quinhydrone is added to prevent the further production of acid. The samples are then electrometrically titrated at 20° to the initial  $p_H$  of the wort, using 0.1N-NaOH. The retardation of acid formation by the antiseptic is determined by comparison with a control flask in which no hop decoction is added. The % retardation is a measure of the relative antiseptic vals. of the hops. The results show the same order of grading of the hops when compared with those obtained by the methods of Chapman and of Ford and Tait.

C. RANKEN.

**Determination of dry matter (or moisture) in pressed yeast.** L. FLETCHER (J. Inst. Brew., 1931, 37, 506–508).—In the standard method the weighed, unwashed yeast is moistened with abs. EtOH and dried for 7–9 hr. in a water-oven in a current of air. A content of  $\text{H}_2\text{O}$  which is 0.5–1% higher is obtained

if the yeast is first extracted by abs. EtOH and the dehydrated yeast dried in the water-oven together with the alcoholic extract. The combined solids from the yeast and the extract permit the calculation of the  $H_2O$ . If the yeast is dried at  $105^\circ$  in a vac., except for a minute current of air, and the  $H_2O$  is absorbed by  $H_2SO_4$ , the content of  $H_2O$  is approx. 2.5% higher than that obtained by the standard method. C. RANKEN.

**Yields of yeast from molasses.** G. MEZZADROLI and P. VEREMEENCO (Atti III Cong. Naz. Chim. pura appl., 1929, 576—579; Chem. Zentr., 1931, i, 2813).—Beet molasses is a better nutrient than cane-sugar molasses. The yield was increased by aëration, a large impregnation, addition of phosphate, slight acidity, presence of N, and suitable dilution, and was decreased by addition of EtOH or active C. A. A. ELDRIDGE.

**Fermentation of polyhydric alcohols by the colon-aërogenes group of bacteria.** C. F. POE and J. T. FIELD (J. Bact., 1931, 21, 11).—In media containing inositol, *Escherichia* cultures produced no acid, but *Aerobacter* gave a distinct acid reaction. Sorbitol yielded acid in all cultures. In every case where acid and gas were obtained the products included  $CO_2$ ,  $H_2$ , AcOH,  $HCO_2H$ , succinic and lactic acids.

A. G. POLLARD.

**Determination of ethyl and butyl alcohols in fermentation mixtures.** C. H. WERKMAN and O. L. OSBURN (J. Bact., 1931, 21, 20—21).—The liquor is neutralised and distilled and any  $MeCHO$ ,  $COMe_2$ , etc. are removed by precipitation with 2:4-dinitrophenylhydrazine. From the residual liquid the alcohols are distilled and oxidised by means of  $K_2Cr_2O_7$  and  $H_3PO_4$  and the acids distilled off. A portion of the distillate is titrated to determine the total acidity, and in another portion the ratio of AcOH and BuOH is ascertained by partitioning in  $Pr^i_2O$ . A. G. POLLARD.

**Adsorption in beer filters.** J. DE CLERCK (Bull. de l'école sup. de Brass., Louvain, 1931, 31, 67; Woch. Brau., 1931, 48, 409—414, 423—426, 429—434).—During washing, pulp adsorbs alkalis which are removed by the beer during filtration. The earlier portions of the beer are thus partly neutralised and the  $pH$  is raised. Extractives are removed from the beer first filtered, principally colouring matters and those colloids which affect surface tension and thus foaming capacity. The loss of these properties may amount to 25% on the first beer going into trade. Viscosity and total N are altered to a smaller extent. New pulp forms a less dense mass than pulp which has been in use for some months and of which the fibres are partly disintegrated. Less pressure is required to filter through the former and it has a much lower adsorptive power, but soon allows fine turbidity to pass through. The first-filtered beer is inferior in biological stability to that filtered later. F. E. DAY.

**Examination of two samples of old bottled beer.** A. C. CHAPMAN (J. Inst. Brew., 1931, 37, 540—541).—Compared with the same beer kept in bottle for 16 years and examined 34 years ago, a second sample kept in bottle for 51 years, which had remained quite sound, showed a marked increase in the content of esters,

whilst the volatile acidity had almost disappeared. The content of furfuraldehyde remained const., but the amount of EtOH had slightly decreased. The yeast cells in the deposits were shrunk, but they developed with a vigorous fermentation when inoculated into sterile wort, and at the end of 2 months some cells showed structures which resembled spores. A similar deposit from a sample of "King's ale" bottled for 29 years yielded no growth when seeded into sterile wort. C. RANKEN.

**Chemical composition of various types of [German] beer.** W. WINDISCH, P. KOLBACH, and E. SCHILD (Woch. Brau., 1931, 48, 415—418, 427—429).—Analytical results are given for 58 varieties.

F. E. DAY.

**Have electric currents action on fermentation?** F. STOCKHAUSEN and R. KOCH (Woch. Brau., 1931, 48, 403—407, 419—423).—With d.c. applied during the whole fermentation harmful effects were found to begin with 0.1—0.6 milliamp./sq. cm. (0.05—0.3 milliamp./sq. cm. anode intensity), using Pt electrodes. The effect is probably on the wort rather than directly on the yeast. A.c. of 0.5 milliamp./sq. cm. is not harmful. Damage to fermentations by stray currents of the order of intensity met with in practice can occur only if Cu is brought into solution. F. E. DAY.

**Enzymic clarification of grape juice.** J. J. WILLAMAN and Z. I. KERTESZ (New York Agr. Expt. Sta., Tech. Bull., 1931, No. 178, 15 pp.).—The turbidity which develops in grape juice during storage and is not filterable has been traced to the presence of pectic substances. An enzyme which is capable of converting the pectin into sol. substances and a coagulable material has been found in cultures of *Penicillium glaucum*, Link, on a synthetic medium containing sucrose. The enzyme solution may be satisfactorily conc. by evaporation in vac.; the optimum acidity is  $pH$  3.0—3.6, whilst the optimum temp. is  $38$ — $40^\circ$ , the solution being completely inactivated at  $55^\circ$ . The enzyme may be added at any stage of the commercial prep., but if the addition is subsequent to the removal of tartrate reheating to  $38^\circ$  is necessary. The coagulum carries down suspended matter so that if commercial pectin be added, the process can be used to clarify other juices which do not contain pectin.

H. J. DOWDEN.

**Chemistry of grape juice.** E. L. GREEN and Z. I. KERTESZ (New York Agric. Expt. Sta., Tech. Bull., 1931, No. 181, 14 pp.).—Enzymic clarification of grape juice by the method of Willaman and Kertesz (cf. preceding abstract) removes about two thirds of the pectin; the ppt. formed is shown to consist of carbohydrate material. On keeping the juice the pectin and tartaric acid decrease without necessarily clearing the juice. E. B. HUGHES.

**Fluorescent indicators for the determination of the acidity of coloured wines.** J. M. GALLART (Anal. Fís. Quím., 1931, 29, 490—493). J. M. CLAVERA (*Ibid.*, 494—496).—A criticism of Volmar and Clavera's method (B., 1931, 693), and a reply. H. F. GILLBE.

**Yeasts in fermenting honey.**—See XIX.

## PATENTS.

**Manufacture of yeast by the aeration process.**

H. J. and H. C. JANSEN (B.P. 357,541, 19.6.30).—Yeast with either improved reproductive or increased fermentative power is obtained by withdrawing intermittently or continuously part of the fermenting liquid, which is deprived of its suspended yeast and returned to the fermentation vat, so that the yeast content of the fermenting liquid is maintained at a predetermined value.

C. RANKEN.

**Clarifying or separating the [yeast] head from fermenting liquor, in particular in brewing.** A. HALLERMANN (B.P. 357,458, 13.1.31).—A perforated metal sheet which allows the passage of the rising head is fixed closely above and is distributed over the entire surface of the fermenting liquid, whereby the solid constituents are retained on the subsequent fall of the head.

C. RANKEN.

**Manufacture of beer [containing vitamins].**

H. VAN DE SANDT (B.P. 357,732, 20.8.30. Ger., 13.9.29).—Vitamin preps. obtained by autolysis of yeast or malt germs at approx. 50° are added to the beer after fermentation.

C. RANKEN.

Esters of EtOH.—See III.

## XIX.—FOODS.

**Wheat and flour studies. XIX. Effect on their bread-making properties of extracting flours with ether, with special reference to the gas-retaining powers of doughs prepared from ether-extracted flours.** A. H. JOHNSON and W. O. WHITCOMB (Cereal Chem., 1931, 8, 392—402).—The improved baking quality of Et<sub>2</sub>O-extracted flours is due to their superior gas-retention in the dough. Addition of fat to doughs prepared from such flours reduced their gas-retaining powers, and allowing the dough to rise several times, with "punching" at the proper time, produced a similar result.

E. B. HUGHES.

**Bactericidal properties of ether, alcohol, chloroform, and carbon disulphide.** A. C. THAYSEN and L. H. WILLIAMS (Zentr. Bakt. Par., 1931, II, 84, 252—260).—Wollney's method (Zentr. Bakt., 1892, 11, 752) of sterilisation by immersion in Et<sub>2</sub>O is modified for use with flour. The sample is treated with Et<sub>2</sub>O for 48 hr. to kill non-sporing organisms. Remaining spores are encouraged to germinate (e.g., by storage in a moist atm. at 30°) and the Et<sub>2</sub>O treatment is repeated for a short period. Changes in the condition of the protein of the flour are small. CS<sub>2</sub> and CHCl<sub>3</sub> may be used on a similar manner, but results are less satisfactory.

A. G. POLLARD.

**Evaluating new wheat varieties by use of the baking test.** C. E. MANGELS and T. E. STOA (Cereal Chem., 1931, 8, 381—391).—Variation in loaf vol. with change in fermentation period and loaf shape are the most important characteristics in judging wheat varieties by baking tests.

E. B. HUGHES.

**Utility of the Tag-Heppenstall moisture-meter for determining the moisture content of ground wheat.** W. F. GEDDES and C. A. WINKLER (Cereal Chem., 1931, 8, 409—414).—H<sub>2</sub>O in ground wheat is determined by measuring the electrical conductivity

of a stream of wheat passing between two revolving rollers, which serve as electrodes. The method of using the meter and its applications are outlined.

E. B. HUGHES.

**Moisture determinations in wheat with special reference to quick methods.** D. A. COLEMAN (Cereal Chem., 1931, 8, 315—335).—Comparative tests have been carried out on various rapid moisture-testing devices some of which operate on the dielectric, others on the conductivity, principle.

W. J. BOYD.

**Protein and moisture determinations in wheat with special reference to conditions covering preparation of samples for analysis.** J. T. FLOHIL (Cereal Chem., 1931, 8, 307—312).—Procedure in sampling wheat and preparing the sample for analysis is discussed in relation to errors in protein and moisture determinations.

W. J. BOYD.

**Ashing methods [in determining ash in flour].** W. C. MEYER (Cereal Chem., 1931, 8, 312—315).—The direct and indirect methods (cf. B., 1930, 584, 836), with and without the use of O<sub>2</sub>, have been compared. The accuracy of the direct method is confirmed. When O<sub>2</sub> is used greater care is necessary, but accurate results can be obtained in approx. 2 hr.

W. J. BOYD.

**Supplementary procedure with the [A.A.C.C.] basic baking test for use with low diastatic flours.** M. C. MARKLEY and C. H. BAILEY (Cereal Chem., 1931, 8, 300—305).—In order to study accurately the factors influencing the baking quality of flour which are carried in the genetic constitution of the wheat it is necessary to ensure constancy in chemical composition and the presence of sufficient diastase to allow normal fermentation. The addition of 3% of flour from sprouted wheat is a convenient method of supplying the required diastase.

W. J. BOYD.

**Report of activities [on experimental baking test].** P. P. MERRITT and M. J. BLISH (Cereal Chem., 1931, 8, 267—292; cf. B., 1931, 461).—The factors responsible for variations in the results of the standard baking test obtained by different operators have been studied. Machine mixing tends to produce larger loaves than hand mixing unless a restricted mixing time is used. Prolonged machine mixing gives greater variability than hand mixing. It is not possible to establish time factors for different types of machine so as to give identical results, as these factors vary for different flours. With correct adjustment of sheeting rolls and compression plate, especially the latter, machine moulding gives results closely approximating to those obtained by hand moulding, with reduced variability. Various types of oven have been studied. When loaves are inserted singly or in pairs, forced draught and introduction of an open pan of water into the oven have little effect on loaf quality. When several loaves are introduced at a time a set of dummy loaves should be baked first and a pan of water should be placed in the oven. Lowering the baking temp. by 10° affected crust colour only.

W. J. BOYD.

**Separation of "gassing power" (diastatic activity) from "strength" in baking tests.** H. JORGENSEN (Cereal Chem., 1931, 8, 361—374).—In bread-baking tests, the proportions of the ingredients

should be so adjusted that there is an excess of fermentable sugar throughout fermentation and proofing, in order to separate "gassing power" from "strength" of flour. E. B. HUGHES.

**Relation of quality of dry skim milk to baking strength.** O. SKOVHOLT and C. H. BAILEY (Cereal Chem., 1931, 8, 374—380).—The quality of dried milk for breadmaking is improved by preheating to above 77°. Attempts to determine the cause of improvement were not successful. E. B. HUGHES.

**Chemical leavening agents and their characteristic action in doughs.** R. A. BARACKMAN (Cereal Chem., 1931, 8, 423—432).—An apparatus and procedure are described for following the reaction of chemical leavening agents during mixing and keeping of mixes used for foodstuffs. E. B. HUGHES.

**Composition of bread.** C. B. MORISON (Cereal Chem., 1931, 8, 415—417).—Analyses of modern American bread show higher protein, fat, and ash content than figures given in text books. E. B. HUGHES.

**Cooking of cereal porridges.** N. T. CUNNINGHAM (Cereal Chem., 1931, 8, 403—408).—The % of gelatinisation as a measure of the cooking of cereal porridge is determined by the blue colour produced with I, a fully gelatinised starch solution being employed as a standard. E. B. HUGHES.

**Soya-bean cake as a food. I. Oil-extracting process and digestion coefficient of the protein.** S. IZUME and Y. YOSHIMARU. **II. Nutritive value of the alcohol-extracted oil cake.** S. IZUME, Y. YOSHIMARU, and I. KOMATSUBARA. **III. Effect of addition of the soya-bean oil cake to other grain.** S. IZUME and I. KOMATSUBARA (J. Agric. Chem. Soc., Japan, 1931, 7, 87—96).—I. Extraction with hot EtOH denatured the protein, the solubility of which fell from 88% to 24%; that of the benzene-extracted cake was 82% and of the pressed cake 45%. There is little change, however, in digestibility of the protein of various cakes.

**II.** No significant differences in nutritive val. of the proteins were observed. The vitamin-A and -D contents of the EtOH-extracted cake are low; the vitamin-B content is less than that of the fresh bean.

**III.** The incomplete nutritive val. of cereals is supplemented by addition of EtOH-extracted soya-bean cake.

CHEMICAL ABSTRACTS.

**Factors for converting percentages of nitrogen in foods and feeds into percentages of proteins.** D. B. JONES (U.S. Dept. Agric., 1931, Circ. No. 183, 21 pp.).—The factor 6.25 has been generally used, with exceptions, in calculating protein content from N content on the unwarranted assumptions that all proteins contain 16% N and that all the N in foodstuffs and feeds is protein-N. The following factors have been calc. from data accumulated by many different workers: wheat endosperm 5.70; wheat embryo 5.80; wheat bran 6.31; whole wheat, rye, barley, and oats 5.83; rice 5.95; maize 6.25; coconut, most oil seeds, hazel nut, and walnut 5.30; almonds 5.18; Brazil and pea nut 5.46; soya bean 5.71; gelatin 5.55; milk 6.38; eggs, meats, and leguminous seeds 6.25.

W. J. BOYD.

**Detection of ammonia in milk.** F. KOPATSCHEK (Milch. Zentr., 1931, 60, 309—310).—To 10 c.c. of the sample 30 c.c. of distilled H<sub>2</sub>O and 10 c.c. of 1.5% uranyl acetate solution are added and the mixture is shaken and filtered. 10 c.c. of the filtrate are shaken with 2 c.c. of 34% Rochelle salt solution and 1 c.c. of Nessler's reagent is added. The colour is observed within 1 min. as the solution soon becomes turbid. A golden-yellow, slightly orange-red tint is faintly positive, a pale yellow is negative, and an intense orange-red is strongly positive. A blank test is carried out using 10 c.c. of distilled H<sub>2</sub>O, 5—6 drops of uranyl acetate, 2 c.c. of Rochelle salt solution, and 1 c.c. of Nessler's reagent. A pale yellow colour should develop. A negative result of this test, by which 0.003% of NH<sub>3</sub> is detectable, indicates the absence of NH<sub>3</sub> due to bacterial or other contamination. W. J. BOYD.

**Effect of heat-treatment on the quality of dry skim milk and condensed skim milk for ice cream.** O. E. WILLIAMS and S. A. HALL (U.S. Dept. Agric. Circ., 1931, No. 179, 10 pp.).—Among dried skim milks, the best for ice-cream mixtures were samples prepared by spray-drying, in particular those heated at 83° before drying. Superheating of condensed milks improved their quality for ice-cream manufacture, but the product was still inferior to that obtained by spray-drying. A. G. POLLARD.

**Hydrogen-ion determination of butter.** O. F. HUNZIKER, W. A. CORDES, and B. H. NISSEN (J. Dairy Sci., 1931, 14, 347—372).—The quinhydrone electrode may be used for determining the  $p_H$  of butter. The curd-serum mixture must be used whole, since dilution produces marked changes in  $p_H$ . Removal of the curd from the serum causes slight lowering of the  $p_H$ .

A. G. POLLARD.

**Effect of manner of preparation and calcium content on the consistency of cheese.** G. WODE (Kungl. Landtbruks-Akad. Handl. Tidskr., 1930, 69, 1032—1043; Chem. Zentr., 1931, i, 2815—2816).—The higher is the Ca content of cheese the softer it is. During the prep. of cheese the Ca passes into the whey, the more readily the more acid is the milk. By rapid separation the loss of Ca is reduced. A. A. ELDRIDGE.

**Properties of some acid caseins.** R. W. BELL and S. P. GOULD (J. Dairy Sci., 1931, 14, 337—346).—Factors affecting the paper-coating properties of casein are examined. The reaction of casein is largely controlled by the conditions under which it is pressed. A well-washed sample has  $p_H$  4.6 and low free acid and ash contents. Best coating properties are associated with a min. of free acid. The keeping quality and strength of casein are reduced by the presence of milk fat. Casein prepared by precipitation with HCl at temp. < 35.5° forms a soft fine curd, difficult to drain and wash. The most satisfactory ppt. with H<sub>2</sub>SO<sub>4</sub> is obtained by the use of 1:30 acid. Deterioration of the dried product is not rapid if the moisture content is < 8%.

A. G. POLLARD.

**Factors affecting the quality of commercial zanning peas.** C. B. SAYRE, J. J. WILLAMAN, and, Z. I. KERTESZ (New York Agr. Expt. Sta., Tech. Bull. 1931, No. 176, 76 pp.).—A puncture and a crushing

test for tenderness in raw peas have been developed, the latter being capable of detecting peas which will be tender when canned. The changes which occur during maturation comprise decrease in sugars, increase in starch, protein, and dry matter, whilst CaO increases in the skins and is associated with increased hardness. Toughness and the CaO content of the skin also increase with lapse of time after shelling, and, after canning, the CaO content of the skins is very much greater than before canning. K fertilisers hasten maturity, KCl and  $K_2SO_4$  being equally satisfactory, and in addition, tend to reduce Ca intake and thus produce softer peas.  $CaCl_2$  definitely increases the hardness besides reducing the yield through bad germination. A quality index is proposed, based on the product of the tenderness of each size and the % of the various sizes of pea.

H. J. DOWDEN.

**Determination of sugar in apples.** MARILLER (Bull. Assoc. Chim. Sucr., 1931, 48, 160—162).—According to Warcollier, sucrose may represent from 5 to 42% of the total sugars in apples. During storage inversion occurs and also loss of sugar, especially of dextrose, the ratio of dextrose to laevulose never exceeding 0.5. Boinot found that the total sugar content, determined after inversion by the Clerget method, varied widely according to the method of defecating or preparing the extracts. Defecation with basic Pb acetate led to higher vals. than defecation with EtOH, or than alcoholic extraction or digestion, differences up to 24% of the total sugar content being obtained; but Pigcot has shown that the Clerget method does not effect complete inversion of sucrose in presence of EtOH.

J. H. LANE.

**Nomogram for use in connexion with Gutzeit arsenic determinations on apples.** W. J. YOUNG (Contr. Boyce Thompson Inst., 1931, 3, 363—373).—The probable error of the analytical process is discussed, and is probably less than the error of sampling. A nomogram is prepared for determining the precision of the sampling procedure.

A. G. POLLARD.

**Factors influencing the pectin content of stored apple pomace.** Z. I. KERTESZ and E. L. GREEN (New York Agr. Expt. Sta. Tech. Bull., 1931, No. 179, 14 pp.).—Samples of dried, ground pomace containing 7.5, 20, 33.3, 50, and 60%  $H_2O$  were stored in sealed jars for 5 months at room temp. The sol. pectin was determined by extraction with cold  $H_2O$  and that derivable from protopectin by repeated extraction with  $N/30-HCl$  at  $97^\circ$ .  $H_2O$ -sol. pectin increases with increasing  $H_2O$  in the pomace, reaching a max. at 33.3%  $H_2O$ . Acid-hydrolysable pectin suffers a corresponding decrease, and as this portion has the greater commercial val. the  $H_2O$  content of the pomace should be kept as low as possible during storage. With mould-inoculated samples no growth occurred below 20%  $H_2O$  content, but at 33.3 and 50%  $H_2O$  the total pectin extracted from the mould-infected samples was 14 and 28% lower, respectively, than that from samples kept sterile. H. J. DOWDEN.

**Effect of solid carbon dioxide on transportation diseases [in fruit].** C. BROOKS (Phytopath., 1931, 21, 103).—The  $CO_2$  content of the atm. of small storage containers or refrigerators is conveniently increased by the

use of solid  $CO_2$  to a degree which has the same inhibiting effect on the rotting and softening of warm fruit as a temp. fall of  $17-22^\circ$ . If the gas is allowed to escape within 18—24 hr. no objectionable flavour develops in the fruit except in a few cases. The inhibitory action of the gas ceases soon after it is liberated, but the interval of time permits the thorough cooling of the container or refrigerator.

A. G. POLLARD.

**Preservation of lemon juice.** B. MELIS (Boll. Uff. Staz. Sperim. Ind. Essenze, 1931, 6, 154—157).—The pulp of the lemons is washed to remove traces of essential oil; this may be done by means of a perforated travelling belt under a  $H_2O$  spray. Partial drying is then effected by fans at room temp. The pressed juice is conc. in a vac. until the acidity reaches 80%. Special enamelled containers are completely filled with the conc. liquid and  $H_2$  at a pressure of 1.5 atm. is admitted. The gas is absorbed by the colloidal particles; on releasing the pressure the impurities are carried upwards and the clear juice is discharged from the lower part of the apparatus. The juice is conveyed by rail in carboys in which the upper space has been filled with  $H_2$ . O. F. LUBATTI.

**Characteristics of yeasts found in fermenting honey.** G. E. MARVIN, W. H. PETERSON, E. B. FRED, and H. F. WILSON (J. Agric. Res., 1931, 43, 121—131).—During the granulation of honey the  $H_2O$  present remains in the syrup, which ultimately becomes sufficiently dil. to permit the growth of yeasts. The slow fermentation process produces about equal quantities of  $CO_2$  and EtOH, with small amounts of non-volatile acids. Fermentation is prevented by heating honey to  $71^\circ$  and cooling rapidly. Honey stored at temp. below  $11^\circ$  does not ferment.

A. G. POLLARD.

**Evaluation of potato flakes as fodder and as raw material for distilleries.** B. LAMPE (Z. Spiritusind., 1931, 54, 254—255).—The proportion of lumps in the flakes which do not fall away easily on stirring in  $H_2O$  should be small, as such aggregations have a greater moisture content and higher acidity than the loose flakes. The moisture content should be determined in both the loose and lumpy flakes and should not exceed 15%. Normal flakes are pale yellow, but they may be darker without loss of quality. The odour of the flakes when dry and also when stirred into  $H_2O$  at  $50-60^\circ$  should be aromatic, breadlike, and devoid of mustiness, and the steam from flakes boiling in  $H_2O$  should be neutral to litmus. The  $pH$  of the liquid obtained by stirring 10 g. of flakes in 150 c.c. of  $H_2O$  at room temp. for 15 min. should not exceed 6.2, and  $> 4$  c.c. of  $N-NaOH$  per 100 g. of flakes should be necessary to titrate the liquid to  $pH$  7.07 with neutral-red. If the flakes are for distillery use, the amount of EtOH yielded by the inversion method should be determined also.

C. RANKEN.

**Buckwheat middlings as a protein supplement for growing and fattening swine.** J. H. LONGWELL (Proc. Amer. Soc. Animal Production, 1928, 98—101).—Buckwheat middlings are superior to wheat middlings, but deficient in vitamins-A and -D. CHEMICAL ABSTRACTS.

**Starch equivalent of maize silage.** G. FINGERLING, P. EISENKOLBE, B. HIENTZSCH, M. JUST, and K. SCHMIDT (Landw. Versuchs-stat., 1931, 112, 243—260).—The

digestibility of the org. matter of maize silage averages 80%. The starch equiv. is definitely higher than that allocated by Kellner. A. G. POLLARD.

**Determination of sand in vegetable feeding-stuffs.** W. LEPPER (Chem.-Ztg., 1931, 55, 782—783; cf. B., 1930, 1129).—A reply to Rub's criticisms (B., 1931, 564). In the latter method it is considered that org. material may be carried down with the sand, and sand may be retained by the material floating in the  $\text{CCl}_4$ . W. J. BOYD.

**Rancidity accelerated by porous packings.** W. RABAK (Oil and Fat Ind., 1931, 8, 373—374).—Card-board packings of cakes etc. absorb fat which becomes rancid. Hard-finish non-porous boards (e.g., silicated and waxed) develop least rancidity; the residual  $\text{H}_2\text{O}$ -sol. chemicals in the board appear to be without influence. E. LEWKOWITSCH.

**Preservation of fish nets.**—See V. **Analysis of butter.**—See XII. **[Potato storage].**—See XVI. **Determining sugars in honey.** **Starch gels.**—See XVII. **Grape juice.**—See XVIII.

#### PATENTS.

**Preservation of milk.** H. D. ELKINGTON. From OLIGODYN A.-G. (B.P. 357,688, 21.7.30).—The original qualities of fresh milk may be preserved for several days by a process involving a 1–10-min. contact with an oligodynamically active substance, a short pasteurisation treatment in which the heating is interrupted before any deleterious effects can occur, a rapid cooling, and final retention in cold storage. C. JEPSON.

**Making chewing-gum base [containing rubber].** W. W. TRIGGS. From SWEETS LABORATORIES, INC. (B.P. 357,350, 25.8.30. Addn. to B.P. 347,376; B., 1931, 696).—A solution of an alkali, e.g.,  $\text{NaOH}$ , is added to rubber latex, so that the latter separates into 2 layers; the upper layer may again be treated with alkali and the creaming repeated. Resinous material, e.g., coumarone resin, is then added to the cream, and the mixture is heated with stirring so as to remove  $\text{H}_2\text{O}$  and render the mass homogeneous. After removal of residual alkali by washing, the product is mixed with hard, hydrogenated oil. D. F. TWISS.

**Apparatus [rotary toasting etc. ovens] for treating food materials.** II. J. C. FORRESTER. From KELLOGG Co. (B.P. 357,515, 17.5.30).

**[Apparatus for] preservation or keeping and display of food substances [especially fish].** M. T. ZAROTSCHENZEFF, and LIVERPOOL REFRIGERATION Co., LTD. (B.P. 358,392, 24.1.31).

**Grinding of org. peroxides.**—See III. **Beer containing vitamins.**—See XVIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Modification of hypnotic action through changes in chemical structure.** H. A. SHONLE (Ind. Eng. Chem., 1931, 23, 1104—1108; cf. A., 1930, 1047).—The narcotic action of a number of unsym. dialkyl-barbituric acids has been studied. In the ethyl-alkyl series, as the mol. wt. of the alkyl group increases, max. effect occurs at the amyl group, thereafter diminishing; the secondary alkyl groups are approx. twice as effective as the primary and isoprimary isomerides, but this is

not due to a group having optical activity. If the Et group be replaced by a more complex one, the effectiveness is lessened, but at the same time a certain balance must exist between the mol. wts. of the two substituent radicals. The relationships of the groups are changed by replacing Et by allyl. The effectiveness of alkyl groups in barbituric acids is 50 times that of the group in alcohols. Unsaturation usually increases effectiveness, provided the groups have like numbers of C atoms. Introduction of  $\text{CH}_2\text{Ph}$  or propargyl produces tremors and convulsions. Arylethylbarbituric acids, aryl-methyl- and -ethyl-hydantoins have been studied, the  $\text{CH}_2\text{Ph}$  group being out of its homologous position in the Ph,  $\text{CH}_2\text{Ph}$ , and  $\text{CH}_2\cdot\text{CH}_2\text{Ph}$  series. Chemical structure seems to affect the activity by modifying the physico-chemical properties of the compounds, such as  $\text{H}_2\text{O}$ -solubility, and partition coeffs. between  $\text{H}_2\text{O}$  and oil. F. R. SHAW.

**Acetylsalicylic acid in citrate and other solutions.** S. H. STROUD (Sydney Univ. Reprints, 1927, [iii], 1, No. 33—44, 2—7).—Acetylsalicylic acid is hydrolysed (40% in 3 days at room temp.; completely on boiling for a few min.) by solutions of  $\text{KOAc}$ , or K, Li, or Na citrate, in solutions of which it is often prescribed. CHEMICAL ABSTRACTS.

**Derivative of acetylsalicylic acid.** S. H. STROUD (Sydney Univ. Reprints, 1927, [iii], 1, No. 33—44, 2—10).—A solution of acetylsalicylic acid in  $\text{KOAc}$  solution deposits crystals, m.p. 170–5° (also obtained by half neutralising an alcoholic solution with  $\text{KOH}$ ), composed of 1 mol. of the acid associated with 1 mol. of the K salt, probably through an oxonium salt. CHEMICAL ABSTRACTS.

**Determination of halogens in pharmacopœial organic compounds.** U. O. OAKDALE and J. L. POWERS (J. Amer. Pharm. Assoc., 1930, 20, 881—885).—The U.S.P. and Thompson-Oakdale (A., 1930, 799, 1303) methods are compared. Analyses of  $\text{CHI}_3$ , carbromal, etc. illustrate the accuracy of the latter method. Discrepancies in thyroid analyses are ascribed to inaccuracies in the U.S.P. method. H. DAVSON.

**Extracts obtained in the manufacture of ethereal oils from conifers.** P. A. BOBROV (Trud. Vyat. Nauk. Issledov. Inst. Kraev., 1928, 4, 76—86).—A by-product obtained in the manufacture of fir-tree oil, on hydrolysis, affords phenols, pyrocatechol, resorcinol, a carbohydrate, protocatechuic and gallic acids, and a neutral substance. The tanning qualities of the extracts were determined. CHEMICAL ABSTRACTS.

**Report [on tobacco curing].** Tobacco.—See XVI. **Bactericides.** Lemon juice.—See XIX. **Sapocresols.**—See XXIII.

#### PATENTS.

**Manufacture of 1-phenyl-2-aminoalcohols(1) hydroxylated in the phenyl nucleus [i.e., compounds allied to adrenaline].** I. G. FARBERIND. A.-G. (B.P. 353,361, 26.11.30. Ger., 26.11.29).—A hydroxylated aryl ketone is oximinated, and first the oximino-group and then the CO group is reduced. *p*-Hydroxypropiofenone gives an oximino-compound, m.p. 147°, from which  $\beta$ -amino- $\alpha$ -*p*-hydroxyphenyl-propyl alcohol (hydrochloride, m.p. 147°) is obtained.

Oximino-*m*-hydroxypropiofenone, m.p. 130°, yields the corresponding *m*-hydroxy-compound (hydrochloride, m.p. 180°), and oximino-3:4-dihydroxypropiofenone, decomp. 212°, is reduced to the 3:4-dihydroxy-aminoalcohol (hydrochloride, m.p. 178°). *m*-Hydroxypropiofenone, m.p. 69°, is obtained by diazotising the amine, b.p. 138—140°/2 mm., from *m*-nitropropiofenone, m.p. 96—97°. C. HOLLINS.

**Manufacture of morphine and other alkaloids from opium.** A./S. FARMAKON (B.P. 353,069, 17.4.30. Nor., 17.4.29).—The crude material freed from fats etc. is basified (to  $p_{\text{H}}$  9) and the dried mass extracted with an org. solvent (mixture of an alcohol and a halogenated hydrocarbon, e.g., MeOH and  $\text{CHCl}_3$ ), from which morphine is precipitated by addition of  $\text{H}_2\text{O}$  or  $\text{COMe}_2$ . The mother-liquors yield other alkaloids on evaporation to dryness with sand and extraction with  $\text{Et}_2\text{O}$ .

C. HOLLINS.

**Manufacture of [antiseptic] tobacco.** W. T. ARMITAGE and W. BELL (B.P. 357,517, 19.5.30).—The tobacco is sprayed with a mixture (proportions given) consisting of menthol, eucalyptus oil, pini pumilio, cinnamon ver. oil, and spirit, and then stored for about 14 days in airtight containers. II. ROYAL-DAWSON.

**Manufacture of a crystallised product from *Digitalis*.** CHEM. FABR. VORM. SANDOZ (B.P. 357,926, 26.2.31. Ger., 1.3.30).—Moist *Digitalis* leaves (any species) are extracted with non-miscible org. solvents in presence of easily sol. salts giving an extract which is separated, concentrated, and mixed with  $\text{Et}_2\text{O}$ , thus precipitating the glucosides in the form of tannoids. The ppt. is digested with  $\text{Et}_2\text{O}$ , then dissolved in an aq. org. solvent, tannin is eliminated, and the solution conc. until precipitation occurs. The ppt. is recrystallised. A further quantity may be obtained by suitably working up the tannoid ppt. The product (C 60.4%, H 8.0%) has  $[\alpha]_D^{20} +25^\circ$  (4% in dioxan), and is 5 times as sol. in MeOH as digitoxin, to which it has a cardio-active effect quantitatively equal but qualitatively somewhat different. E. H. SHARPLES.

**Manufacture of hormone preparations which are active *per os*.** SCHERING-KAHLBAUM A.-G. (B.P. 357,555, 10.4.30. Ger., 22.4.29).—The hormone, e.g., insulin, and a difficultly saponifiable lipoid substance, particularly one of the lipins produced in the manufacture of female sexual hormones, e.g., placenta lipins, are dissolved in a volatile solvent, with or without absorption-promoting agents such as bile acids, polysaccharides, etc., and the solution is evaporated.

E. H. SHARPLES.

**Culture of bacteria for a mixed auto-vaccine.** A. BECKER (B.P. 357,553, 20.3.30. Ger., 16.1.30).—Sp. excitors of the disease obtained either from the faeces, tonsils, sputum, or other centre of the disease are grown on banana agar with or without blood or egg-white, in approx. the same ratio as they occur in the normal diseased body and for a length of time varying with the number of white corpuscles in the patient. Germs from the different sources may be mixed to form a combined auto-vaccine. E. H. SHARPLES.

**Finely-divided phenolphthalein.**—See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Experimental study of several methods of representing photographic sensitivity.** R. DAVIS and G. K. NEELAND (Bur. Stand. J. Res., 1931, 7, 495—517).—The relationship of the sensitivity of various photographic emulsions to the amount of development has been investigated, the sensitivity being expressed by  $10/I$ , where  $I$  is the intercept made by the tangent to the sensitivity curve at the point of max. slope on a horizontal line representing the fog density, by  $10/I$ , where  $I$  is the exposure val. of the intersection of the tangent with the zero density line, and by  $10/E_m$ , where  $E_m$  is the exposure corresponding with the point in the toe of the characteristic curve where the gradient is 0.20. The sensitivity expressed in any of these ways normally rises to a max. as the time of development increases, then falls again, the optimum time for development depending on the type of emulsion used. The sensitivity of any given emulsion cannot be adequately represented by a single numerical val.; a graph of the  $10/E_m$  index against development time seems most useful. From the shape of the latter curve for emulsions other than the panchromatic or process types it appears that where the utmost sensitivity of the plate is required development should not be carried very far. R. CUTHILL.

**Sensitometry.** H. BAINES (Chem. & Ind., 1931, 706—711).—A review.

### PATENTS.

**Manufacture of photographic sensitive material.** I. G. FARBERIND. A.-G. (B.P. 357,817, 23.10.30. Ger., 23.10.29).—The emulsion support is made of two or more layers, consisting of cellulose derivatives, rubber, or artificial resins; the layers are superimposed by casting the second layer of material (from solution) on to the first, when dry. Any layer (preferably the middle one of three) may be dyed to form a colour filter. The complete support is emulsion-coated on both sides. The layers can be split apart, for printing, by repeatedly bending one corner. Such films may be used for simultaneous two-colour photography. J. LEWKOWITSCH.

**Production of photographic screens, more particularly multi-colour screens.** V. VON GERDANOVITS (B.P. 358,187, 8.8.30. Ger., 9.8.29).—Fine-grain screens are made by applying the colours (lines or grains) to a stretched, thin rubber surface; on contraction the requisite closeness of grain is obtained.

J. LEWKOWITSCH.

**Colour photography.** E. SOMMAVILLA (B.P. 357,625, 20.6.30).—A double picture length of film is coated with emulsion on both sides for one length, the other length having emulsion on one side and white pigment on the other. The first two emulsions are coloured red and blue, respectively, the third yellow, before printing. The negative film (three lengths) is folded round the positive film, so that the two opposite are in register. Light for exposure passes through complementary colour filters so that light falling on one emulsion is prevented by absorption from reaching the opposite emulsion. Printing of the red and blue positives is effected through line screens. The yellow positive is folded over into register with the others for viewing. J. LEWKOWITSCH.



**Colour kinematography.** C. W. FENNINGER, Assec. of F. E. IVES (B.P. 357,604, 24.5.30. U.S., 25.5.29).—The coloured images are produced on the picture record while the sound record is protected by a waterproof coating. The sound record may be made red, simultaneously with the red image, if the colour produced is sufficiently opaque to light affecting the photo-electric cell. [Stat. ref.]

J. LEWKOWITSCH.

**Carbon and like processes employed in colour photography.** L. W. OLIVER, W. G. CLARE, and COLOUR PHOTOGRAPHS (BRIT. & FOREIGN), LTD. (B.P. 357,548, 23.6.30. Cf. B.P. 340,605; B., 1931, 368).—In the "carbon" and "Carbro" processes, regenerated cellulose (*e.g.*, "cellophane") is used for the support during development, and another material, *e.g.*, paper or glass, for the final support. Transference to the latter is aided by an adhesive (gelatin) solution as mountant, and by treating with a hardening solution before stripping the development support.

J. LEWKOWITSCH.

**Development of photographic negatives.** F. HEPNER (B.P. 357,218, 16.6.30).—The negatives are developed in a very dil. solution containing one developing substance (*e.g.*, metol, quinol, etc.) and  $\text{Na}_2\text{SO}_3$  (or  $\text{K}_2\text{S}_2\text{O}_5$ , etc.) only. The negatives are placed horizontally and kept motionless. Al or rustless steel utensils can be used without deterioration.

J. LEWKOWITSCH.

**Protecting the image-bearing surface of kinema films.** O. DEMARET (B.P. 358,224, 6.9.30. Belg., 14.8.30).—A thin skin of cellulose is applied with transparent adhesive, or is formed directly on the surface by immersing it in a cellulose varnish. [Stat. ref.]

J. LEWKOWITSCH.

## XXII.—EXPLOSIVES; MATCHES.

**Calcium nitrate as a constituent of explosives.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 118—119; Chem. Zentr., 1931, i, 2833).—Comparative experiments on explosive force are recorded. A  $\text{Ca}(\text{NO}_3)_2$  mixture dried at  $120^\circ$  was superior to a saltpetre blasting powder.

A. A. ELDRIDGE.

**Constituents of smokeless powder.** M. GIUA and G. RACCIU (Atti R. Accad. Sci. Torino, 1930, 65, 205—212; Chem. Zentr., 1931, i, 2833).—A study of the stabilising effect of vaseline and petroleum on nitroesters.

A. A. ELDRIDGE.

**Stability of pentaerythrityl nitrate and hexogen.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 132—136; Chem. Zentr., 1931, i, 2833).—The stability of pentaerythrityl nitrate is less than that of nitro-explosives or hexogen owing to its ester nature. The stability was not reduced by the presence of powdered metals.

A. A. ELDRIDGE.

### PATENTS.

**Manufacture of explosives containing calcium nitrate.** DYNAMIT-A.-G. VORM. A. NOBEL & Co. (B.P. 357,432, 5.12.30. Ger., 4.6.30).—Commercial  $\text{Ca}(\text{NO}_3)_2$  is melted in its own  $\text{H}_2\text{O}$  of crystallisation, and dehydrated above  $100^\circ$  with agitation so as to give a fine powder. It is then mixed with other ingredients,

*e.g.*, charcoal, sawdust, or trinitrotoluene, and the mixture is incorporated with nitroglycerin. A suitable composition contains  $\text{Ca}(\text{NO}_3)_2$  62, trinitrotoluene 18, sawdust 6, charcoal 4, and nitroglycerin 10%.

W. J. WRIGHT.

**Diminishing the explosiveness of explosive gaseous mixtures.** W. P. JORISSEN and J. BOOY (B.P. 357,530, 14.5.30. Holl., 22.5.29).—One or more halogen or  $\text{PhOH}$  derivatives, or both, which act as negative catalysts for explosive reactions, are added in the form of an atomised vapour or liquid or of a finely-divided solid.

W. J. WRIGHT.

**[Manufacture of strips of] matches.** N. H. WALDINGER (B.P. 358,273, 3.10.31).

Nitrocellulose solutions.—See XIII.

## XXIII.—SANITATION; WATER PURIFICATION.

**Acid sludge digestion.** E. L. PEARSON and A. M. BUSWELL (Ind. Eng. Chem., 1931, 23, 1144—1145).—An unseeded sludge-digestion experiment failed to develop normal digestion for some unknown reason, but became acid, the  $p_{\text{H}}$  decreasing to 6.1 and the volatile acid content increasing to 3800 p.p.m. in 130 days. The vol. of gas produced was below normal, though the sludge foamed persistently, the average composition of the gas being  $\text{CH}_4$  54.8%,  $\text{CO}_2$  28.0%,  $\text{H}_2$  0.9%,  $\text{N}_2$  16.3%. Under such conditions it would appear that the decomp. of protein matter is retarded less than that of the grease.

C. JEPSON.

**Sludge-digestion capacity.** E. L. PEARSON and A. M. BUSWELL (Ind. Eng. Chem., 1931, 23, 1154—1155).—Experiments carried out with a 1330-gal. digestion tank operating at  $27^\circ$  with a detention period of 10.7 days (on a basis of dry org. matter) indicate that a 90% yield of gas may be obtained with a capacity allowance of 0.5 cu. ft. per head. The sludge produced would be inoffensive, but not a well-drying one, and the tank should be fitted with mechanical thickeners for the digesting sludge and with adequate scum and foaming control.

C. JEPSON.

**Sludge drying beds.** J. F. SKINNER (Sewage Works' J., 1931, 3, 390—399).—Drying beds completely covered with glass cost three times as much to construct as open ones. The maintenance and insurance costs of the covered beds are also high, but flies and odours are more under control and about double the quantity of sludge can be dealt with.

C. JEPSON.

**Bacterial studies before and after chlorination at the Madison sewage-disposal plant.** B. DOMOGALLA (J. Bact., 1931, 21, 9).—In this Imhoff system chlorination reduced bacterial numbers by 50%, *B. coli* by 90%,  $\text{H}_2\text{S}$  producers by 80%, and protein digesters by 50%. Rates of nitrification and denitrification were not appreciably altered by chlorination.

A. G. POLLARD.

**Partial and complete sterilisation of activated sludge and effect on purification [of sewage].** H. HEUKELEKIAN (Sewage Works' J., 1931, 3, 369—373).—Inhibition of protozoan activity by KCN or  $\text{PhMe}$  results in an increase in the bacterial population. KCN and  $\text{Cl}_2$  deflocculate the sludge and cause an increase in the

biochemical  $O_2$  demand of the effluent, but PhMe produces an opposite effect. C. JEPSON.

**After-effect of ferric chloride on sludge digestion.** W. RUDOLFS and L. R. SETTER (Sewage Works' J., 1931, 3, 352—361).—Mixtures of fresh sewage solids and ripe sludge were made in which one or other ingredient, or both, had been pretreated with  $FeCl_3$ . Those containing treated ripe sludge showed a retarded rate of gasification, and only treated fresh solids showed an accelerated rate though less total gas. The ripe sludges produced from all the mixtures when used as seed material showed a marked increase in gas production, probably due to some of the intermediate reaction products of the first experiment having reacted with the  $FeCl_3$  and been broken down further in the second experiment when a new flora had been established. C. JEPSON.

**Imhoff tanks at Philadelphia.** T. F. NEVIN (Sewage Works' J., 1931, 3, 400—404).—An offensive, greasy scum on the settling compartment was beaten down by spray from a water-hose, but, later,  $Ca(OH)_2$  had to be piped down and distributed through the digesting material to restore alkalinity and obtain satisfactory digestion. A daily upward application of rinsing water to the digesting sludge for a 5-min. period is effective in preventing undue densification of the sludge, its adhesion to the tank sides, and the formation of scum by entrained gases. Excessive foaming has been prevented by applying water from a  $\frac{3}{4}$ -in. nozzle. C. JEPSON.

**Operation of New Castle sewage plant.** F. J. NUGENT (Sewage Works' J., 1931, 3, 404—410).—An average daily flow of 4,800,000 gals. is treated in Imhoff tanks fitted with gas collectors. About  $\frac{1}{2}$  cu. ft. of gas (17%  $CO_2$ ) is obtained *per capita* per day, but the thermal val. of most of it is wasted by burning the gas as a means of odour prevention only. C. JEPSON.

**Removal of grease and oils from sewage by the Imhoff method.** W. A. ALLEN (Sewage Works' J., 1931, 3, 374—376).—The type of grease separator proposed by Imhoff, in which agitation and aeration are obtained by forcing air through diffusers placed along one side of the sewage carrier, has proved successful at Pasadena in preventing undue amounts of grease from reaching the activated-sludge plant. The present unit allows a detention of 1.3 min. and is being enlarged to permit a 3.0-min. stay. C. JEPSON.

**Los Angeles method of grease removal [from sewage].** G. A. PARKES (Sewage Works' J., 1931, 3, 377—379).—Agitation and aeration produced by the injection into the sewage of fine streams of high-pressure screened sewage has proved more successful at this plant than the diffused-air arrangement proposed by Imhoff. C. JEPSON.

**Effects of some insecticides on the sewage sprinkling-filter fly, *Psychoda alternata*.** W. W. FRYE, M. LEVINE, and E. R. BECKER (Sewage Works' J., 1931, 3, 345—351).—Attempts to control the adult *Psychoda* fly by destruction of the larvæ in the filter films by means of chemicals were unsuccessful,  $Cl_2$ ,  $CS_2$ ,  $CuSO_4$ , nicotine sulphate, pyrethrum, and several arsenate compounds being tried. Adult flies were kept in check by spraying the walls and ceilings of enclosed

filters with a kerosene-pyrethrum mixture, but this method might not be applicable to open filters.

C. JEPSON.

**Antiseptic and germicidal efficiency of "pyridium" on various organisms.** B. E. PROCTOR (J. Bact., 1931, 21, 54).—Pyridium (benzeneazo-2:6-diaminopyridine hydrochloride) proved efficient against a number of organisms. A. G. POLLARD.

**Purification of waste waters from leather factories by aeration in presence of activated sludge and of slag.** L. F. KABAKOVA and M. M. KALABINA (Trans. Centr. Comm. Protec. Water Reservoirs, 1927, No. 6, 119—152).—Activated sludge can be obtained from the ppts. of the waste waters. In the case of aeration this sludge produces nitrification of the waste water. The best purification is obtained with 33% sludge. The concentration of the waste water can be lowered by treatment with Al or Fe salts, coagulation with sludge, etc. A non-putrefying product is obtained from highly conc. waste waters, after dilution or coagulation, by oxidation in contact with brown-coal slag.

CHEMICAL ABSTRACTS.

**Sapocresols.** S. JENČIČ (Arh. Hemiju, 1931, 5, 197—203).—Commercial sapocresol solutions exhibit an abrupt max. viscosity at 60%  $H_2O$  at 16°; this max. is less marked at higher temp. and is absent at 25°. The concentration of K stearate required to form jellies with *o*-, *m*-, and *p*-cresol is 10 times that of Na stearate. K stearate at concentrations < 25% of stearate does not yield jellies with *m*-cresol, < 15% with *o*-cresol, and < 22% with *p*-cresol. R. TRUSZKOWSKI.

**Boiler-water chemistry.** H. E. JONES (Chem. & Ind., 1931, 50, 854—858).

**Chemical risks in the rubber industry.**—See XIV.

PATENTS.

**Insecticide.** W. MOORE, Assr. to AMER. CYANAMID Co. (U.S.P. 1,797,877, 24.3.31. Appl., 3.8.28).—The material comprises a quaternary base produced by the reaction of a *tert*-alkylamine and a halogen derivative of an unsaturated hydrocarbon; *e.g.*, an equimol. mixture of rimethylamine and ethylene dichloride is heated in EtOH solution at 60—75° for 22 hr. under pressure, whereby trimethylchloroethylammonium chloride is obtained which has a highly toxic action on plant lice.

A. R. POWELL.

**Insecticide and fumigant.** G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,795,593, 10.3.31. Appl., 23.4.26).— $CaCN_2$  and NaF are fused in an electric furnace at 1400° and the product is cooled rapidly below 400°. With an excess of NaF leaching the fused mass affords a solution of NaCN. With only a small amount of NaF the product consists mainly of  $Ca(CN)_2$ .

A. R. POWELL.

**Base-exchanging filters [for water].** S. A. KRÜGER (B.P. 357,401, 24.10.30. Denm., 26.4.30).—The pre-filtering media placed above the softening material should have a lower sp. gr. than the latter, so that it may all be smaller in size and retain its position on the surface and its function as a protector without requiring a metallic separating device or screen. C. JEPSON.

**Insecticide.**—See XVI.

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

DEC. 18 and 25, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Influence of composition of the solution under treatment on decolorisation with active carbons.** V. EDELSTEIN (Z. Zuckerind. Czechoslov., 1931, 56, 49—55).—Neutral salts exerted a small and acid salts a distinct effect in increasing the decolorising effect;  $\text{CaCl}_2$ , on the contrary, through its peptisation effect diminished it.  $\text{K}_2\text{HPO}_4$  by its buffering action raised the decolorising effect, emphasising the importance of identical conditions as regards buffering and actual  $[\text{H}^+]$ .

J. P. OGILVIE.

**Portable apparatus for the complete analysis of technical gases.** E. OTT (Gas- u. Wasserfach, 1931, 74, 970—971).—The apparatus described previously (cf. B., 1929, 999) is improved by the addition of a case to contain the burette and serve also as a support for the main case during use. An additional pipette, which may be used for gas storage, or for the absorption of CO by  $\text{Cu}_2\text{Cl}_2$  where the use of  $\text{I}_2\text{O}_5$  is not permissible, is added. The method of carrying out the analysis is briefly described. The apparatus is suitable for use by Pauschardt's method (B., 1931, 791). The  $\text{I}_2\text{O}_5$  reagent is prepared by mixing 1 pt. of  $\text{I}_2\text{O}_5$  with oleum and I in a mortar and then shaking the paste with 14—15 pts. of 10% oleum for 3 hr. The reagent may be revived by the addition of 25% oleum.

A. B. MANNING.

**Heat-transmission rates round a tube in a transverse current of fluid.** J. SMALL (Engineering, 1931, 132, 569—570).

**Filters for sugar factories.**—See XVII.

See also A., Nov., 1262—3, **Comparative spectrophotometry.** 1264, **Capillary viscosimeter.** **Filtration of colloidal solutions.**

### PATENTS.

**Rotary drum furnace.** C. LEAN. From J. D. BRACKELSBERG (B.P. 358,352, 29.11.30).—A rotary drum furnace for smelting etc. is mounted on a cradle so that it can be tilted about a transverse axis which approx. passes through the centre of gravity. The standards which support the cradle may also support a platform for charging etc. The taphole for metal is remote from the axis, but the outlet for gases (when level) and slag (when tilted) is axially at one end, a corresponding inlet for fuel and a new charge at the other end being formed.

B. M. VENABLES.

**Continuous heating furnace.** S. E. DIESCHER, Assr. to S. DIESCHER & SONS (U.S.P. 1,800,170, 7.4.31. Appl., 25.9.29).—A furnace for sheets etc. has a conveyor bed composed of rollers adjacent pairs of which are embraced by large rings. The bending moment on the roller shafts

inside the furnace is reduced by hanging weights on extensions of the shafts outside the furnace.

B. M. VENABLES.

**Tubular heat-exchanging apparatus, for pre-heating air or other gases.** A. E. WHITE. From SHAW-PERKINS MANUFG. Co. (B.P. 358,179, 29.7.30).—The horizontal tubes which carry the gas to be heated are divided into groups which are mounted on rails in a framework, in such a way as to permit considerable expansion of the whole and a certain amount of differential expansion of the parts.

B. M. VENABLES.

**Apparatus for preparing and introducing disin-crustants into steam boilers.** FILTRATORS, LTD., and V. V. SAKS (B.P. 358,034, 28.3.30).—Steam is passed downwards through a cage containing vegetable seeds, and the liquid extract accumulates in the lower part of the surrounding vessel, whence it is discharged at intervals by an automatic valve acting as a steam trap.

B. M. VENABLES.

**Manufacture of anti-incrustation briquettes or blocks, more especially intended for use in steam boilers, economisers, and radiators.** J. F. BARTON and G. BUTLER-BARTON (B.P. 358,184, 7.8.30).—Vegetable materials containing tannin, e.g., quebracho extract, are mixed with about 5% of powdered Zn, and, if desired, small quantities (2—3%) of asphaltum and graphite.

A. B. MANNING.

**Ternary composition of matter [for use in refrigerators].** R. W. DAVENPORT, Assr. to CHICAGO PNEUMATIC TOOL Co. (U.S.P. 1,803,098, 28.4.31. Appl., 9.10.26).—The composition comprises 70—90% of  $\text{CH}_2\text{Cl}_2$ , 8—25% of a hydrocarbon oil lubricant miscible with  $\text{CH}_2\text{Cl}_2$ , and 2—10% of MeOH.

L. A. COLES.

**Pulverising installation.** BABCOCK & WILCOX, LTD. From C. PETERS (B.P. 358,219, 5.9.30).—In a gas-borne pulverising system, in addition to the classifier for dust lifted above the mill there is provided a screen below the mill through which fine heavy material (e.g., ash-forming material from coal) is permitted to drop while fine light material (the coal itself) is blown upwards through the mill by a current of gas branched off from the main circulation on the outlet side of the fan. Coarser particles of both stone and coal are retained by the screen, caught by the grinding elements, and reduced until they either fall through the screen or are borne upwards by the gas current.

B. M. VENABLES.

**Pulverisers.** ERIE CITY IRON WORKS, Assees. of C. D. ST. CLAIR (B.P. 357,669, 9.7.30. U.S., 16.7.29).—The housing of a pulveriser is split vertically, enabling the front portion to be removed without disturbing the feeding arrangements or the bearings. The diaphragms

\* The remainder of this set of Abstracts will appear in next week's issue.

dividing the mill into compartments in series along the shaft are also split and provided with renewable linings.

B. M. VENABLES.

**Pulverising and like mills.** E. W. GREEN and G. R. UNTHANK (B.P. 357,542, 19.6.30).—An apparatus of the double-thrust ball-bearing type is described. The lowest ball race and the middle pair are driven in opposite directions; the top race is stationary, but pressed down by springs. An air classifier and means such as a fluted cone for preliminary reduction of the feed are provided.

B. M. VENABLES.

**Disintegration of materials.** C. E. BLYTH (B.P. 358,007, 28.6.30).—The material is fed into a rotor running at such a high speed (20,000 r.p.m. or more) that mere centrifugal pressure is sufficient to crush it. The apparatus is stated to be suitable for comminution of limestone for the manufacture of cement and for the pulverisation of coal. The wall of the centrifuge on which the material is caught may be provided with apertures to discharge the fine material. B. M. VENABLES.

**Mill for grinding or reducing chocolate or other material.** A. SONSTHAGEN (B.P. 357,562, 21.5.30).—A machine of the disc-grinder type is described in which both discs rotate in the same direction at different speeds, producing considerable centrifugal force with a slower relative shearing motion; a formula is given by means of which the most suitable speeds can be calc.

B. M. VENABLES.

**[Rotary drum] filters.** MINING & INDUSTRIAL EQUIPMENT, LTD., and H. MEAD (B.P. 358,076, 4.6.30).—A cellular drum filter, in which radial pipes are eliminated by forming the cells in triangular shape (in transverse section) and in stream-line form (in longitudinal section), the apices of the triangles being connected to the ported trunnions, is constructed with each cell as a separate interchangeable unit, preferably assembled alternately right- and left-hand, so that both trunnions are utilised.

B. M. VENABLES.

**Centrifuges.** H. G. C. FAIRWEATHER. From NAT. ACME Co. (B.P. 357,988, 30.6.30).—The bowl is composed of four co-axial, cylindrical walls entirely without apertures, all the discharge and transfer ports being formed in the driving hub or cover. Counting the walls 1—4 outwards, the flow of fluid is through the space between 1 and 2, then between 3 and 4, and between 2 and 3; discharge ports are provided from the outer two spaces, the radius of the outermost ones being adjustable by forming them as eccentric holes in screwed plugs. A centrifugal clutch and a brake are described.

B. M. VENABLES.

**Screw presses for separating liquid from pulp or pasty substances.** H. A. HARTNER (B.P. 357,873, 10.12.30).—A screw-conveyor press is provided with means for altering the feeding point so that the degree of compression may be varied by altering the length of screw in use. Several alternative fixed entrances or a single entrance through a sliding sleeve may be provided.

B. M. VENABLES.

**Means for evaporation, concentration, or condensation of fluids or liquids.** R. SELIGMAN, and ALUMINIUM PLANT & VESSEL Co., LTD. (B.P. 357,605,

24.5.30).—A heat-exchanger of the filter-press type is adapted to evaporation and/or condensation by forming comparatively large passages along the upper and lower edges of the plates. Preferably the channels for the fluid to be treated are vertical and direct between the passages, whilst those for the heating or cooling medium are zig-zag. The apparatus may be divided into stages by the insertion of differently formed plates and frames so that, in evaporation, the liquid may be treated several times and the vapour from one stage used to heat liquid in another.

B. M. VENABLES.

**Atomisation or distribution of liquid or semi-liquid materials.** E. ROTHEIM (U.S.P. 1,800,156, 7.4.31. Appl., 30.9.27. Norw., 8.10.26).—A coating composition is dissolved in  $\text{Me}_2\text{O}$  and stored in a metal bottle provided with a spraying head. Spraying is effected by the pressure of the  $\text{Me}_2\text{O}$  (3.7 atm. at 20°), which evaporates close to the point of issue from the bottle so that the substance is dispersed not merely by the mechanical action of the spray, but also because it was, a very short time before, dispersed in the solvent.

B. M. VENABLES.

**Bubbling apparatus for treating liquids with gases.** INDUSTRIEKKEMISKA AKTIEB. (B.P. 357,599, 22.4.30. Swed., 22.4.29).—The gas flows upwards through perforated plates and the liquid downwards through seals, being maintained in a thin layer on each plate. The perforations are so spaced that the bubbles formed round and released from each aperture do not touch and coalesce; if the holes have a diam. of 2 mm. or less the spacing should be at least five times the diam., but if the latter is over 2 mm. a spacing of twice the diam. may be sufficient. The holes may be bevelled, inclined to promote movement in the liquid by the issuing gas, or provided with helical vanes to produce rotation of the gas.

B. M. VENABLES.

**Filling elements or packing for chambers for treatment of liquids with gases.** E. ROTHAMMEL (B.P. 358,258, 24.9.30).—The elements comprise boxes having four sides and an apertured bottom, and also one or more internal fins; they are laid in order in the tower like bricks, the bottoms of the course above forming the lids of the course below and the apertures not being alined.

B. M. VENABLES.

**Filters for gases.** H. WITTEMEIER, Assee. of DEUTS. LUFTFILTER BAUGES. M.B.H. (B.P. 357,990, 30.6.30. Ger., 20.7.29. Addn. to B.P. 355,431; B., 1931, 954).—The filter is composed of two sheets of expanded metal rolled together with the slots oppositely inclined in the alternate sheets.

B. M. VENABLES.

**Removal of dust from gases by washing.** G. H. C. CORNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 358,101—3, 4.7.30).—In an apparatus of the type described in B.P. 321,268 (B., 1930, 41), saturation of the gases is considered to be the main function of the countercurrent sprays. Subsequently the gas is cleaned by flowing over vertical, irrigated surfaces comprising, in (A), tubes or baffle plates, the velocity of the gases being  $> 12$  ft./sec.; in (B) baffles of glazed vitreous or other material and having pear-shaped bulges at the top to ensure that all the water is spread on the surfaces;

and in (c), baffle plates in two or more sets, bent at an angle which decreases in the direction of flow of the gas, *e.g.*, three sets having angles of 120°, 105°, and 90°, respectively, the velocity being  $\geq 12$  ft./sec.

B. M. VENABLES.

**Purification of gases by diffusion.** F. H. ROGERS. FROM SKALA RESEARCH LABORATORIES, INC. (B.P. 358,086, 30.6.30).—The constituent that is desired in a pure state is caused to diffuse through a heated sheet of Pt, Pd, SiO<sub>2</sub>, or other material that will act suitably as a filter; the sheet is heated by an electric discharge which also causes the filtered constituent to become luminous, whereby its purity may be examined by a spectroscope.

B. M. VENABLES.

**Device for withdrawing low-pressure gases from high-pressure vessels.** P. GILGENBERG (B.P. 358,260, 24.9.30).—Two reducing valves are used in series with an expansion space in between.

B. M. VENABLES.

**Apparatus for bringing gases and/or vapours into intimate contact with liquids.** S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & CO., LTD. (B.P. 357,712, 7.8.30).—The gas is caused to flow in a zig-zag manner horizontally through irrigated brushes which are arranged in vertical planes; these consist of long narrow brushes with the bristles alternately pointing upwards and downwards.

B. M. VENABLES.

**Sound-deadening compositions adapted to be applied to vehicle body constructions etc.** G. B. DUNFORD (B.P. 357,514, 17.5.30. U.S., 18.5.29).—The composition (for use as a spray) is composed of bituminous substances, a volatile solvent for them, fibre, and colloidal clay. Less than 10% of H<sub>2</sub>O should be present, preferably only a trace.

B. M. VENABLES.

**Refrigerating apparatus.** P. SCHLUMBOHM (B.P. 359,171, 1.10.30. Ger., 1.10.29).

**Refrigeration [apparatus].** W. G. NOLCKEN (B.P. 359,064, 23.7.30).

**Absorption refrigerating apparatus.** ELECTROLUX, LTD., ASSECS. OF H. FREDHOLM (B.P. 358,844, 17.2.31. Swed., 18.2.30).

**Heat insulation [for refrigerators etc.].** ELECTROLUX, LTD., ASSECS. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 359,246, 13.11.30. Ger., 14.11.29).

**Evaporators for refrigerating apparatus.** BRIT. THOMSON-HOUSTON CO., LTD., ASSECS. OF H. S. WOODRUFF (B.P. 359,119, 28.8.30. U.S., 28.8.29).

**Temperature-controlling devices or thermostats [for use with fuel burners].** BRIT. HARTFORD-FAIRMONT SYND., LTD., T. WARDLEY, and T. E. BOWEN (B.P. 358,545, 12.6.30).

**Metal-melting furnace.**—See X. **Treatment of gases.**—See XI. **Pasteuriser.**—See XVIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Producers for carbonising plants.** E. W. SMITH (Gas J., 1931, 196, 204—207).—The advantages of step-grate producers are discussed, particularly with reference to their freedom from clinkering. Ample producer

capacity should be attached to all carbonising plants, and it is recommended that a mean grate area corresponding to the gasification of 10 lb. of coke per sq. ft. per hr. should be allowed. An average normal depth of fuel bed in step-grate producers is 5 ft. 6 in., but if small coke or breeze is to be used, this depth might be reduced to 3 ft. Coke breeze may be used providing that sufficient grate area is allowed. Operating costs of step-grate producers are shown to be half those of mechanical producers.

C. B. MARSON.

**Torbanite carbonisation and products in South Africa.** E. NEUFELD (J. Chem. Met. Soc. S. Africa, 1931, 32, 24—28).—Carbonisation is carried out preferably in an internally-heated retort, using scavenging gases as the heating medium. The optimum size of material is  $\frac{1}{2}$ —1 in.; the carbonising temp. should not exceed 500°; the yield of oil should be at least 90% of the laboratory test, and the max. dust content < 0.5%. An Ermelo torbanite (H<sub>2</sub>O 1.0, ash 23.4, C 60.9, H 7.9, S 0.5, O + N 6.3%) yielded 35.5% of tar consisting of light motor spirit (to 150°) 12.8%, heavy motor spirit 10.4%, Diesel oil 17.1%, light engine oil 32.5%, heavy engine oil 10.8%, paraffin wax 7.8%, coke 4.9%, gas and loss 4.1%. The oils contained creosote oil (8.5%) which is preferably extracted from the crude oil with EtOH. The results of distilling another torbanite in an externally-heated retort are also tabulated; the products were of lower quality than those obtained with an internally-heated retort. The nett revenue from the process is estimated at 25s. per ton of torbanite carbonised.

A. B. MANNING.

**Thermochemical behaviour of brown coal during the carbonisation process.** P. NIEMANN (Brennstoff-Chem., 1931, 12, 386—389).—Brown coal has been carbonised in an apparatus comprising an electrically-heated retort, a receiver, and a cooling coil, the whole of which was immersed in a calorimeter. With an inert material in the retort the temp. of the calorimeter was a linear function of the time. The temp.-time curve of the calorimeter during the carbonisation of the brown coal departed from the linear in a direction indicating the occurrence of exothermic reactions over the range corresponding with retort temp. of 200—800°.

A. B. MANNING.

**[Evaluation and assay of coal for gas manufacture.]** A. McDONALD (Gas World, 1931, 95, 397—398).—Comparison of Gray-King assays and working results over a period of 3 years shows that factors may be devised which can be used to predict the yields of gas, coke, tar, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and liquor obtained in various types of vertical retort. A first approximation to the thermal result as obtained in the assay plant can be calc. from the proximate analysis by the formula: therms/ton =  $3.6V - 5.5V^2/(V + C)$ , where  $V$  is the % volatile matter and  $C$  the calorific val. of the gas in B.Th.U./lb.; to obtain the corresponding works' figures this amount must be multiplied by a factor varying between 1 and 1.4, depending on the type of plant and degree of steaming. The coke yield can be obtained in cwt./ton by dividing the % by 5.3. Crit. temp. in the assay of coal are: initial aq. condensate 430°, initial gaseous product 460°, initial tarry matter

510°, first active decomp. 590°, final tarry matter 650°, second active decomp. 670°, third active decomp. 730°. Coals which produce the best coke evolve most of their gas between 590° and 670°, and it is suggested that the quantity of gas evolved during this interval may be taken as a measure of the coke quality.

C. B. MARSON.

**Chemical value of coke-oven gas.** F. MUILETT (Chem.-Ztg., 1931, 55, 826—827).—Known processes for the separation and utilisation of the individual constituents of the gas are reviewed. II. E. BLAYDEN.

**Influence of pressure on some water-gas reactions.** F. FISCHER and H. PICHLER (Brennstoff-Chem., 1931, 12, 365—372).—The rate of the reaction between  $H_2O$  and semi-coke at 500° ( $C + 2H_2O = CO_2 + 2H_2$ ) is reduced by increasing the pressure, owing to the formation of a layer of inert gases on the surface of the coke. If this layer is removed by raising the temp. or by adding alkali the secondary formation of hydrocarbons commences. In the catalytic hydrogenation of CO, increased pressure favours the deposition of products of high mol. wt. on the catalyst, the activity of which therefore decreases more rapidly than at the ordinary pressure. Low pressures, e.g., 0.1 atm., are unsuitable for the reduction of CO to hydrocarbons since a greater vol. of catalyst is necessary to bring about the same % conversion. The course and the velocity of the reactions between  $CH_4$  and  $CO_2$  or  $H_2O$  are greatly influenced by the pressure. Whilst the equilibrium  $CH_4 + CO_2 \rightleftharpoons 2CO + 2H_2$  at 900° under 1 atm. pressure can, with the aid of a Ni catalyst, be moved almost completely over to the right-hand side, the same result can be reached at considerably lower temp. by diminishing the pressure; e.g., at 500—600° under 0.01 atm. pressure the whole of the  $CH_4$  is converted into CO and  $H_2$ . The reaction velocity increases with diminution in pressure. The equilibrium concentrations have been determined as a function of the pressure; the observed vals. agree with those calc. from the equilibrium consts. of the reactions  $2CO = C + CO_2$  and  $2H_2 + C \rightleftharpoons CH_4$ . A. B. MANNING.

**Benzol recovery from gas.** A. WEINDEL (Brennstoff-Chem., 1931, 12, 385—386).—The yield of benzol obtained by oil-scrubbing a gas may be greater in practice than is indicated by testing the gas, e.g., by the active-C method, before and after scrubbing. This is probably due to separation of tar fog, with its included benzol, in the tubes leading to the active-C absorber. Other minor anomalies in the benzol-recovery process are briefly discussed. A. B. MANNING.

**Formation of aromatic substances in brown-coal tars.** E. DONATH and R. FOLGNER (Chem.-Ztg., 1931, 55, 665—666).—The occurrence of aromatic substances in tars from hard coal is ascribed to the presence in the coal of protein derived from animal residues during the coal formation. A mixture of cellulose derivatives and protein on distillation gave a tar resembling that from coal. Lignite with added protein, in this case a relatively high proportion of casein, was distilled and the tar gave characteristic reactions for anthracene derivatives. When fractionated at 120°, 230°, and 280°, the relative proportions

were 13, 34.2, and 7.2%, respectively. The amounts of neutral oils, phenolic substances, pyridine bases, etc. were also determined; the coke was a caking one. The possibility that other protein wastes (e.g., slaughter-house refuse, leather scrap, etc.) will give similar results is to be investigated. H. J. DOWDEN.

**Catalytic pressure-hydrogenation of a benzine obtained by cracking brown-coal tar.** J. VARGA and I. MAKRAY (Brennstoff-Chem., 1931, 12, 389—390).—The benzine, which contained 3.8% of S and more than 50% of unsaturated hydrocarbons, was hydrogenated in a rotating autoclave in the presence of a  $MoO_3$  catalyst and with an initial  $H_2$  pressure of 80 atm. At temp. of 380°, 400°, 420°, and 440°, the % S contents of the products were 2.7, 1.3, 0.07, and 0.02, whilst the corresponding contents of unsaturated hydrocarbon were 28.5, 20.0, 4.0, and 1.5%. The greater part of the unsaturated hydrocarbons was converted into aliphatic hydrocarbons. The sp. gr. of the benzine decreased and the proportion boiling below 100° increased. The "gum" content decreased from 21.4 mg. in the crude benzine to 4.4 mg., 2.8 mg., and nil in the products obtained at 380°, 420°, and 440°, respectively. The yields were 92.3 vol.-% at 420° and 88.3 vol.-% at 440°. The consumption of  $H_2$  was 443 litres/kg. at 440°. A. B. MANNING.

**Removal of weak organic acids from oils by treatment with caustic alkali.** P. MEYER (J. Inst. Petroleum Tech., 1931, 17, 621—629).—It is shown by experiment and calculation that the more dil. solution of alkali removes the larger amount of acid substances such as mercaptans, phenols, etc. On the large scale, complete removal can be effected by countercurrent washing, this treatment being also applicable for the removal of  $H_2S$  from the oils.

E. DOCTOR.

**Sp. heat of some crude oils and their residues when topped to 100°.** H. R. LANG (J. Inst. Petroleum Tech., 1931, 17, 585—591; cf. B., 1930, 649).—The deviation between sp. heat-temp. curves for topped and untopped samples of crude oils is attributed to the presence of gas or air. The amount of C residue (Conradson) of topped oils is in indirect proportion to the sp. heat.

E. DOCTOR.

**Activated vegetable carbons. Production and use as decolorising agents.** A. E. WILLIAMS (Chem. Tr. J., 1931, 89, 423—424, 447—448).

**Decolorising with active carbons. Gas-analysis apparatus.**—See I. Varnish pitches.—See XIII.

See also A., Nov., 1226, Activated charcoal. 1233, Water-gas equilibrium. 1249, Condensation of hydrocarbons electrically. 1256, Adsorption and separation of  $H_2$ . 1258, Determination of CO. 1282, 2:3:6:7-Tetramethylantracene in low-temp. tar. 1286, Phenols of coal tar. 1299, Optically active components of petroleum. 1318, Determination of traces of  $C_2H_2$ .

PATENTS.

**Plant for extracting dust from minerals such as coal.** S. HUNTER (B.P. 358,329, 10.11.30).—The coal falls down a sloping wall constituted by a series of

rollers and louvres. An air blast carries the dust through the spaces between the rollers or louvres, behind which are arranged successively a second set of louvres and a set of vertical plates for the purpose of grading the dust. The finest dust which is carried past the vertical plates is removed in a cyclone separator and the cleaned air is then recirculated.

A. B. MANNING.

**Manufacture of agglomerated balls or briquettes from coal, without the addition of tar.** H. HARDY (B.P. 357,330, 30.7.30. Belg., 8.2.30. Addn. to B.P. 356,236; B., 1931, 1035).—Globular coal particles are compressed while still hot, and conveyed to a retort for further heating.

D. K. MOORE.

**Manufacture of agglomerated balls or briquettes from coal without the addition of tar.** H. HARDY (B.P. 358,181, 30.7.30. Belg., 30.11.29. Addn. to B.P. 356,236; B., 1931, 1035).—Ashy coal or carbonaceous schist is heated until the coal particles become spheroidal, and these are separated from the ash or schist by flotation in water, and finally are reheated and briquetted.

A. B. MANNING.

**Device for burning lump or granular fuel.** STOCKHOLMS AKTIEBOLAGET PRIVAT (B.P. 358,172, 24.7.30. Ger., 12.11.29).—The fuel layer on a travelling grate is arranged in such a manner that the large pieces lie underneath and the smaller on top, by raking or otherwise agitating the fuel near the front of the grate close to the fuel-admission point, and at the same time passing a powerful current of air through the fuel at that point. The device permits a thick fuel layer to be worked with a correspondingly high combustion effect without excessive power consumption for a high under-grate blast pressure.

A. B. MANNING.

**Vertical carbonising retorts.** WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 357,146, 16.6.30).—The retort for intermittent carbonisation is about double the length of the ordinary retort, the upper part forming a carbonisation zone externally heated, and the lower part a zone in which water-gas is produced and the coke cooled; this lower part is surrounded by a recuperator. A layer of coke breeze placed on the top of the coke after the withdrawal of some of the latter from the bottom prevents fine coal from passing into the coke.

D. K. MOORE.

**Retort for the distillation of coal, oil shale, or other solid carbonaceous material, and adapted for use in conjunction with a rotary cement kiln.** E. S. HANDASYDE (B.P. 358,110, 5.7.30, 1.11.30, and 18.3.31).—The material is passed down a vertical cylindrical retort containing a series of slightly coned rotating plates in such a manner as to be carried completely round on any one plate before being discharged, by means of a scraper, on to the next lower plate. The retort is heated externally with hot gases and internally by superheated steam, which may be supplied either to the top or the bottom of the retort, and/or to each of the rotating plates. The carbonised material is discharged from the bottom hopper of the retort over a rotating cone-shaped plate into a closed pit containing water, whence it is removed. When the retort is used in conjunction with a cement kiln a finely-divided mixture of

oil shale with coal and limestone may be fed into the retort and the carbonised product conveyed to the kiln. The hot gases from the latter are utilised for the external heating of the retort and for superheating the steam supplied thereto.

A. B. MANNING.

**Destructive distillation of coal and tar mixtures.** BARRETT CO., ASSECS. OF S. P. MILLER (B.P. 358,233, 13.9.30. U.S., 13.9.29).—Coal and tar are introduced simultaneously but independently into a coke oven, and the mixture is then coked. A layer of coal is preferably first charged on to the bottom of the oven.

A. B. MANNING.

**Distillation of solid carbonaceous materials.** H. NIELSEN and B. LATNG (B.P. 357,975, 28.6.30).—The process of B.P. 287,037 (B., 1928, 356) is carried out by using superheated steam as the heating medium, the latent heat of the steam being recovered by injecting effluent liquor from the process into the mixed steam and oil vapours leaving the retort, whereby their temp. is reduced to approx. 100°, the liquor being subsequently transferred to an evaporator wherein it is evaporated, under decreased pressure if desired, by further heat interchange with the spent distilling medium. Part of the steam generated may be used to replenish the distilling medium.

A. B. MANNING.

**Manufacture of carbon black.** J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 357,749 and 358,178, [A] 8.9.30, [B] 28.7.30).—(A) Diacetylene or its substitution products is subjected to partial combustion in air and/or oxides of C. When working with oxides of C a dehydrogenerating catalyst (Pt-asbestos) is used. The narrow outlet tube from, and the safety wire netting in, the burner are water-cooled. (B) In the production of C black by the catalytic decomp. of gaseous hydrocarbons, the clogging of the apparatus by adhering C is prevented by making its inner surface of material which effects no appreciable decomp. of the hydrocarbons, e.g., of Ag, Cu, Cr, brass, Ni- and Ni-Cr-steels.

D. K. MOORE.

**Production of carbon from flames.** BURMAH OIL CO., LTD., S. T. MINCHIN, and R. E. DOWNER (B.P. 357,133, 13.6.30).—Finely-divided C is deposited on conductors, one of which may be rotated, and the C continuously scraped off; the other, forming the burner, is charged with a.c. or d.c. and held in the flame of burning hydrocarbon fuels. The yield is increased by keeping the temp. of the C deposit uniform or by reducing it.

D. K. MOORE.

**Production of active carbon and gases containing free hydrogen.** METALLGES. A.-G. (B.P. 357,778, 24.9.30. Ger., 4.10.29).—Carbonaceous substances, preferably of high ash content, e.g., low-temp. coke, are treated with mixtures of steam and gas which contain up to about 6% of O<sub>2</sub>, at temp. of 500–600°. The steam-gas mixtures are preheated to 120–200°, and the supply of further extraneous heat is preferably avoided. The process is so regulated that loss of material by combustion lies between 5% and 40%. A cheap C of sufficiently high activity for many purposes is obtained.

A. B. MANNING.

**Treatment of activated carbon [to absorb water vapour].** SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIÈRES (B.P. 357,812, 17.10.30. Fr., 25.11.29).—The C is



impregnated with a solution of a dehydrating salt, *e.g.*,  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{K}_2\text{CO}_3$ , or an org. hygroscopic liquid, *e.g.*, glycerin, and dried at 150–200°. After use it may be reactivated by heating at 150–200°. D. K. MOORE.

**Manufacture of products from carbonaceous materials.** H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 358,180, 29.7.30. Cf. B.P. 353,506; B., 1931, 872).—Colloidal Re compounds (*e.g.*, sulphide) on suitable carriers, which may be active C or fine coal, are used as catalysts. D. K. MOORE.

**Rotary-grate gas generators.** M. ORB (B.P. 357,768, 20.9.30).—The generator has a stepped rotary ash pan filled with  $\text{H}_2\text{O}$  and rotating with the grate. The ash is conveyed from the interior of the shaft to the annular surface forming the outer step, by means of vanes rotating with the grate and also by stationary guide scoops. The ash is finally removed from the outer step by means of a stationary scoop. A. B. MANNING.

**Gasification of solid fuels.** SOC. OXYTHERMIQUE, Assees. of M. FRANKL (B.P. 357,853, 20.11.30. Ger., 2.12.29).—Steam is introduced alternately into the top of each of two producers side by side and connected at the bottom. The gas formed in the first producer flows upward through the second, heating the fuel by its sensible heat.  $\text{O}_2$  is passed through a thermostatically controlled valve into the gas-forming producer when the temp. of the fuel in it falls below 800°.

D. K. MOORE.

**Separation of naphthalene from coal-distillation gases.** GAS CHAMBERS AND COKE OVENS, LTD. From F. J. COLLIN A.-G. (B.P. 357,847, 15.11.30).—Warm coal gas passing up a tower vaporizes a solvent for  $\text{C}_{10}\text{H}_8$ , *e.g.*, tar oil, introduced as a spray about half-way up the tower. In the upper part the gas and vapour meet a spray of  $\text{H}_2\text{O}$ , which condenses the vapours, and this liquid dissolves the  $\text{C}_{10}\text{H}_8$ .

D. K. MOORE.

**Final cooling of gases from coal-distillation ovens and the like.** KOPPERS COKE OVEN CO., LTD. From H. KOPPERS A.-G. (B.P. 357,801, 9.10.30).—A  $\text{H}_2\text{O}$ -insol. oil in which  $\text{C}_{10}\text{H}_8$  is sol., *e.g.*, petroleum, is used instead of tar (cf. B.P. 336,484; B., 1931, 102) for washing the cooling water.

D. K. MOORE.

**Enriching a lean combustible gas.** H. A. BRASSERT and C. W. ANDREWS, Assrs. to H. A. BRASSERT & CO. (U.S.P. 1,795,829, 10.3.31. Appl., 31.12.23).—The gas together with a controlled proportion of air is passed through a mass of partly consumed coke at high temp., and then through a mass of coke at a higher temp. (sufficient to convert  $\text{CO}_2$  into CO), while a controlled quantity of preheated air is introduced laterally into the coke; the gas is then passed through a mass of coal at a lower temp.

H. S. GARLICK.

**Conversion of gaseous hydrocarbons into hydrogen.** UNION CHIM. BELGE SOC. ANON. (B.P. 357,956, 29.5.30. Belg., 31.8.29).— $\text{CH}_4$  is partly converted into  $\text{H}_2$  by the following catalytic endothermic reactions:  $\text{CH}_4 + 2\text{H}_2\text{O} = \text{CO}_2 + 4\text{H}_2$ ;  $\text{CH}_4 + \text{H}_2\text{O} = \text{CO} + 3\text{H}_2$ . The gases are heated between their successive passages over various layers of catalyst and the apparatus is heated externally. A mol. excess of steam relatively to the hydrocarbons is used.

D. K. MOORE.

**Distillation of tar.** BARRETT CO., Assees. of S. P. MILLER (B.P. 357,885, 22.12.30. U.S., 28.12.29).—Steam, superheated to at least 500°, is brought into direct and intimate contact with a spray of tar of such concentration that the steam is almost instantly cooled to a temp. below that at which objectionable decomp. of the tar occurs. The spray is also so directed as to keep the walls of the still flushed with tar. The steam and oil vapours, and the residual high-m.p. pitch, are removed continuously from the still. The vapours are condensed, their heat content being utilised in pre-heating and partly distilling the tar supplied to the still.

A. B. MANNING.

**Coking of pitch.** BARRETT CO., Assees. of M. L. HAMLIN (B.P. 358,375, 22.12.30. U.S., 28.12.29).—The violent ebullition and foaming which may cause blockage of the retort outlets during coking are prevented by adding the pitch in portions and waiting until the ebullition of one portion has practically subsided before the next is added.

D. K. MOORE.

**Production of low-boiling hydrocarbons from tars, oils, etc.** E. G. BROWN, H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 357,589, 25.6.30).—Hydrocarbons boiling up to 200° are obtained by hydrogenating the vapours of tar, phenol, etc. under pressure at 400–600°, using  $\text{Fe}_3\text{O}_4$  with or without the addition of Sn, Mo, W, Cr, or V as catalyst.

D. K. MOORE.

**Production of hydrocarbons of low b.p. from carbonaceous materials.** V. MONCADA (B.P. 358,124, 8.4.30).—The gas obtained by carbonising coal, wood, tar, or oil after passing in turn through a dephlegmator maintained at 400° (in which dust is removed and other products are condensed), a purifier containing coils of Ni, Cu, or Fe wire to remove S, and at 350–400° over a reducing mixture of finely-divided metal and C to reduce some of the O compounds, *e.g.*, PhOH, is hydrogenated by passing over coils of Fe, Ni, Co, or V wire at 180–250°. After condensation, in which  $\text{H}_2\text{O}$  containing  $\text{NH}_3$  etc. and hydrocarbon oils are condensed, the gas is washed with  $\text{H}_2\text{O}$  containing  $\text{SO}_2$ , obtained by regeneration of the S purifiers, to remove  $\text{NH}_3$ . It is then passed over activated C to remove light hydrocarbon oils.

D. K. MOORE.

**Retort for treatment of oil shales, bitumens, hydrocarbons, etc.** A. STEWART (U.S.P. 1,800,366, 14.4.31. Appl., 13.3.28).—A cylindrical retort having a conical bottom provided with a central outlet aperture and a gas outlet at the top is placed vertically in a stack heated from below. Secured to the wall of the retort is a series of vertically disposed zig-zag chutes. The retort is heated by means of a central pipe extending throughout its length having a heat-resisting filling and horizontal heat-deflecting elements thereon. Material enters the chutes through hoppers, and residue is recovered and discharged from the bottom of the retort.

H. S. GARLICK.

**Apparatus for cracking and distilling hydrocarbons.** J. H. ANDERSON, Assr. to ALTITHERMO ENG. Co. (U.S.P. 1,800,672, 14.4.31. Appl., 8.10.28).—A conical, metallic retort having a smooth and unobstructed inner surface is rotatably supported within a heating

chamber with its axis inclined downwardly towards its smaller end and with the lowest longitudinal line of its conical wall in a substantially horizontal position. Hollow, inclined, axial trunnions open out of the retort at its opposite ends and project through the opposite end-walls of the heating chamber, and are connected respectively to means for supplying the material to be treated and a receiver for the products of the operation. A bed of loose metal bodies extends the length of the retort, a screen preventing their discharge through the lower trunnion, but permitting the free flow of the products.

H. S. GARLICK.

**Conversion of petroleum oil.** R. CROSS, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,796,507, 17.3.31. Appl., 9.7.24).—Oil is raised to conversion temp. without substantial cracking and is passed to a vaporising chamber, heavily lagged to prevent heat losses, where it collects in considerable quantity. Lighter fractions are distilled off, passed to a dephlegmator, and the escaping vapours condensed and collected. Unvaporised products are directed to a stagnant pool which can be heated to facilitate settling of the free carbonaceous material. The heavy settlings from the bottom of the pool and the liquid, relatively C-free oil from a vertical restricted column in the pool, are separately withdrawn, whereby turbulency in the main body of oil is prevented.

H. S. GARLICK.

**Treatment of petroleum hydrocarbons.** W. M. CROSS, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,800,436, 14.4.31. Appl., 27.2.26).—Oil is raised to cracking temp. in a heating stage and passed to a conversion stage where temp. and pressure conditions are maintained to permit cracking without substantial distillation; it is thereafter passed to an evaporation stage wherein a portion of the cracked product is distilled. The residual oil is passed to a separate still. The lighter fractions are removed and condensed and then returned through the heating stage for re-treatment; the residual fluid is passed to the burners of the heating stage and back to the still in a closed cycle, sufficient being withdrawn for fuel purposes.

H. S. GARLICK.

**Treatment of petroleum products.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,800,823, 14.4.31. Appl., 24.3.27).—Sludge acid after separation from acid-treated oil and substantially free from extraneous moisture is subjected to the action of an acid-resisting centrifuge, whereby acid and oil are separated from the sludge and may be collected separately.

H. S. GARLICK.

**Breaking of petroleum emulsions.** J. HYMAN and A. F. SCHLANDT, Assrs. to PURE OIL Co. (U.S.P. 1,800,887, 14.4.31. Appl., 26.3.29).—The emulsion is treated first with  $\text{SO}_2$  and subsequently with  $\text{H}_2\text{S}$ , then heated, and set aside to allow the constituents to separate.

H. S. GARLICK.

**Breaking of petroleum emulsions.** C. H. M. ROBERTS, Assr. to TRETOLITE Co. (U.S.P. 1,802,090, 21.4.31. Appl., 24.1.27).—The emulsion is pumped through a flow-line into which is introduced an inert gaseous medium.  $\text{H}_2\text{O}$  is added and the mixture passed through a homogeniser. A chemical demulsifier is

subsequently added to the emulsion as it travels through the flow-line into a settling tank.

H. S. GARLICK.

**Refining of hydrocarbon oils.** A. S. RAMAGE, Assr. to GYRO PROCESS Co. (U.S.P. 1,796,621, 17.3.31. Appl., 27.8.26).—S and Cl are removed from aromatic, olefinic, and naphthenic hydrocarbons, and mixtures thereof, which have been pretreated with an agent of the group comprising  $\text{H}_2\text{SO}_4$  and active Cl-compounds, by dissolving such hydrocarbons in an EtOH solution of an alkali hydroxide, permitting the solution to stratify, and separately removing the purified oil and the EtOH-alkaline solution containing the impurities.

H. S. GARLICK.

**Production of motor fuel.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,800,822, 14.4.31. Appl., 17.3.26).—A non-detonating fuel for high-compression motors consists of 15–20% of a pressure distillate produced by cracking low-temperature coal tar blended with 85–80% of a straight-run or cracked gasoline, or both, obtained from petroleum.

H. S. GARLICK.

**Manufacture of non-detonating motor fuels.** A. B. BROWN and F. W. SULLIVAN, JUN., Assrs. to STANDARD OIL Co. (U.S.P. 1,797,819, 24.3.31. Appl., 12.11.25).—A high-boiling hydrocarbon is subjected to conversion temp. ( $> 370^\circ$ ) under pressure in the presence of a nitrogenous org. compound of the substituted- $\text{NH}_3$  type, e.g., urea, dicyanodiamide, guanidine, thereby producing cyclic N compounds in the oil and a fuel product of the gasoline range, which is distilled from the reaction mass.

H. S. GARLICK.

**Lubricating oils.** STANDARD OIL DEVELOPMENT Co., Assees. of E. M. CLARK (B.P. 357,717, 9.8.30. U.S., 5.10.29. Addn. to B.P. 349,342; B., 1931, 832).—Oils produced by the process of the prior patent are characterised by the following properties: the viscosity (Saybolt) in sec. at  $210^\circ \text{F.}$  ( $V_{210}$ ) is greater than that indicated by the relation  $V_{100} = 0.0758(2/V_{210}) + 12.56(V_{210}) - 524.6$ ; the Conradson value (% C) is  $< 0.0047(V_{210}) - 0.15$ ; the flash pt. ( $^\circ \text{F.}$ ) is higher than  $V_{210} + 410$ ; low pour point and high sp. gr.; high resistance to oxidation. The products may be blended with straight mineral lubricating oils.

A. B. MANNING.

**Treatment of lubricants.** W. O. STEVENS, Assr. to ANTI HOT BOX Co., INC. (U.S.P. 1,796,310, 17.3.31. Appl., 15.6.29).—Wood ashes, preferably from hardwood, with or without the addition of a relatively small quantity of bone char, are thoroughly dried and placed in an oil-pervious container which is immersed in the oil to be treated (using about 1 lb. of ashes to 5 gals. of oil) for sufficient time to allow thorough penetration.

H. S. GARLICK.

**Compression of coal cakes.** N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., G.M.B.H. (B.P. 358,981, 16.7.30. Ger., 16.7.29).

**Gas burners.** GAWA PATENTVERWALTUNGS A.-G. (B.P. 358,821, 24.12.30. Ger., 13.2.30).

**Anti-incrustation briquettes for boilers etc. Pulverising installation. Disintegrating materials. Sound-deadening compositions.**—See I.

Dispersions. Oxidation of org. compounds.—See IH. CS<sub>2</sub>.—See VII. Asphalt-coated metal.—See X. Dynamo brush.—See XI.

### III.—ORGANIC INTERMEDIATES.

**Formation of vanillin from sulphite-cellulose waste liquors and its gravimetric determination.** M. HÖNIG and W. RUZICZKA (Z. angew. Chem., 1931, 44, 845—847).—Sulphite-cellulose waste liquors, when boiled with NaOH, give vanillin, which, after partial purification, can be determined as *m*-nitrobenzoylhydrazide. The yield is greatly decreased by passage of air during boiling (owing to oxidation) (cf. B., 1928, 292), and greatly increased by heating for 3 hr. at 160—170°/5—6 atm. R. S. CAHN.

**Recovery of volatile solvents.** THORMANN (Zentr. Gewerbehyg. Unfallverhüt., 1931, 18, 67—75; Chem. Zentr., 1931, i, 3032).—Dangers and precautions are discussed. A. A. ELDRIDGE.

See A., Nov., 1245 and 1248, Autoxidation of PhCHO. 1248, Dissociation of BzOH vapour. 1249, Condensation of hydrocarbons electrically. 1272, Prep. of Et oxalate. 1273, Polymerisation of MeCHO. Detection of COMe<sub>2</sub> in presence of MeCHO. 1282, 2 : 3 : 6 : 7-Tetramethylantracene. 1286, Hydrogenation of PhOH. Phenols of coal tar. 1288, Separation of cresols. 1293, Prep. of phenylglyoxal. 1296, 1-Methylantraquinone series. 1309, Reduction products of pyocyanine etc. dyes. 1333, Formation of citric acid by moulds. 1334, Bacteria producing trimethylene glycol. Lactic-acetic fermentation.

#### PATENTS.

**Production of acetaldehyde from methane and carbon dioxide.** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 353,455, 23.4.30. Ger., 29.4.29).—CO<sub>2</sub> and excess CH<sub>4</sub> are led through a high-frequency field of above 10,000 periods/sec. and above 20,000 volts, with or without catalysts, *e.g.*, quartz glass. The conversion is about 40%. C. HOLLINS.

**Manufacture of acetic anhydride.** I. G. FARBENIND. A.-G. (B.P. 353,688, 21.7.30. Ger., 1.8.29).—AcOH vapour is led into a molten phosphate, heat being supplied electrically, *e.g.*, by a current between the lead-in tube and the containing vessel, which are made of Cu, Cr-Ni, etc. C. HOLLINS.

**Manufacture of acetic anhydride.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 354,159, 6.2.31).—AcOH and AcCl or SOCl<sub>2</sub> are introduced into a column attached to a still containing boiling Ac<sub>2</sub>O. Ac<sub>2</sub>O condenses and is removed from the still by an overflow. The exit gases are washed to remove excess AcCl. C. HOLLINS.

**Manufacture of aldols.** T. H. DURRANS and F. W. LEWIS (B.P. 353,413, 21.3.30).—MeCHO and H<sub>2</sub>O are added to anhyd. Na<sub>2</sub>CO<sub>3</sub> at 15°, or are percolated through the Na<sub>2</sub>CO<sub>3</sub>; aldol is isolated by extraction or distillation. The H<sub>2</sub>O must not exceed 25% of the wt. of Na<sub>2</sub>CO<sub>3</sub>. C. HOLLINS.

**Manufacture of iodomethanesulphonic acid, its homologues and their salts.** A. CARPMAEL. From

I. G. FARBENIND. A.-G. (B.P. 353,477, 24.4.31. 5, 11.7, 12.7., and 7.11.30).—CH<sub>2</sub>I<sub>2</sub>, CHI<sub>3</sub>, or homologues are heated with aq. neutral sulphite, or chloro- or bromomethanesulphonic acid or homologues with an iodide in H<sub>2</sub>O or an org. solvent, to give iodoalkylsulphonic acids, the salts of which are contrast media for X-ray photography. Na iodomethanesulphonate is obtained in 95% yield from CH<sub>2</sub>I<sub>2</sub> and aq. Na<sub>2</sub>SO<sub>3</sub> at 70°; the methylamine salt, m.p. 144—145°, is similarly prepared; ethylidene iodide gives Na  $\alpha$ -iodoethane- $\alpha$ -sulphonate. Diethylamine iodomethanesulphonate is prepared from Mn chloromethanesulphonate and NHEt<sub>2</sub>.HI in H<sub>2</sub>O at 180—200°. Na  $\alpha$ -iodopropane- $\alpha$ -sulphonate is also described. C. HOLLINS.

**Manufacture of aliphatic ketones.** BRIT. CELANESE, LTD., H. F. ONLEY, W. H. GROOMBRIDGE, and E. B. THOMAS (B.P. 353,467, 16.4.30).—In the conversion of C<sub>2</sub>H<sub>2</sub>, aliphatic alcohols, aldehydes, or esters by steam into aliphatic ketones there is used a catalyst prepared by heating, *e.g.*, at 1400—1600° for 1 hr., an alkaline-earth oxide with a weak acidic oxide (Al<sub>2</sub>O<sub>3</sub>, ZnO, or SiO<sub>2</sub>) to give a hydraulic product which is powdered and caused to set by addition of H<sub>2</sub>O. The oxides may be generated *in situ* by heating suitable compounds, or may be co-precipitated, *e.g.*, by adding aq. NaOH to a 10% solution of Ca(NO<sub>3</sub>)<sub>2</sub> and Zn(NO<sub>3</sub>)<sub>2</sub>. With a CaO-ZnO "cement" catalyst at 465—480° steam and EtOH give 60—70% yield of COMe<sub>2</sub>. C. HOLLINS.

**Manufacture of alcohols.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,047, 10.4.30).—Hydrocarbons of b.p. above 180° are air-oxidised in liquid phase at atm. pressure in presence of at least 2% of acids of dissociation const.  $2.5 \times 10^{-4}$  to  $2 \times 10^{-13}$  until the sap. val. reaches 70. *E.g.*, air laden with 22 g. of AcOH per cu. in. is blown through hard paraffin wax at 180° for 3 hr. to give a 40% conversion into alcohols. Other suitable compounds are H<sub>3</sub>BO<sub>3</sub>, Bz<sub>2</sub>O, Sb<sub>2</sub>O<sub>5</sub>; Mn naphthenate may also be present. C. HOLLINS.

**Manufacture of polymerised vinyl alcohols.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,866, 24.3.30).—A vinyl ester is heat-polymerised in presence of org. or inorg. per-compounds (NaBO<sub>3</sub>, Bz<sub>2</sub>O<sub>2</sub>, BaO<sub>2</sub>, etc.) and org. acid anhydrides (Ac<sub>2</sub>O), in absence of solvents, and the product is hydrolysed, *e.g.*, with EtOH and H<sub>2</sub>SO<sub>4</sub>. C. HOLLINS.

**Production of dispersions.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,926, 31.1. and 29.8.30).—Viscous or solid polymerides of alkylene oxides are used as dispersing agents. Dispersions in H<sub>2</sub>O of bleached montan wax, gum mastic, pyroxylin lacquers, castor oil, tritolyl phosphate, ozokerite with montan wax, stearin, and nigrosine (for boot polish), montan wax with carnauba wax and castor oil (for floor polish), wool grease with H<sub>2</sub>BO<sub>3</sub> and glycerol (for salves), green earth with fuchsin (for lime distemper), BaSO<sub>4</sub> with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, BaCl<sub>2</sub>, and Hansa-green G [precipitated with BaCl<sub>2</sub> and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> for a colour lake], Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> with Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>HPO<sub>4</sub>, Alizarin-red, and Turkey-red oil [precipitated with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and BaSO<sub>4</sub> for wallpaper paint], Para-red, and Al powder are described. C. HOLLINS.

**Preparation of sulphonic acids of the aliphatic series [wetting agents].** H. T. BÖHME A.-G. (B.P. 353,475, 24.4.30. Ger., 3.5.29).—An  $\alpha$ -brominated aliphatic acid above  $C_5$  ( $\alpha$ -bromolauric acid) is boiled with alkali sulphite, e.g.,  $(NH_4)_2SO_3$ , and the hot solution is acidified with HCl to ppt. the oily sulphonic acid.

C. HOLLINS.

**Photochemical oxidation of organic and inorganic compounds.** I. KREIDL, C. ROSEN, and K. RUTTER (VEREIN. CHEM. FABR. KREIDL, HELLER, & Co.), Assees. of F. NOZICKA (B.P. 354,096, 12.9.30. Austr., 19.10.29).—The oxidation by irradiation in presence of anthraquinone as catalyst (Eckert) is assisted or continued by means of other catalysts. PhMe in presence of anthraquinone and  $Ca(OH)_2$  and in contact with  $O_2$  gives on insolation BzOH; fatty acids are obtained from paraffin oil, cinchoninic acid from 4-methylquinoline,  $CrO_3$  from  $Cr_2(SO_4)_3$ , and PhCl from  $C_6H_6$  and HCl.

C. HOLLINS.

**Synthesis of aromatic compounds [acids, carbamides, and ketones from phenols or amines and carbon dioxide] at high pressure.** G. T. MORGAN and D. D. PRATT (B.P. 353,464, 20.3.30).—Phenols or arylamines react with  $CO_2$  at 100–200°/40–150 atm. in presence of  $AlCl_3$ ,  $ZnCl_2$ ,  $FeCl_3$ , etc. to give carboxylic acids, carbamides, or benzophenones. Examples are the conversion of: PhOH into salicylic acid at 100°/90 atm., into 4:4'-dihydroxybenzophenone at 105–110°/70 atm., into the 2:4'-compound at 150°/70 atm., into  $Ph_2O$  at 200°/120 atm.; *o*-cresol into 2:4'-dihydroxy-3:5'-dimethylbenzophenone at 150°/70 atm.; *m*- and *p*-cresols into cresotic acids at 150°/70 atm.;  $NH_2Ph$  into carbamides at 130°/60 atm.;  $NHPhEt$  into *N*-ethylecarbamides at 110–150°/60 atm. or into *p*-ethylaminobenzoic acid with  $ZnCl_2$  at 150°/70 atm.;  $NPhMe_2$  into *p*-dimethylaminobenzoic acid with  $ZnCl_2$  at 150°/70 atm., or into Michler's ketone etc. with  $AlCl_3$  at 150°/65 atm.

C. HOLLINS.

**Production and purification of salicylic or o-cresotic acid or their salts.** GRAESSER-MONSANTO CHEM. WORKS, LTD., R. GRAESSER-THOMAS, and E. MATHER (B.P. 353,921, 29.4.30).—The crude aq. Na salicylate is seeded below 20° to give the hexahydrate, which is then dehydrated, if desired after recrystallisation. Na *o*-cresotate is cryst. as trihydrate below 50°.

C. HOLLINS.

**Manufacture of thiazole compounds.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 353,577, 16.5.30. U.S., 18.5.29).—A thiocarbamide,  $ArNH\cdot CS\cdot NRR'$ , in which Ar = aryl with a free *o*-position, R = H, alkyl, or aryl, and R' = H or alkyl, is treated with  $SO_2Cl_2$  in an org. diluent ( $PhCl$  or  $C_2H_4Cl_2$ ) to give a 2-amino- or substituted 2-amino-arylenethiazole. In the examples, Ar = *p*-EtO- $C_6H_4$ , *o*- and *p*- $C_6H_4Me$ , 5-chloro-*o*-tolyl, Ph,  $\alpha$ - $C_{10}H_7$ ; R = H; R' = H, *o*- $C_6H_4Me$ , and Me.

C. HOLLINS.

**Manufacture of arylsulphonic esters of 2:8-dihydroxynaphthalene-6-sulphonic acid.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 353,578, 16.5.30).—2:8-Dihydroxynaphthalene-6-sulphonic acid is treated with an arylsulphonyl chloride. With rather more than 1 mol. and corresponding alkali the 8-ester is formed, which gives the di-ester on further esterifica-

tion. Hydrolysis of di-ester yields the 2-ester. The 8-ester is also prepared from  $\gamma$ -acid or an *N*-acyl- $\gamma$ -acid.

C. HOLLINS.

**Manufacture of acridine derivatives.** A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 353,537, 30.4.30).—7:10-Dihalogeno-2-alkoxyacridines are synthesised by condensing a 2:4-dihalogenobenzoic acid with a 4-alkoxyaniline, acridonating the product, and finally treating with  $PCl_5$ ,  $POCl_3$ ,  $PBr_5$ , or P and I, preferably in an org. solvent at raised temp. Examples are: 3'-chloro-4-methoxydiphenylamine-6'-carboxylic acid, m.p. 214–216°, cyclised to acridone, m.p. above 270°, and treated with  $PCl_5$  in  $PhCl$  to give 7:10-dichloro-2-methoxyacridone, m.p. 160–161°, or with P and I in  $PhCl$  for the 7-chloro-10-iodo-compound, m.p. 211–212°, or with  $PBr_5$  for the 7-chloro-10-bromo-compound, m.p. 181–182°; 7:10-dichloro-2-ethoxyacridine, m.p. 162–163°, from 3'-chloro-4-ethoxydiphenylamine-6'-carboxylic acid, m.p. 224–225°, via the acridone, m.p. above 270°; 10-chloro-7-bromo-2-methoxyacridone, m.p. 160–161°, from 3'-bromo-4-methoxydiphenylamine-6'-carboxylic acid, m.p. 212–213° (acridone, m.p. above 270°); 10-chloro-7-iodo-2-methoxyacridone, m.p. 164–165°, from 3'-iodo-4-methoxydiphenylamine-6'-carboxylic acid, m.p. 219–220° (decomp.) (acridone, m.p. above 270°).

C. HOLLINS.

**Manufacture of hydroxylated anthrones.** IMPERIAL CHEM. INDUSTRIES, LTD., A. G. PERKIN, and E. J. CROSS (B.P. 353,479, 24.1.30).—A polyalkoxyanthraquinone containing an  $\alpha$ -alkoxyloxy-group is subjected to simultaneous reduction and complete deacylation, e.g., with  $SnCl_2$  and  $HCl$ . 1-Hydroxylated 10-anthrone is obtained.

C. HOLLINS.

**Manufacture of benzanthrone.** I. GUBELMANN, R. J. GOODRICH, and E. T. HOWELL, Assrs. to NEWPORT CHEM. CORP. (U.S.P. 1,791,309, 3.2.31. Appl., 6.2.28).—An anthraquinone is reduced to the anthranol in  $H_2SO_4$  with a Cu salt and sufficient Zn or other more electropositive metal to displace all the Cu from solution. The Cu is used up as it is precipitated.

C. HOLLINS.

$C_{10}H_8$  from coal gas.—See II. [AcOH from] manufacture of cellulose acetate.—See V. Fatty acid derivatives.—See XII. Diaryl resins.—See XIII.

#### IV.—DYESTUFFS.

Monel metal [vats].—See X.

See also A., Nov., 1285, New azo dyes. 1291, [Dyes from] cyclic acid imides. 1295, Org. S-N linking. 1309, Reduction products from pyrocyanine etc. dyes.

PATENTS.

**Manufacture of anthraquinone derivatives.** A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 353,932, 24.4.30).—A leuco- $\alpha$ -hydroxyanthraquinone (which may be produced *in situ*) is condensed in aq. suspension or solution with  $NH_3$  or an alkyl- or aralkylamine in presence of  $H_3BO_3$ .  $\beta$ -Sulphonic groups are eliminated during reaction. 4:8-Diaminoanthrarufin-2:6-disulphonic acid with  $Na_2S_2O_4$ ,  $H_3BO_3$ , and aq.  $NH_3$  gives 4:5:8-triamino-1-hydroxyanthraquinone

(blue on acetate silk). 1 : 8-Diamino-5-methylamino-4-hydroxyanthraquinone (green-blue), the corresponding 4 : 8 : 5 : 1-compound (red-blue), and a product from leuco-1 : 4 : 5 : 8-tetrahydroxyanthraquinone and  $\beta$ -hydroxyethylamine (blue-green) are also described.

C. HOLLINS.

**Manufacture of derivatives of naphthazarin.** I. G. FARBERIND. A.-G. (B.P. 354,158, 24.11.30. Ger., 23.11.29. Addn. to B.P. 316,950; B., 1931, 150).—The naphthazarin intermediate of the prior patent is condensed in  $\text{H}_2\text{SO}_4$ , without isolation, with an aliphatic aldehyde (paraformaldehyde). A black pigment separates on dilution.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** I. G. FARBERIND. A.-G. (B.P. 349,713—4, [A] 8.4.30, [B] 9.4.30. [A, B] Ger., 9.4.29).—(A) An *o*-halogenoanthraquinonecarboxylic ester or acid is condensed with a carbazolated amino- or diamino-dianthraquinonylamine, and the product is acridonated. Examples are: carbazole from 4-amino- or 4 : 4'-diamino-1 : 1'-dianthraquinonylamine with Me 2-bromoanthraquinone-1-carboxylate, acridonated (olive-green); carbazole from 5 : 5'-diamino-1 : 1'- or -1 : 2'-dianthraquinonylamine with Me 1-chloroanthraquinone-2-carboxylate, acridonated (black-brown); carbazole from 5 : 5'-diamino-1 : 1'-dianthraquinonylamine with Me 2-chloroanthraquinone-3-carboxylate (red-brown). (B) Carbazoles from 4- or 5-( $\beta$ -anthraquinonylcarboxylamido)dianthraquinonylamines are synthesised. Examples are: 4 : 4'-diamino-1 : 1'-dianthraquinonylamine condensed with anthraquinone-2-carboxyl chloride and carbazolated (yellow-olive); carbazole from 5 : 5'-diamino-1 : 1'- or 1 : 2'-dianthraquinonylamine condensed with anthraquinone-2-carboxyl chloride (orange-yellow); 5-chloro-1-aminoanthraquinone condensed first with anthraquinone-2-carboxyl chloride or its 1-hydroxy-compound, then with 4-amino-1-benzamidoanthraquinone and carbazolated (brown); 1-nitro-4-aminoanthraquinone condensed with anthraquinone-2-carboxylic acid, reduced, condensed with 1-chloro-4-benzamidoanthraquinone, and carbazolated (olive).

C. HOLLINS.

**Manufacture of vat dyes of the isodibenzanthrone series containing chlorine and/or bromine.** I. G. FARBERIND. A.-G. (B.P. 353,886, 25.1.30. Ger., 27.4.29. Cf. B.P. 351,948; B., 1931, 1041).—*iso*Dibenzanthrone or a Cl- or Br-derivative is halogenated with chlorides or bromides of Fe, Al, Sb, Hg, etc., preferably with addition of a flux (NaCl). Solvents, catalysts, and further halogenating agents may be present. Brominated isodibenzanthrone with  $\text{FeCl}_3$  in  $\text{PhCl}$  at 140—150°, and isodibenzanthrone with  $\text{FeCl}_3$ , NaCl, I, and Br at 80—160°, give violet vat dyes; tetrabromo*iso*dibenzanthrone with  $\text{FeCl}_3$  in  $\text{PhNO}_2$  at 145—150° a navy-blue.

C. HOLLINS.

**Manufacture of vat dyes [from alizarin].** IMPERIAL CHEM. INDUSTRIES, LTD., A. G. PERKIN, A. SHEPHERDSON, and N. H. HADDOCK (B.P. 353,422, 17.4.30).—The dihydroxyanthranol obtained by reduction of alizarin is condensed with a tetrahalogenothiophen, e.g., in boiling  $\text{C}_{10}\text{H}_8$ , with or without simultaneous alkylation, e.g., with  $\text{Me}_2\text{SO}_4$ . A partial alkylation may precede the condensation. The product from reduced alizarin,

tetrachlorothiophen,  $\text{Na}_2\text{CO}_3$ , and  $\text{Me}_2\text{SO}_4$  in  $\text{C}_6\text{H}_3\text{Cl}_3$  at 190—200° is an emerald-green vat dye.

C. HOLLINS.

**Manufacture of water-insoluble azo dyes [ice colours and pigments].** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 353,538, 30.4.30).—A non-sulphonated diazo compound is coupled in substance or on the fibre with an arylamide of a hydroxyphenanthrene-*o*-carboxylic acid. Examples are: 2-hydroxyphenanthrene-3-carboxylic *o*-toluidide with diazotised 2 : 5-dichloroaniline (yellow-red), *p*-nitroaniline (brown), *m*-toluidine (brown); the 3-hydroxy-2-carboxylic *o*-toluidide with diazotised 5-nitro-*o*-anisidine (green-black), 2- or 3-aminocarbazole (black), *o*-nitroaniline (brown); and the *p*-anisidide with diazotised 5-nitro-*o*-anisidine (red-black), 4-chloro-*o*-nitroaniline (brown), or 2 : 5-dichloroaniline (bordeaux-red).

C. HOLLINS.

**Manufacture of metalliferous [azo] dyes [for silk].** SOC. CHEM. IND. IN BASLE (B.P. 353,411, 15.3.30. Switz., 16.3.29).—5-Nitro-*o*-aminophenol is diazotised and coupled alkaline with *S*-acid or an *N*-acyl-*S*-acid, and the product is pre-coppered (etc.). Silk, weighted or not, is dyed green-blue shades from a neutral or fatty soap bath.

C. HOLLINS.

**Manufacture of mordant [tris- and poly-azo] dyes.** DURAND & HUGUENIN A.-G. (B.P. 349,609, 28.2.30. Ger., 1.3.29).—Aminoazo dyes, having a salicylic acid as end-component coupled with *p*-phenylenediamine or through *p*-phenylenediamine (with or without another middle component of the  $\text{C}_6\text{H}_6$  series) with an arylamine of the  $\text{C}_6\text{H}_6$  series, are oxidised, preferably in alkaline medium, to convert the  $\text{NH}_2$  into an azo group. Examples are: *p*-aminobenzeneazosalicylic acid with aq. NaOH and  $\text{CaOCl}_2$ , sulphonated with 20% oleum at 100—110° (chrome, brown-yellow); 4-amino-2-sulphobenzeneazosalicylic acid with aq. NaOH and  $\text{CaOCl}_2$ , or  $\text{KMnO}_4$  (chrome, brown); *p*-nitroaniline-*o*-sulphonic acid  $\rightarrow$  aniline (or metanilic acid)  $\rightarrow$  salicylic acid, reduced, oxidised (chrome, deep-brown).

C. HOLLINS.

**Dispersions.**—See III.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Cellulose acetate from wood cellulose.** F. OHL (Kunststoffe, 1930, 20, 217—219; Chem. Zentr., 1931, i, 2700).—The yield and quality of the product from sulphite-cellulose are improved by pretreatment with acids, or preferably alkalis. The solubility of a cellulose in  $\text{H}_2\text{SO}_4$  is a measure of its ready and regular acetylation, which depends on the surface condition and mechanical separation of the fibres. A. A. ELDRIDGE.

**Microchemical detection of minute quantities of perchloric acid [in cellulose acetate].** D. KRÜGER and E. TSCHIRCH (Z. anal. Chem., 1931, 65, 171—176).—The characteristic blue needles which are formed when methylene-blue is added to  $\text{HClO}_4$  serve to detect traces of this acid in cellulose acetate in the manufacture of which  $\text{HClO}_4$  is used as a promoter. The ester is hydrolysed with NaOH in 75% EtOH and the solution neutralised with AcOH. After filtration the solution is evaporated to dryness and the residue moistened with  $\text{H}_2\text{O}$ . A few drops of the liquid are treated with

methylene-blue on a microscope slide; needles separate if as little as 0.0005 mg. of  $\text{HClO}_4$  is present in 1 drop of solution, as the saturated  $\text{NaOAc}$  solution has a salting-out action on methylene-blue perchlorate.

A. R. POWELL.

**Vanillin from sulphite-cellulose waste liquors.**—See III. "Black liquor."—See VII.

See also A., Nov., 1230, Osmometer for lyophilic colloids. 1231, Cellulose derivatives in org. solvents. 1278, Production of highly saponified cellulose acetates. 1317, Basic amino-acids in silk fibroin.

#### PATENTS.

**Extraction of textile fibres from fibrous vegetable materials such as Spanish broom and the like.** C. CALLONI (B.P. 358,143 and 358,160, [A] 10.7.30, [B] 17.7.30. Ital. [A] 10.7.29).—The stalks of the plant, after softening or loosening of the cortex by treatment in a bath which, in (B), is kept in circulation first in one direction and at intervals in the opposite direction, are held firm and subjected to the action of a jet of (A) steam or (B) compressed air, which strikes them end-wise and strips the rind cortex from the end of the stalks; the removal of the fibres from the wood is completed by stripping under  $\text{H}_2\text{O}$ . F. R. ENNOS.

**Production of a horse-hair substitute, particularly for stuffing purposes.** E. ELÖN (B.P. 356,754, 6.6.30. Ger., 4.1.30).—Hard vegetable fibres (coconut, agave, etc.), after freeing from embedding substances, are treated with dil. alkali (2–3%) for 2–3 hr. at 75–100°, washed, dried, and then further treated with hygroscopic and/or oily substances (glycerin,  $\text{MgCl}_2$ , emulsions of fatty acids) and dyed if desired.

F. R. ENNOS.

**Treatment of felt.** IMPERIAL CHEM. INDUSTRIES, LTD., W. ARMITAGE, and W. BAIRD (B.P. 358,104, 4.7.30).—Felt-hat shapes are stiffened and proofed by impregnating them with an alkaline solution of polyhydric alcohol-polybasic acid condensation products in the presence or absence of monohydric alcohols, monobasic acids (e.g., higher fatty acids), or fatty oils at 150° or above and containing  $\geq 40\%$  of fatty acid. A suitable solution is prepared by dissolving 25 pts. of the product obtained by heating 31 pts. of glycol, 74 pts. of phthalic anhydride, and 35 pts. of castor oil at 185°, until the product has an acid val. of 95, in 100 pts. of 0.39% aq.  $\text{NH}_3$  at 60–80° and diluting to 250 pts. The impregnated felt is then dried to volatilise the  $\text{NH}_3$ . If metal hydroxides or carbonates are used for dissolving the resin, the felt should be treated before or after impregnation, with dil. acid.

D. J. NORMAN.

**Manufacture of cellulose acetate [with recovery of acetic acid].** O. SILBERRAD and H. BLEASDALE (B.P. 357,552, 19.3.30).—Cellulose acetate is precipitated in the form of a porous, coherent, easily washed ribbon by gradually diluting the reaction mixture (after hydrolysis of the cellulose acetate to a  $\text{COMe}_2$ -sol. product) with 60–75% of that quantity of  $\text{H}_2\text{O}$  which would be required to cause the appearance of a permanent ppt. of cellulose acetate, and feeding the resulting mixture between wet rollers into a precipitating bath. If the dilution is insufficient a yellow translucent ribbon is

obtained which sticks to the rollers and is difficult to wash; if too great, the coherence of the ribbon is impaired. The process is advantageously combined with an  $\text{AcOH}$  recovery process in which the reaction mixture, after addition of sufficient  $\text{NaOAc}$  to neutralise the acid catalyst, is distilled *in vacuo*, preferably at a gradually rising temp., not above 70° and with continuous agitation, until 40–50% of the  $\text{AcOH}$  is recovered. Suitable apparatus is described.

D. J. NORMAN.

**Nitration of cellulose.** S. A. POLLOCK (B.P. 357,550, 18.3.30).—Nitrocellulose paper of uniform composition, particularly suitable for use as separators in accumulators or in the prep. of acid-resisting lacquers, is obtained by interleaving cotton paper  $< 0.01$  in. thick with thin, corrugated, sheet Al, packing these on edge in a perforated Al basket, and conducting the subsequent operations of drying, nitrating, and counter-current washing in this form.

D. J. NORMAN.

**Treatment [fractionation] of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 357,781, 24.9.30. U.S., 2.10.29).—A batch of, e.g., cellulose acetate of substantially uniform ester content is repeatedly extracted with a liquid which has a selective solvent action on the esters of lower viscosity. Thus by repeatedly extracting a cellulose acetate which is mainly sol. in  $\text{COMe}_2$  with warm  $\text{CHCl}_3$  a purified acetate is obtained which gives solutions in  $\text{COMe}_2$  of high clarity and is particularly suitable for films, laminated glass, etc.

D. J. NORMAN.

**Precipitation and treatment of cellulose derivatives.** U.S. INDUSTRIAL ALCOHOL Co. (B.P. 356,956, 26.9.30. U.S., 4.10.29).—A stream of the org. cellulose derivative (acetate) dispersed in liquid  $\text{SO}_2$  is mingled with a precipitant stream, e.g.,  $\text{H}_2\text{O}$ , in a gastight chamber containing a centrifuge, the cellulose derivative being disrupted, precipitated, and collected in the centrifuge, while the liquid and gas are conducted away by suitable outlets; subsequent treatment of the precipitated material may be carried on without opening the centrifuge.

F. R. ENNOS.

**Treatment of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 357,567, 20.6.30. U.S., 20.6.29).—The moulding or working of cellulose acetate under heat and pressure, e.g., as in the manufacture of splinterless glass, is facilitated if it is pretreated with a liquid which is not a solvent for cellulose acetate at room temp., but becomes one at higher temp. Suitable compounds include the higher alcohols, e.g.,  $\text{BuOH}$ , amyl alcohol, or cyclohexanol, and hydrocarbons of medium or low b.p., e.g., turpentine or petroleum. Small quantities of plasticising agents or compatible synthetic resins, e.g., diphenylolpropane- $\text{CH}_2\text{O}$  resins, may be added to the treating liquid.

D. J. NORMAN.

**Compositions of cellulose esters or ethers.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 358,393, 28.1.31. U.S., 28.1.30).—The pentaerythrityl ethers are used as solvents and plasticisers for cellulose esters or ethers. The various mixtures of the mono-, di-, and tri-ethyl ethers, the prep. of which is described, are pale yellow oils.

D. J. NORMAN.

**Manufacture of artificial silk.** I. G. FARBENIND. A.-G. (B.P. 356,975, 20.10.30. Ger., 18.10.29).—Freshly precipitated bundles of  $> 300$  single fibres are passed, in a parallel position and with avoidance of stretching or other mechanical stress, through washing and treating baths to the receiving device at a speed of over 50 m./min. by means of adjusted rotating guides.

F. R. ENNOS.

**Treatment of regenerated cellulose threads and apparatus therefor.** ACME RAYON CORP. (B.P. 356,737, 5.5.30. U.S., 4.5.29).—Treating liquids are forced transversely through the thread package, which is free to elongate longitudinally by a limited amount.

F. R. ENNOS.

**Washing of artificial silk.** T. F. BANIGAN, ASSR. to DU PONT RAYON Co. (U.S.P. 1,802,935, 28.4.31. Appl., 22.5.29).—The material is washed with  $H_2O$  as long as the economic removal of acid warrants, and then with a solution of a volatile compound which reacts with the acid to form  $H_2O$ -sol. salts (0.015% aq.  $NH_3$ ).

F. R. ENNOS.

**Wet treatment [washing] of artificial silk spinning cakes.** GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 358,151, 12.7.30. Addn. to B.P. 344,279; B., 1931, 437).—The cakes are separately mounted on cylindrical flanged carriers, which interengage with one another to form a column. On treatment with  $H_2O$  (etc.), the upward extension of the cakes due to the loosening of the thread layers is limited by their coming into contact with the lower surface of the flange of the next carrier above.

F. R. ENNOS.

**Drying of artificial silk cakes.** H. A. GILL. From GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 357,972, 20.6. and 20.10.30).—After being pre-dried superficially, the cakes are exposed to an air current which is forced through the thread layers.

F. R. ENNOS.

**[Fireproofing of] artificial materials containing cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 357,667, 8.7.30. U.S., 18.7.29).—Improved fire-resisting properties are imparted to films etc. made from org. derivatives of cellulose by incorporating therewith 2–25% (on the wt. of cellulose derivative) of a halogen, preferably Br, derivative of an aromatic ester of a lower fatty acid, particularly tribromophenyl acetate. Cellulose acetate films containing 3% of this compound do not discolour after prolonged exposure to ultra-violet light.

D. J. NORMAN.

**Pulp digester with interior circulation.** E. MORTERUD (U.S.P. 1,802,266, 21.4.31. Appl., 10.10.29).—A tubular heating chamber having inlet and outlet sieves at its ends runs along the inner wall of the digester and is attached thereto by means of a tubular shank, through which steam is introduced to the chamber. A reversible circulation pump draws the sieved liquor from the digester through the heating chamber in the desired direction.

F. R. ENNOS.

**Digesting system [for fibrous material].** A. F. RICHTER, ASSR. to STEBBINS ENG. & MANUFG. Co. (U.S.P. 1,804,116, 5.5.31. Appl., 12.12.29).—Towards the end of the cooking operation the gases and vapours from the treatment of fibrous material in one digester are passed

into an injector which operates to force the liquor into another digester.

F. R. ENNOS.

(A) **Preparation of fibre from wood and wood waste.** (B) **Digester.** P. G. VON HILDEBRANDT, ASSR. to H. B. SALKELD (U.S.P. 1,803,443—4, 5.5.31. Appl., 20.11.28).—(A) Wood material, after predigestion with  $Ca(OH)_2$  partly to attack the incrustants and open the grain, is further treated with dil. aq.  $NaOH$  while confined under mechanical pressure of a weighted plate in an indirectly heated digester, described in (B), to free the fibre.

F. R. ENNOS.

**Pulping of plant tissues by means of a volatile base.** R. M. HIXON, C. J. PETERSON, and C. H. WERKMAN, ASSRS. to IOWA STATE COLL. OF AGRIC. & MECH. ARTS (U.S.P. 1,802,715, 28.4.31. Appl., 30.6.30).—The plant tissue is cooked with aq.  $NH_3$  under pressure at about  $110^\circ$ , the cellulose pulp is separated, and the residual  $NH_3$  is recovered by distillation. Finally the incrustants remaining in the waste liquor are also recovered.

B. P. RIDGE.

**Production of fibre products.** G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,793,194, 17.2.31. Appl., 25.10.26).—Difficultly hydratable cellulose pulp (high- $\alpha$ -cellulose wood fibre) in the unbeaten state is mixed with a small proportion of similar beaten pulp, and made into a "drier sheet" which is then disintegrated, beaten to a slimy consistency, and run off on a paper machine.

F. R. ENNOS.

**Treating ground wood pulp.** G. A. RICHTER, ASSR. to BROWN Co. (U.S.P. 1,802,984, 28.4.31. Appl., 11.3.27).—Unused ground wood pulp is digested with 4–10%  $NaOH$  solution below  $70^\circ$  for about 1 hr. until its content of non- $\alpha$ -cellulose constituents is reduced by approx. 50%. Alternatively, a solution of  $Na_2S$  or a mixture of this with  $NaOH$  may be employed, but whilst the liquor must be sufficiently conc. to remove pentosans, lignin, etc., mercerisation of the cellulose must not occur. The product is softer and has a higher tear-resistance than pulp from untreated ground wood.

B. P. RIDGE.

**Manufacture of sulphite pulp.** A. M. THOMSEN (U.S.P. 1,803,738, 5.5.31. Appl., 24.8.29).—Part (5–50%) of the waste cooking liquor is added to fresh cooking liquor in preparing a new charge, and  $CaO$  is added to the remainder, thus precipitating a  $Ca$  salt of  $SO_2$ . The latter is washed and returned to the acid-making system, thus effecting economy in  $S$ .

B. P. RIDGE.

**Manufacture of pulp articles.** M. O. SCHUR, ASSR. to BROWN Co. (U.S.P. 1,803,410, 5.5.31. Appl., 20.2.30).—A suitable furnish for making multi-ply sheets or tubes consists of about 65% of well-pulped waste paper, 25% of well-gelatinised long-fibred pulp, and 10% of ground charcoal. The articles are subsequently impregnated with pitch or other materials.

B. P. RIDGE.

**Manufacture of impregnated fibre articles.** M. O. SCHUR, ASSR. to BROWN Co. (U.S.P. 1,803,409, 5.5.31. Appl., 30.7.29).—An aq. suspension of ground charcoal is mixed with cellulose pulp while it is being beaten, and is formed into an interfelted web which is



wound into a thick-walled tube and impregnated with pitch. F. R. ENNOS.

**Incorporating emulsified rubber into paper pulp to produce paper products.** J. A. DE CEW, Assr. to PROCESS ENGINEERS, INC. (U.S.P. 1,799,217, 7.4.31. Appl., 23.1.29).—Better results are obtained if the pulp is beaten separately and the diluted rubber emulsion added to the pulp and coagulated just before the stock reaches the paper machine. D. J. NORMAN.

**Bleaching of paper pulp and the like.** J. NEUMANN (U.S.P. 1,799,601, 7.4.31. Appl., 9.3.25. Renewed 6.8.30).—The material, *e.g.*, waste paper, is agitated for about 0.5 hr. with an alkaline de-inking liquor ( $\text{Na}_2\text{B}_4\text{O}_7$ , KOH,  $\text{Na}_2\text{CO}_3$ , and soap in admixture) at 93° to remove ink, wax, etc. and, after washing, is bleached by treatment for about 10 min. at 38° with a liquor containing NaCl and  $\text{H}_2\text{C}_2\text{O}_4$  in the ratio 1 : 3.

D. J. NORMAN.

**Manufacture of paper.** L. KIRSCHBRAUN, Assr. to FLINTKOTE Co. (U.S.P. 1,802,561, 28.4.31. Appl., 5.10.29).—A non-adhesive emulsion formed by adding a binder in liquid form, *e.g.*, asphalt, resin, waxes, oils, etc., to an aq. paste of  $\text{H}_2\text{O}$  and emulsifying agent, such as clay, soap, dextrin, casein, is mixed with fibrous pulp and passed over the paper-forming machine.

F. R. ENNOS.

**Art of stiffening [sheet materials].** W. A. BOUGHTON, Assr. to BENNETT BOX Co. (U.S.P. 1,799,949, 7.4.31. Appl., 15.12.22).—A flexible sheet of, *e.g.*, cloth, leather, or paper is treated with a dried emulsion of alkali silicate with a fatty oil (drying or non-drying) and/or glycerin.

F. R. ENNOS.

**Drying and stretching of fabrics [prior to stentering].** BRIT. CELANESE, LTD., A. MELLOR, and R. J. MANN (B.P. 358,942, 12.7.30).

**Production of [crêped] textile yarns [by twisting].** BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 358,585, 12.7.30).

**Coiling of artificial silk threads [on winding device of variable diameter].** M. SCHOENFELD (B.P. 358,900, 3.7.30. Ger., 5.8.29).

**Drying artificial silk in the form of cakes or loose packs or bundles.** ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 358,847, 23.2.31. Holl., 22.2.30).

**Apparatus for drying cellulose films and the like.** E. BLEIBLER (B.P. 359,278, 5.12.30. Ger., 14.12.29. Addn. to B.P. 343,814).

**Regenerated sulphate-pulp liquors.**—See VII. Wallboard etc.—See IX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Monel metal [vats].**—See X.

See also A., Nov., 1217, **Measurement of mercerisation.** 1232, **Absorption of acid dyes by cotton.**

### PATENTS.

**Treatment [dyeing] of textile materials.** H. BOLLMANN and B. REWALD (B.P. 353,873, 23.4.30. Ger., 11.1.30).—Addition of lecithin to the dye-bath increases the fastness of the dyeing to rubbing.

C. HOLLINS.

**Treatment of rayon.** A. REILLY, Assr. to TWITCHELL PROCESS Co. (U.S.P. 1,794,342, 24.2.31. Appl., 10.7.29).—A film of mineral oil and true "mahogany" sulphonates free from fatty substances is applied prior to weaving or knitting, and removed by immersion in  $\text{H}_2\text{O}$  before dyeing or finishing.

F. R. ENNOS.

**Colouring of cellulose esters and ethers.** IMPERIAL CHEM. INDUSTRIES, LTD., S. THORNLEY, and A. Y. TWEMLow (B.P. 353,878, 28.4.30).—Non-phototropic yellow shades are obtained by application of 5-chloro-3-nitro-*o*-toluidine or 5-nitro-*m*-4-xylydine.

C. HOLLINS.

**Printing with vat dyes.** C. I. SALA, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,790,950, 3.2.31. Appl., 17.11.25).—Hydroxylated alkylamines, *e.g.*, tri-( $\beta$ -hydroxyethyl)amine, are added to vat dye printing pastes to assist transfer of the colour to the textile.

C. HOLLINS.

**Improvement [sizing, dressing, etc.] of vegetable fibrous material.** L. LILLENFELD (B.P. 358,062, 27.3.30).—The products obtained by the process of B.P. 25,246 of 1911 (B., 1912, 786), *e.g.*, 2-hydroxytrimethylene 1 : 3-sulphide, are incorporated with the viscose sizing solution used in the processes of B.P. 302,115 and 320,062 (B., 1929, 169; 1930, 38); 7–10% (calc. on the wt. of viscose solution) may be used.

D. J. NORMAN.

**Removal of [oil etc.] stains from fabrics.** SOC. ANON. DES PROC. R. AUDUBERT (B.P. 340,077, 22.10.29. Fr., 23.10.28).—The stained fabric (*e.g.*, crêpe-de-chine) is laid on a marble slab, the stain is covered with chamois leather or other thin cloth, a 1 : 1 mixture of fuller's earth and bleaching charcoal pasted with  $\text{C}_6\text{H}_6$  is spread thereon, and the whole is covered with felt and loaded with weights for about 20 min.

A. J. HALL.

**[Drum]-dyeing of yarn [in parti-colour effects].** E. A. BARKER, and BRIT. COTTON & WOOL DYERS' ASSOC., LTD. (B.P. 359,232, 3.11.30).

**Wetting agents.**—See III. **Bleaching of paper pulp.**—See V. **Clarification of [kier] liquor.**—See VII. **Disinfection of textiles etc.**—See XIX.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Sulphuric acid concentrator of fused quartz.** T. FRANTZ (Chem.-Ztg., 1931, 55, 808).—The apparatus comprises a heating chamber containing a collecting pan for the conc. acid supported over a series of gas burners, and a heat-insulated concentrating column arranged over the centre of the pan and containing a series of superimposed, inverted  $\text{SiO}_2$  bells which act as heating surfaces. The acid flows downwards over these bells in a zig-zag manner against the rising stream of hot gases and finally collects as conc.  $\text{H}_2\text{SO}_4$  in the pan. Any acid which distils from the latter is condensed again in the upper zones of the concentrating column.

A. R. POWELL.

**Decomposition of Esthonian phosphorite by ignition with sand.** J. KUUSK (Festschr. Jubil. Ver. Estn. Chem., 1929, 14–20; Chem. Zentr., 1931, i, 3042).—The phosphorite ( $\text{P}_2\text{O}_5$  35, CaO 49.5, F 3.1%,

with varying quantities of  $\text{Fe}_2\text{O}_3$ ,  $\text{CO}_2$ ,  $\text{SO}_3$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ , etc.) occurs with sandstone, so that the average  $\text{P}_2\text{O}_5$  content is 10–20%. Heating at  $1400^\circ$  markedly increases the solubility in 2% citric acid solution. Decomp. begins at about  $1370^\circ$ , and increases with rise of temp. and with increasing sand content, reaching 98.2% of the total  $\text{P}_2\text{O}_5$  with 37% of sand at  $1380$ – $1460^\circ$ . Powdering is disadvantageous. The compound formed is  $3\text{Ca}_3\text{P}_2\text{O}_8 \cdot \text{CaSiO}_3$  or  $6\text{Ca}_3\text{P}_2\text{O}_8 \cdot \text{Ca}_2\text{SiO}_4$ . Fertiliser experiments are recorded. The decomposed phosphorite does not alter the reaction of the soil.

A. A. ELDRIDGE.

**Electrothermal volatilisation of phosphorus from Khibinsk apatites.** N. N. POSTNIKOV and Y. M. RABINOVICH (Udobr. Urozhai, 1930, 2, 910–921).—Apatite was heated electrically ( $1300$ – $1700^\circ$ ) with or without  $\text{SiO}_2$ , with  $\text{CaO}$ , and with addition of  $\text{NaCl}$ . When  $\text{SiO}_2 : \text{CaO} = 0.71$  and the mixture contained 27.7%  $\text{P}_2\text{O}_5$  the slag had an acidity [ $\text{SiO}_2 + \text{X}_2\text{O}_3 : (\text{CaO} + \text{MgO} + \text{K}_2\text{O})$ ] 1.53; under such conditions 93% yields were obtained (16.9 kw.-hr. per kg. of P). With  $\text{SiO}_2 : \text{CaO} = 0.51$  and  $\text{P}_2\text{O}_5 = 30.5\%$  the % yield was lower. The process might be used with slags of acidity 1.08. Addition of  $\text{NaCl}$  (3–4%) increased the fluidity of the slags. Carbides were not detected.

CHEMICAL ABSTRACTS.

**Minerals produced during evaporation of [sulphate-cellulose] "black liquor."** J. ANT-WUORINEN (Suomen Kem., 1931, 4, 98–100).—During evaporation of "black liquor" a form of  $\text{Na Al}$  silicate is deposited which possesses the properties of an artificial zeolite (permutit). Formation does not take place during vac. evaporation provided the temp. does not exceed  $90^\circ$ ; under atm. pressure permutit forms if the temp. rises to  $105^\circ$ , and under 3 atm. pressure at  $70^\circ$  one third of the total  $\text{Na Al}$  silicate present in solution is deposited. The addition to the liquid of certain substances such as sulphanilic acid favours deposition of the permutit; sugars have the opposite effect. H. F. HARWOOD.

**Basic salts in commercial zinc nitrate.** J. V. DUBSKÝ and M. NEVŘALOVÁ (Chem. Listy, 1931, 25, 373–375).—Up to 21% of basic nitrate was found in commercial preps. of fused  $\text{Zn}(\text{NO}_3)_2$ . The basic salts  $\text{Zn}(\text{NO}_3)_2 \cdot 3\text{Zn}(\text{OH})_2$  and  $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{Zn}(\text{OH})_2 \cdot 2\text{H}_2\text{O}$  crystallise from  $\text{H}_2\text{O}$  as  $\text{Zn}(\text{NO}_3)_2 \cdot 5\text{Zn}(\text{OH})_2 \cdot \text{H}_2\text{O}$ .

R. TRUSZKOWSKI.

**Micro-detection of  $\text{HClO}_4$ .**—See V. Monel metal [vats].—See X.

See also A., Nov., 1227,  $\text{ZnO}$ ,  $\text{SiO}_2$ , and  $\text{Al}_2\text{O}_3$  as adsorbents. 1229,  $\text{Th}(\text{OH})_4$  and  $\text{Fe}(\text{OH})_3$  gels. 1231,  $\text{Cr}(\text{OH})_3$  gels. 1246, Prep. of  $\text{Ni}$  catalyst. 1247, Catalysts for synthesis of  $\text{NH}_3$ . Decomp. of  $\text{NH}_3$  by  $\text{Fe}$ . Mixed catalysts. 1248, Preps. of  $\text{F}$ ,  $\text{H}_2\text{O}_2$ , and  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . B hydrides. 1249, Electrolysis of  $\text{Be}$  compounds. Cathodic combustion of  $\text{H}_2$ – $\text{O}_2$  mixtures. Formation and decomp. of  $\text{NH}_3$ . 1252,  $\text{NH}_4$  Ag thiosulphate. 1253, Prep. of phosphorescent substances.  $\text{Al}_2\text{O}_3/\text{Pt}$  and  $\text{Al}_2\text{O}_3/\text{Mn}$  phosphors. 1256, Adsorption and separation of  $\text{H}_2$ . 1257, Determination of traces of bromide in conc. chloride solutions. 1258, Determination of  $\text{H}_3\text{PO}_4$  by filtration, and of  $\text{CO}$ . 1259,

Determination of  $\text{Sr}$  in minerals and rocks. 1268, Esters of P acids. Colour reactions for sol. org. compounds. 1334, N fixation by *Azotobacter*.

#### PATENTS.

**Cooling the reaction zone and preventing explosions in the catalytic oxidation of ammonia.** N. CARO and A. R. FRANK (B.P. 358,850, 3.3.31. Ger., 3.3.30).—Cooling surfaces consisting of flat plates or cylindrical moulds, provided with perforations or channels, are placed directly adjacent to the metallic catalyst zone, so that the gases entering or leaving this zone traverse these surfaces. W. J. WRIGHT.

**Reaction determination and control especially for production of [calcium] hydroxide from quicklime.** C. R. REX (U.S.P. 1,796,396, 17.3.31. Appl., 9.10.28).—Quicklime from the kiln is ground at  $90^\circ$  in a Bonnet mill to pass 8-mesh and the discharge from the mill is passed at  $70^\circ$  to the hydrators, where a predetermined quantity of  $\text{H}_2\text{O}$  is added at such a rate that the temp. remains almost const. at about  $105^\circ$ . The  $\text{Ca}(\text{OH})_2$  is discharged from the hydrators when its vol. reaches the desired val. and the fine material is separated in a Raymond mill, the coarse being returned to the charging bin. A. R. POWELL.

**Burning of carbonate rock [limestone].** G. H. NILES, Assr. to DOHERTY RES. Co. and A. E. TRUESDELL (U.S.P. 1,798,802, 31.3.31. Appl., 20.4.23).—Limestone is burned in a closed kiln under atm. pressure and a portion of the gases from the top of the kiln together with steam are passed back into the bottom of the shaft above the fuel bed at a pressure of 2–3 in. of  $\text{H}_2\text{O}$  below atm. A. R. POWELL.

**Regeneration of [sulphate-pulp waste] liquors containing alkali salts and organic matter.** A. H. WHITE, Assr. to J. E. ALEXANDER and E. G. GOODSELL (U.S.P. 1,801,945, 21.4.31. Appl., 21.10.27).—Lime sludge containing  $\text{CaCO}_3$  and the conc. "black liquor" are run into the upper and lower ends, respectively, of an inclined rotary kiln, and the residues pass into pits in which they are mixed with black liquor in quantity such that  $\text{Na}_2\text{SO}_4$  is reduced to  $\text{Na}_2\text{S}$ , but the temp. does not fall below  $540^\circ$  (preferably  $700^\circ$ ). The product is extracted with water and the solution filtered to remove sludge for re-use in the process. L. A. COLES.

**Treatment [clarification] of alkaline liquors.** H. A. CURTIS and R. L. COPSON (U.S.P. 1,802,806, 28.4.31. Appl., 29.11.29).—Alkaline liquors (e.g., factory wastes such as "kier liquor") are carbonated at approx. atm. pressure with  $\text{CO}_2$  in excess (any bicarbonates formed being reconverted into carbonates by the addition of untreated liquor), and  $\text{CaCl}_2$  is then added to form insol. compounds, which may be removed by sedimentation. Flue gas containing  $\text{CO}_2$  can be used for the purpose. F. YEATES.

**Production of ammonium sulphate nitrate.** GEWERKSCHAFT VICTOR (B.P. 357,396, 358,295, and 358,301, [A, c] 17.10.30, [B] 15.10.30. Ger., [A, c] 17.10.29, [B] 15.10.29).—(A, B)  $\text{H}_2\text{SO}_4$  and  $\text{NH}_3$ , or (c)  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ , and  $\text{NH}_3$  are passed into  $\text{NH}_4\text{NO}_3$ , the operations being carried out under strongly alkaline

conditions in order to avoid N losses due to decomp. of  $\text{HNO}_3$ , and the heat of neutralisation is simultaneously used for the complete or partial evaporation of the solvent  $\text{H}_2\text{O}$ . In (A) the hot vapours mixed with  $\text{NH}_3$  which arise are not condensed, but are led directly into a rectifying column to produce conc.  $\text{NH}_3$  liquor by reflux cooling. In (B, C) the  $\text{NH}_3$  in the vapours is removed by a countercurrent of  $\text{H}_2\text{SO}_4$ . The  $\text{H}_2\text{SO}_4$  is first heated almost to its b.p. at the prevailing pressure. Operations are carried out under vac. to avoid acid losses.

F. YEATES.

#### Production of ammonium sulphate nitrate.

H. H. MEYERS, Assr. to ARMOUR FERTILISER WORKS (U.S.P. 1,801,677, 21.4.31. Appl., 7.12.28).— $\text{H}_2\text{SO}_4$  ( $d$  1.70 or above) is brought into contact with oxides of N and air, the mixture of acids so formed then being neutralised with  $\text{NH}_3$ ; the flow of reactants may be countercurrent. Alternatively, oxides of N, air in excess, and  $\text{NH}_3$  may be introduced directly into  $\text{H}_2\text{SO}_4$  to produce the double salt on evaporation. The oxides of N may be produced from  $\text{NH}_3$  by the use of a heated Pt-gauze catalyst.

F. YEATES.

**Production of alkali phosphates from ferrophosphorus compounds.** METALLGES. A.-G. (B.P. 358,756, 30.10.30. Ger., 27.1.30).—Fe-P having a high P content is heated with  $\text{Na}_2\text{SO}_4$  or  $\text{K}_2\text{SO}_4$  in the presence of sufficient coal or coke to cause practically the whole of the S to combine with the Fe and thus prevent escape of  $\text{SO}_2$ .

L. A. COLES.

**Manufacture of magnesium ammonium phosphates poor in water contents.** KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 358,637, 1.8.30. Ger., 6.8.29. Addn. to B.P. 329,495; B., 1930, 736).—Potash waste liquors or mixtures of  $\text{H}_3\text{PO}_4$  or alkali phosphate with Mg salts are treated with gaseous  $\text{NH}_3$ , a coarsely-cryst. product being obtained which can be easily dried. If  $\text{MgO}$  or  $\text{Mg(OH)}_2$  is used instead of Mg salts, addition of  $\text{CaO}$  for decomposing the  $\text{NH}_3$  salts is unnecessary.

W. J. WRIGHT.

**Manufacture of cyanates and cyanides of alkali metals.** H. WITTEK (B.P. 358,745, 24.10.30).—Alkali hydroxides or carbonates are treated with a mixture of  $\text{NH}_3$  and  $\text{CO}_2$  at 400–800° at atm. or raised pressure to yield cyanates, or in the presence of C if cyanides are desired. The process is improved by the presence of halides, cyanates, etc.; by the exclusion of metals causing decomp. of the  $\text{NH}_3$ ; by using reaction chambers lined with ceramic material; by the use of catalysts (Cu, Mn, or their alloys, Ag,  $\text{Al}_2\text{O}_3$ , active C), which may be placed in the supply and reaction chambers and mixed with the material; by admixture of hydrocarbons with the  $\text{CO}_2$ ; and by regulation of the  $\text{H}_2\text{O}$  content of the furnace gases.

L. A. COLES.

**Production of pure alkali fluorides.** E. HENE (B.P. 358,468, 4.4.30).—Decomp. of thionates in alkali fluoride solutions is effected by adding acids or acid salts, e.g.,  $\text{HF}$  or  $\text{KHF}_2$ , and heating the solution to 300°. The original solution of  $\text{K}_2\text{S}$  may be treated with  $\text{HF}$  so as to give a solution of  $\text{KHF}_2$  containing free  $\text{HF}$ , the S being then removed and the pure  $\text{KHF}_2$  decomposed into  $\text{KF}$  and  $\text{HF}$  by heating.

W. J. WRIGHT.

**Preparation of iodides of alkali or alkaline-earth metals.** J. H. VAN DER MEULEN (B.P. 358,728, 13.10.30. Ger., 14.10.29).—Solutions containing the metal oxides, hydroxides, carbonates, or bicarbonates are heated with I and reducing agents which yield only  $\text{CO}_2$  or  $\text{CO}$  and  $\text{H}_2\text{O}$ , e.g.,  $\text{CH}_2\text{O}$ ,  $\text{HCO}_2\text{H}$ ,  $\text{H}_2\text{C}_2\text{O}_4$ , or salts of the acids.

L. A. COLES.

**Continuous calcination of gypsum.** A. W. TYLER (U.S.P. 1,798,857, 31.3.31. Appl., 31.8.25).—Finely-ground gypsum is fed by means of a horizontal screw conveyor, tapering to a smaller pitch at its discharge end, into the bottom of a series of vertical flues up which it is blown by a current of steam superheated to 160–180°. In these flues the material is dehydrated to  $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ , which is separated from the steam in a settling chamber from the bottom of which the plaster is removed by a second screw conveyor to a cooling chamber.

A. R. POWELL.

**Drying and dehydrating moist high-grade neutral calcium hypochlorite.** I. G. FARBERIND. A.-G. (B.P. 357,411, 10.11.30. Ger., 15.11.29).—The material (which may also contain  $\text{CaCl}_2$ ) is dried by hot air in two stages, but in one working operation (i.e., continuously). In the first stage pre-drying takes place at 100° or above (preferably about 150°). In the second the pre-dried material, while still under the influence of the first treatment, is completely dehydrated by exposing it to a freshly introduced current of air at a still higher temp. Suitable apparatus is described.

F. YEATES.

**Manufacture of magnesium chloride from chlorine and a magnesium base.** W. R. COLLINGS, Assr. to Dow CHEM. Co. (U.S.P. 1,801,661, 21.4.31. Appl., 4.10.28).—Dil.  $\text{Cl}_2$  derived from the electrolysis of a fused chloride bath reacts with  $\text{Mg(OH)}_2$  suspended in  $\text{H}_2\text{O}$  in presence of  $\text{MgSO}_3$  [obtained by absorbing  $\text{SO}_2$  in part of the  $\text{Mg(OH)}_2$ ], to form a crude solution of  $\text{MgCl}_2$  and  $\text{MgSO}_4$ . The solution is purified by treatment with a base and is then evaporated under high vac., yielding crystals of  $\text{MgSO}_4$  (which are removed) and a mother-liquor of  $\text{MgCl}_2$  substantially free from S, which is further conc. to produce cryst. hydrated  $\text{MgCl}_2$ . This is dried and used as feed to the fused bath. The residual sulphate in the mother-liquor after most of the  $\text{MgSO}_4$  crystals have been collected is removed either by treatment with  $\text{CaCl}_2$  to ppt.  $\text{CaSO}_4$  or by concentration of the liquor to a point just short of separation of  $\text{MgCl}_2 \cdot 6$  (or 4)  $\text{H}_2\text{O}$ , when more  $\text{MgSO}_4$  crystallises out.

F. YEATES.

**Manufacture of titanium dioxide.** W. W. TRIGGS. From J. BLUMENFELD (B.P. 358,492, 7.7.30).—Mixtures, both preheated to about 400°, of approx. equal vols. of air and  $\text{TiCl}_4$  vapour (1 vol.) and of equal vols. of steam and air (10 vols.) are introduced separately into the top of an upright reaction chamber maintained at about 400°, and the  $\text{TiO}_2$  is collected in a dust chamber below at 200–400°.

L. A. COLES.

**Condensation of arsenic-sulphur compounds from gases containing them.** PATENTAKTIEBOLAGET GRÖNDAL-RAMÉN (B.P. 358,863, 16.4.31. Swed., 30.4.30).—Volatile As-S compounds in furnace gases are condensed in the form of powder by the addition of an

inert gas at such a temp. that the resulting temp. of the gaseous mixture is equal to or below the dew point of the sulphides. The compounds are then melted in the absence of air to decompose any  $\text{As}_2\text{O}_3$ . Part of the non-condensable gas obtained is used as the inert diluent. F. YEATES.

**Formation of colloidal suspensions containing copper.** G. E. SANDERS (U.S.P. 1,803,870, 5.5.31. Appl., 3.10.27).—The suspensions are prepared from mixtures of a casein compound, *e.g.*, Ca or Na caseinate, and material containing Cu, *e.g.*, Cu carbonate, Bordeaux mixture, in proportions such that there is not  $< 1$  pt. of actual casein per 2 pts. of Cu. L. A. COLES.

**Dual-effect compression method and apparatus for producing carbon dioxide snow.** J. C. GOOSMANN (U.S.P. 1,795,772, 10.3.31. Appl., 11.8.28).—The apparatus comprises a dual-effect compressor and condenser for making liquid  $\text{CO}_2$ , a storage vessel for the liquid connected through an expansion valve with an intermediate receiver with a snow-forming machine, and means for passing the gas produced in the intermediate receiver and the snow machine to the low-pressure intake of the compressor. A. R. POWELL.

**Production of carbon disulphide.** CHEM. FABR. KALK G.M.B.H., and H. OEHME (B.P. 358,249, 20.9.30).—A mixture of  $\text{CO}_2$  and  $\text{H}_2\text{S}$  is passed over glowing C to form  $\text{CS}_2$  containing CO and  $\text{H}_2$ . The  $\text{CS}_2$  is separated from them by cooling, the residual gases being returned to the raw coal gas which is the source of the initial  $\text{CO}_2$  and  $\text{H}_2\text{S}$ . An inert diluent, *e.g.*,  $\text{N}_2$ , may be present. F. YEATES.

**Recovery of oxygen and/or nitrogen with simultaneous recovery of argon.** GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 358,842, 5.2.31. Ger., 6.2.30).—Gaseous  $\text{N}_2$  under pressure from the double-stage rectification for recovery of  $\text{O}_2$  and  $\text{N}_2$  is used for heating and indirect cooling of a third rectification column in which A is recovered. The mixture treated in the last column contains only part of the  $\text{O}_2$  recovered, being taken from the upper, instead of the lower, end of the second column containing 90–98%  $\text{O}_2$ . W. J. WRIGHT.

**Apparatus for making ozone.** J. M. DAILY, Assr. to AMER. OZONE CO. (U.S.P. 1,803,600, 5.5.31. Appl., 7.1.28).—Pairs of vertical plates spaced apart to form a combined air passage and spark gap are set radially around and communicate with a central air conduit, and the interposed triangular compartments contain alternately air-cooled, high-tension electrodes and  $\text{H}_2\text{O}$ -cooled, grounded electrodes. The  $\text{H}_2\text{O}$  compartments are provided with means for circulating  $\text{H}_2\text{O}$  through them, and with removable backs for cleansing purposes. L. A. COLES.

**Production of elemental sulphur.** R. C. BENNER and A. P. THOMPSON, Assrs. to GEN. CHEM. CO. (U.S.P. 1,795,705, 10.3.31. Appl., 8.9.26).—A mixture of pyritic ore and bituminous coal is passed downwards through a closed cylindrical chamber at the bottom of which is introduced a mixture of  $\text{SO}_2$  and air, whereby the S from the ore and the gas is evolved in the free state together with  $\text{H}_2\text{S}$ , COS, and some unchanged  $\text{SO}_2$ .

The issuing vapours are passed through partly dehydrated bauxite at  $300^\circ$  to liberate the remaining S, and thence to a condenser in which practically all the S is recovered. A. R. POWELL.

**Production of sulphur from sulphur dioxide.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 357,178, 16.6.30).—Semi-coke is used in the reduction of S from  $\text{SO}_2$  by C at high temp. The semi-coke is prepared by carbonising coal with the hot sulphurous gases and then decomposing the resulting tar vapours by causing the gases to traverse a bed of highly-heated coke. The residual gas is in a suitable condition for the separation of S by condensation. Sufficient  $\text{O}_2$  is introduced with the initial gas to make the reduction exothermic. The process is carried out in a pair of vertical retorts communicating at the top, so that the flow of initial gas can be reversed periodically. F. YEATES.

**Production of sulphur [from sulphur dioxide].** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 358,580, 11.7.30).— $\text{SO}_2$  preheated to  $400$ – $500^\circ$  and containing little or no  $\text{O}_2$  is led through a comparatively deep bed of coal or the like, the balance of air required to support the reaction of  $\text{SO}_2$  with highly-heated C for the production of S being introduced at an intermediate depth, while the gaseous products are withdrawn at a still lower depth. (Cf. B.P. 357,178; preceding.) F. YEATES.

**Recovery of sulphur from gases [containing carbon disulphide and carbon oxysulphide].** S. I. LEVY and C. LEAVER (B.P. 358,851, 11.4.31).—The interaction of the gases with  $\text{O}_2$  or  $\text{SO}_2$  is effected in presence of refractory material impregnated with an Fe compound (*e.g.*,  $\text{Fe}_2\text{O}_3$ ) at  $600$ – $1000^\circ$ , a high yield of S being thus obtained. W. J. WRIGHT.

**Treating [removing sulphur trioxide from] sulphur dioxide.** BRIT. THOMSON-HOUSTON CO., LTD., Assces. of B. W. NORDLANDER (B.P. 358,431, 1.7.30. U.S., 1.7.29).— $\text{SO}_2$  (preferably dried) is passed through S, or S fused with bentonite, C black, or other substance inert to S, the material being in a finely-divided state. Reaction proceeds at room temp. Finely-divided  $\text{SeS}_2$  is used advantageously at  $80$ – $85^\circ$ . The spent S is reactivated by heating it at  $75$ – $80^\circ$  (and preferably passing an inert gas over it simultaneously), or the  $\text{SeS}_2$  by re-grinding. Apparatus claimed consists of upright co-axial cylinders between the inner walls of which the purifying material is packed, being supported on a perforated plate through which the gas passes upwards. Heated fluid circulates between the outer walls when operating with  $\text{SeS}_2$  above room temp. A battery of purifiers may be set up.  $\text{SO}_2$  containing only 0.00005%  $\text{SO}_3$  can be thereby produced. F. YEATES.

**Production of gases containing hydrogen sulphide or hydrogen sulphide mixed with sulphur dioxide.** K. VON SZOMBATHY, K. KELL, and P. SCHMITZ (B.P. 358,558, 8.7.30. Switz., 8.7.29).—Air saturated with  $\text{H}_2\text{O}$  vapour by passage through  $\text{H}_2\text{O}$  at about  $70^\circ$  is passed over an incandescent mixture of C and material containing S, *e.g.*,  $\text{FeS}_2$ , preferably in the presence of  $\text{Fe}_2\text{O}_3$  as catalyst. L. A. COLES.

Apparatus for manufacture of solid carbon dioxide. P. BROTHERHOOD, LTD., H. M. DUNKERLEY, and CARBON DIOXIDE CO., LTD. (B.P. 358,820, 24.12.30).

Pure gases by diffusion.—See I. Gases containing  $H_2$ .  $H_2$  from gaseous hydrocarbons.—See II. Oxidation of inorg. compounds.—See III. Ferrophosphorus.—See X. Fertilisers.—See XVI. Bi salts.—See XX.

### VIII.—GLASS; CERAMICS.

Presence of calcium sulphate in clay [suspensions]. Filtration process. A. CASTIGLIONI (Ind. Chim., 1931, 6, 15—18; Chem. Zentr., 1931, i, 2656).—Ca and alkali-metal sulphates flocculate clay suspensions. If the  $SO_4$  content is small, filtration is rendered possible by addition of deflocculating agents in considerable quantity, and if high,  $CaSO_4$  is preferably converted into  $BaSO_4$  by means of  $Ba(OH)_2$  or  $BaCO_3$ . A process in which the clay is treated with  $Na_2CO_3$  solution equiv. to the  $CaSO_4$  content is described.

A. A. ELDRIDGE.

Electrical conductivity of minerals and ceramic bodies at high temperatures. R. JÄGER (Keramos, 1930, 9, 87—90).—A discussion. Porcelain becomes a conductor at 280—320°. Bodies of high alkali content are, in general, better conductors than those of low alkali content. A material ("argilit") containing  $SiO_2$  46.02,  $Al_2O_3$  37.62,  $Fe_2O_3$  1.90,  $CaO$  0,  $MgO$  2.31, alkalis 0.12, with loss on ignition 12.32%, when fired, had good insulating properties at 500°.

CHEMICAL ABSTRACTS.

$H_2SO_4$  concentrator.—See VII. [Refractories for] induction furnaces.—See XI.

See also A., Nov., 1223, Diphasic nature of glass. 1230, Flocculated clay suspensions.

### PATENTS.

Manufacture of porcelain. A. M. FAIRCHILD and E. L. RATHBURN, Assrs. to MANTLE LAMP CO. OF AMERICA (U.S.P. 1,802,641, 28.4.31. Appl., 23.8.28).—A mixture of clay,  $ZrO_2$ , Zr acid phosphate (m.p. cone 10; decomp. to normal phosphate, m.p. cone 27), and alkaline-earth phosphates is fired at cone 14.

J. A. SUGDEN.

Refractories. P. G. WILLETTS (U.S.P. 1,802,296—8, 21.4.31. Appl., [A] 11.11.26, [B] 11.12.26, [C] 14.3.27).—A mixture of clay and diaspore (or bauxite) is ground to pass 325-mesh, mixed with 30% of its wt. of  $H_2O$ , and pugged (or, preferably, made into a slip, blunged, filter-pressed, and pugged). After drying, it is broken up, fired at cone 31, crushed to about 12-mesh, mixed with a proportion of the starting materials, dry-pressed (with not more than 12% of  $H_2O$ ), and fired to cone 30. By using suitable proportions of clay and aluminous mineral, the final product may contain 80% or more of mullite and have a true porosity of 10% (apparent porosity 2%). The material withstands load at high temp. and corrosion by molten glass.

J. A. SUGDEN.

Cellulose derivatives [for laminated glass].—See V. [Glass] furnaces.—See XI.

### IX.—BUILDING MATERIALS.

Hydraulic and white magnesian cements. A. C. VOURNAZOS (Z. anorg. Chem., 1931, 200, 237—244).—By the action of  $SiO_2$  gel on  $MgO$  in presence of  $H_2O$  at room temp., the compound  $O:Si:(O \cdot Mg \cdot OH)_2$  is slowly formed. Natural Santorin pozzuolanas contain 17—19% of amorphous  $SiO_2$ . By mixing the proportions of this material and of  $MgO$  necessary for forming the compound a cement is obtained which has a setting time of 48 hr. (air) or 90 hr. ( $H_2O$ ). The strengths in compression of air- and water-cured mortars containing 21% of sand at 7 and 28 days and 1 year were 81, 105, 201, and 65, 72, 141 kg. per sq. cm., respectively. The max. strengths were attained within 1.5 years, and were 38 kg. per sq. cm. in tension and 285 kg. per sq. cm. in compression. The mortar attacks Fe less readily than does Portland cement mortar, and has almost the same coeff. of expansion as Fe; it is not attacked by  $H_2O$  or by atm.  $CO_2$ . The hardened material is greyish-white, and can be coloured by replacing 5% of the sand by a mineral colour without appreciable loss of strength. A white mortar can be obtained by using artificially prepared  $SiO_2$ ,  $MgO$ , and white sand; the setting time in air is 40 hr. When polished, the material closely resembles marble in appearance. H. F. GILLBE.

Retardation of setting of gypsum cement by alcohols. S. YAMANE (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 16, 276—283).—Retardation is due to the reduction of the difference between normal and supersaturation solubility of  $CaSO_4$  in 30% alcoholic ( $EtOH$ ,  $MeOH$ ,  $Pr^oOH$ ) solutions. R. TRUSZKOWSKI.

Modification of the physical properties of cement produced by carbonation. F. L. BRADY (Cement, 1931, 4, 1105—1109).—The calc. linear expansion (0.053%) due to the conversion of  $Ca(OH)_2$  into  $CaCO_3$  in a normal Portland cement-sand mortar does not correspond with the contraction actually observed. This is probably due to carbonation proceeding further and decomposing part of the cement gel. Test specimens showed marked increase in contraction when stored in  $CO_2$ , the relative humidities being carefully controlled, and this shrinkage of the gel may be a factor in producing surface defects of concrete. C. A. KING.

Mineral formation of fused kaolin-lime mixtures. J. WEYER (Zement, 1931, 20, 264—265; Chem. Zentr., 1931, i, 3040).—Fusion of 9  $CaO$  : 1 kaolin always gave unstable  $2CaO, SiO_2$ . Mixtures richer in  $CaO$  gave  $3CaO, SiO_2$ . At temp. above 1450°  $3CaO, SiO_2$  and  $3CaO, Al_2O_3$  were formed. A. A. ELDRIDGE.

See A., Nov., 1225,  $CaSO_4$  in sea-water.

### PATENTS.

Brick and like kilns. C. R. IVORY (B.P. 358,053, 2.7.30).—A down-draught kiln is provided with recesses in the floor a few in. wide in which is placed combustible material which, when the kiln attains a moderate temp. from the fires under the bag walls, becomes ignited and, in the later stages of the firing, increases the temp. of the lower portion of the stack of goods, giving rise to more even firing. The network of cross- and longitudinal flues under the kiln is divided into four sections

having separate damper-controlled main flues from each and preferably a chimney for each pair.

B. M. VENABLES.

**Manufacture of wallboard, pressboard, etc.** F. S. VIVAS (U.S.P. 1,800,121, 7.4.31. Appl. 3.12.29).—Satisfactory products are obtained at lower temp. and pressures than are normally used, *e.g.*, at 100–107°/150–200 lb. per sq. in., if the fibrous material is beaten in a dextrin solution of *d* 1.14 (at 38° for a porous board) or of *d* 1.17 (at 93° for a compact board). A suitable dextrin product is prepared by heating 40 pts. of starch with 60 pts. of glycerin and diluting the resulting clear solution with 100 pts. of EtOH. Fireproofing agents may be incorporated with the board.

D. J. NORMAN.

**Disintegrating materials. Sound-deadening compositions.**—See I. Rotary cement kiln.—See II. Dispersions.—See III. Burning limestone. Calcining gypsum.—See VII. [Cement] furnaces.—See XI.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Value of low-total carbon cast irons.** W. WEST (Metallurgia, 1931, 4, 187–192).—Castings containing < 3.3% C are subject to drawing and sinking. Test bars of various compositions cast in oil-sand moulds point, however, to the superior mechanical properties of Fe containing 2% C, 2.2% Si, 0.8% Mn, 0.06% S, and 0.40% P. An alloy of this analysis cast from 1450° had a tensile strength of 21 tons per sq. in. together with a high transverse test val.

E. H. BUCKNALL.

**Origin of iron in corrosion products due to London atmosphere.** E. WILSON (Engineering, 1931, 132, 535).—Corrosion products on Al and Cu conductors exposed to London atm. contained traces of Fe compounds sol. in HCl, probably FeS, which was found also in different samples of soot. The origin is most probably due to the presence of pyrites in coal which is burned in the ordinary manner. The relative magnetic susceptibility of constituents of the corrosion products is given.

C. A. KING.

**Solubility, peptisation, wetting, and flotation.** A. W. FAHRENWALD (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 59–64).—Flotation of a mineral particle occurs when there is little or no chemical bonding affinity between the H<sub>2</sub>O phase and the mineral, *i.e.*, when the energy transition from phase to phase is abrupt. Peptisation is the result of a strong chemical bonding force between the solid surface mols. and H<sub>2</sub>O mols. or ions in the H<sub>2</sub>O phase. Zn blende is feebly peptised in H<sub>2</sub>O, but strongly peptised in solutions containing < 15–20 millimols. of NaCN or < 5 millimols. of Na<sub>2</sub>HPO<sub>4</sub> or Na<sub>2</sub>S per litre. With higher concentrations of these reagents the adsorbed polar films on the ZnS particles are removed and the mineral is amenable to flotation. There appears to be a relation between peptisation, wettability, polarity, and flotation.

A. R. POWELL.

**Differential wetting effects in flotation.** G. BARSKY and S. A. FALCONER (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 95–104).—The differential wetting effects of H<sub>2</sub>O and pine oil on mineral surfaces have been

examined by means of a device which registers the change of pressure due to displacement of one of the liquids by the other when mineral layers, one wetted with H<sub>2</sub>O and one with oil, are held tightly together in a closed compartment. The results obtained show that reagents which produce flotation of the mineral alter its surface, so that pine oil displaces H<sub>2</sub>O thereon, whereas depressants increase the affinity of the surface for H<sub>2</sub>O in preference to oil. The rate of displacement of H<sub>2</sub>O by oil on galena increases rapidly with decrease in particle size.

A. R. POWELL.

**Influence of flotation on smelting.** G. L. OLDRIGHT (Trans. Amer. Electrochem. Soc., Sept., 1931, 60, 105–116).—The effect of higher-grade products which can be obtained by flotation on the methods employed in smelting Cu, Zn, and Pb ores is discussed with reference to the practice in present use in some American smelting works. In Cu smelting, blast furnaces are now rarely used as most of the gangue is removed in the flotation operation. High-grade Zn concentrates are usually treated by hydrometallurgical methods, and high-grade Pb concentrates provide a more fusible charge to sinter and smelt, but often the smaller amount of slag-forming material per unit of metal is so difficultly fusible that large quantities of old, more readily fusible slags have to be returned to the furnace with the new charge. Nevertheless, in all three cases, reduction of the gangue material in the smelter charge has reduced costs and frequently yielded a much purer product than that previously obtained.

A. R. POWELL.

**Amalgamation, cyanidation, and flotation [of gold and silver ores].** F. VOGEL (Chem.-Ztg., 1931, 55, 806–807).—The application of these three methods in the treatment of complex Au and Ag ores is discussed. It is suggested that amalgamation of the coarsely ground ore, followed by flotation after a further grinding, and cyanidation of the flotation tails after sliming should give the best extraction at min. cost.

A. R. POWELL.

**Autogenous and electrical welding of monel metal.** F. SCHÜPPEL and W. KÄSTNER (Z. Metallk., 1931, 23, 286–289).—Monel metal can be satisfactorily welded without fluxes by O<sub>2</sub>–C<sub>2</sub>H<sub>2</sub>, by electric-resistance methods, or by means of the arc, and the joints so produced are just as resistant as is the original metal to corrosion by dye-baths, alkalis, soap solutions, and bleaching liquors. Solutions containing Cu, used as mordants in dyeing furs, and HCO<sub>2</sub>H solutions cannot be handled in monel metal vessels. The tensile strength of welds in monel metal made by any of the above-mentioned methods is generally at least 90% of that of the unwelded metal, and the ductility is almost as good.

A. R. POWELL.

**Steel for making die-casting moulds for aluminium alloys.** A. R. PAGE (Hausz. V.A.W. Erftw. Aluminium, 1931, 3, 83–85; Chem. Zentr., 1931, i, 2926).—Cr–V-steel and W-steel were least susceptible to erosion, but the results were somewhat indefinite.

A. A. ELDRIDGE.

**Simple aluminium cast alloys.** W. CLAUS (Hausz. V.A.W. Erftw. Aluminium, 1931, 3, 54–66; Chem. Zentr., 1931, i, 2926).—A discussion.

A. A. ELDRIDGE.

**Effect of iron on the electrical conductivity and tensile strength of aluminium.** M. KURODA (Suppl. Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 16, 1—2).—The addition of up to 1.5% Fe increases the electrical resistance and tensile strength of Al wire, whether hard-drawn or annealed. The increase varies directly with the Fe content and is, for 1% Fe, 1.5 kg./sq. mm. in strength and 0.15 mΩ cm. in resistance. Fe also tends to increase elongation. C. IRWIN.

**Occurrence of small quantities of gases and oxides in aluminium and its alloys.** R. STERNER-RAINER (Z. Metallk., 1931, 23, 274—279).—Recent work on the determination of such gases and oxides and on methods for the degassing of melts prior to casting are reviewed. For most practical purposes it is sufficient to hold the molten metal at 710—730° for some time prior to casting. Of the various treatments which have been proposed for removing adsorbed gas from the molten metal the best results are obtained by that in which a mixture of kieselguhr with coal dust and  $\text{NH}_4\text{HF}_2$  is made into a paste with  $\text{CCl}_4$  and pushed down to the bottom of the crucible with an inverted Fe spoon. A. R. POWELL.

**Microscopical and thermal examination of Al-Sb-Mg alloys.** E. LOOFS-RASSOW (Hausz. V.A.W. Erftw. Aluminium, 1931, 3, 20—32; Chem. Zentr., 1931, i, 2926—2927).—The three systems Al-AlSb, Al-Mg<sub>3</sub>Sb<sub>2</sub>, and AlSb-Mg<sub>3</sub>Sb<sub>2</sub> exhibit binary or quasi-binary eutectics. Al-AlSb and AlSb-Mg<sub>3</sub>Sb<sub>2</sub> are completely miscible in the liquid state, whilst the system Al-Mg<sub>3</sub>Sb<sub>2</sub> shows a miscibility gap. Addition of Sb to Al alloys containing Mg produces the new structure element Mg<sub>3</sub>Sb<sub>2</sub> which is only slightly sol. in Al, the solubility increasing with rise of temp.

A. A. ELDRIDGE.

**Chromium plating for the most useful metals and alloys.** K. ALTMANNBERGER (Chem.-Ztg., 1931, 55, 817—818).—The selection of intermediate coatings on which a finishing coating of Cr is to be plated depends on the base metal, and the purpose for which the coating is applied, e.g., as a protection against corrosion, for the production of a hard surface layer, or as a protection against scaling at high temp. For Fe articles a rust-protective coating is obtained by applying a thin Cu coating in a KCN bath, thickening up the coating in a  $\text{CuSO}_4$  bath, covering the Cu with Ni until a total thickness of 0.025 mm. is obtained, then finishing with the usual Cr plating. Al alloys are preferably plated first in an alkaline Zn solution, then with Ni in a citrate bath before Cr plating. A. R. POWELL.

**Colouring of cadmium[-plated] deposits.** H. KRAUSE (Z. Metallk., 1931, 23, 283—285).—An "oxidised Ag" finish may be imparted to Cd-plated articles by immersing them for a short time in a cold solution containing 60 g. of  $\text{KClO}_3$  and 35—40 g. of  $\text{Cu}(\text{NO}_3)_2$  per litre, whereby they become coated with a firmly adherent, bright black deposit which is rendered more permanent by burnishing with a brush coated with wax or by lacquering. A good brown colour resistant to the salt-spray test is obtained by immersing the plated articles in a hot solution containing 10—15 g. of  $\text{Cu}(\text{NO}_3)_2$  and 2.5 g. of  $\text{KMnO}_4$  per litre. Richer brown

tones may be obtained by using more conc.  $\text{KMnO}_4$  solutions containing  $\text{Cd}(\text{NO}_3)_2$ , with or without a small quantity of  $\text{FeCl}_3$ . A. R. POWELL.

**Composition and properties of sand for aluminium moulds.** A. RODEHÜSER (Hausz. V.A.W. Erftw. Aluminium, 1931, 3, 75—83; Chem. Zentr., 1931, i, 3049).

**Dilatometric study of binary magnesium-aluminium alloys in the cast state.** W. CLAUS and S. BRIESEMEISTER (Hausz. V.A.W. Erftw. Aluminium, 1931, 3, 40—53; Chem. Zentr., 1931, i, 2926).

[Metal-melting] induction furnaces.—See XI. Corrosion of tinplate.—See XIX.

See also A., Nov., 1220, Mg. Electric properties of metals. Magnetic Cu-Ni. Sintering of powdered Fe. Vaporisation of Mg in vac. 1223, Al-Cr. Alloys of Ag with Bi, Sb, and As. Systems Au-Sb and Ag-Sn. Au-Sn and Cu-Si alloys. 1224, Systems Cu-Mg, Au-Cu, Ag-Hg, Cu-Pb, Al-Si, Au-Ni, and Au-Bi. 1235, Systems FeO-MnO, MnS-MnO, MnS-MnSiO<sub>3</sub>, MnS-Fe<sub>2</sub>SiO<sub>4</sub>, and Ag-Cu-O. 1238, Deposition of Ni. 1249, Be. Cu-plate. 1253, Pure Ce. 1261, Determination of large quantities of Mn. 1263, Structure [of paint films on metals]. 1264, Cr-steel vessels for analysis.

#### PATENTS.

**Metallurgical furnace.** F. H. LOFTUS (U.S.P. 1,792,021, 10.2.31. Appl., 25.4.21).—A regenerative open-hearth furnace is provided with a burner on the Venturi valve principle in which part of the preheated air under increased pressure forms a central core about which is an envelope of preheated gas surrounded by an outer layer of preheated air. A. R. POWELL.

**Cupola furnaces.** W. G. GASS (B.P. 358,267, 30.9.30).—The wall of the cupola proper terminates a short distance above the charging hole, and a surrounding casing of larger diam. starts below the top of the cupola and extends upwards, forming a mixing chimney in which fumes and fresh air drawn in through the annular space mingle. To divert the cross-flame produced when the charging hole is opened, a baffle is provided on the top of the opposite wall.

B. M. VENABLES.

**Furnace for melting metals of relatively low m.p. [e.g., type metal].** (SIR) W. G. ARMSTRONG WHITWORTH & Co. (ENGINEERS), LTD., and I. L. HALL (B.P. 356,037, 12.11.30).—In a furnace of the type in which the metal is cast by means of a piston working in a cylinder which pumps the metal to the casting nozzle, the casting cylinder is suspended from a revoluble member on the furnace casting and means are provided for circulating the combustion gases over the surface of the metal in the pot to prevent oxidation. A. R. POWELL.

**[Annealing] furnace.** F. A. FAHRENWALD (U.S.P. 1,791,404, 3.2.31. Appl., 19.2.29).—A continuous annealing furnace for heat-treating metal strips, sheets, etc. is provided with a series of equally-spaced horizontal shafts journaled in apertures in the side-walls and provided with circular discs close to the sides of the



furnace, the discs carrying conveyor rolls eccentric to the shafts.

A. R. POWELL.

**Heat-treatment of metal [wire etc.].** WELLMAN SMITH OWEN ENG. CORP., LTD., and A. OGILVY-WEBB (B.P. 357,575, 21.5.30).—Metallic wire, strip, or sheet is passed through a vertical furnace comprising preheating, heating, and cooling zones any or all of which may be muffled if desired. The jackets of the cooling zone are divided into sections with independent supplies of water, and if the wire has a downward travel the preheating zone may be heated by gases rising from the heating zone; the gases may also preheat the air of combustion.

B. M. VENABLES.

**Manufacture of pure metals [iron, nickel, chromium, and their alloys].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 358,494, 31.3.30).—Impure Fe or Fe alloys containing C and Ni, Cr, or Si are heated in HCl and/or Cl<sub>2</sub> and the resulting chlorides are separated by fractional sublimation or collected in one container if an alloy is desired. The purified chlorides or mixtures thereof are then reduced in H<sub>2</sub> either with or without previous conversion into oxides by treatment with steam.

A. R. POWELL.

**Manufacture of commercial products [ferrophosphorus etc.] from phosphatic material.** J. W. WALTON, ASSR. to TENNESSEE PRODUCTS CORP. (U.S.P. 1,803,417, 5.5.31. Appl., 23.3.29).—A mixture of phosphatic material, C, material containing Fe, and a flux is heated in a blast furnace provided with means for withdrawing part of the gases from the combustion zone. Ferrophosphorus and slag (which is worked up to slag wool) are withdrawn from the base, the gases removed from the combustion zone are passed through H<sub>2</sub>O to deposit a sludge from which P is subsequently recovered, and the gases leaving the top of the furnace are treated for the recovery of dust for use in polishing.

L. A. COLES.

**Manufacture of [asphalt-]coated metal sheets.** L. W. BLANCHARD (B.P. 355,796, 26.5.30).—The sheets are coated on each side simultaneously by passing them in turn through a preheating chamber, a pair of superimposed rolls by means of which the coating is applied to the surface, and a pair of chilling and pressing rolls.

A. R. POWELL.

**Casting or moulding metals.** W. W. SPOONER (B.P. 358,641, 6.8.30).

**Drum furnace. Heating furnace.**—See I. Dispersions.—See III. Regenerative treatment of electrolyte.—See XI. Rubber-metal bond.—See XIV.

## XI.—ELECTROTECHNICS.

**The low-frequency induction furnace and its scope.** A. G. ROBIETTE (Metallurgia, 1931, 4, 153—155, 175—176).—The development of the normal-frequency, cored induction furnace for the melting of Cu-Zn, Cu-Ni, Cu, and Zn, and in superheating grey cast Fe is described. The life of refractories of various types and makes is also discussed with special reference to the Rohn process of sintering furnace linings *in situ*.

E. H. BUCKNALL.

**Conductivity of ceramic bodies etc.**—See VIII. Fe corrosion products. Welding monel metal.

**Conductivity of Al. Cr-plate. Colouring Cd-plate.**—See X. Ageing of rubber.—See XIV.

See also A., Nov., 1212, Vacuum thermo-elements. 1220, Electric properties of metals. Cu-Ni alloys. 1224, Au-Ni and Au-Bi as conductors. 1238, Deposition of Ni. 1248, Prep. of F, H<sub>2</sub>O<sub>2</sub>, and (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>. Electrolysis of B<sub>2</sub>H<sub>6</sub>. 1249, Be. Cu-plate. Cathodic combustion of H<sub>2</sub>-O<sub>2</sub> mixtures. Condensation of hydrocarbons. Formation and decomp. of NH<sub>3</sub>. 1253, Pure Ce. 1263, Photoelectric cells for colorimetry. Fe resistance lamps. Na coulometer. Resistivity of mineral waters.

## PATENTS.

**Electric furnace.** F. A. HANSEN, ASSR. to HEVI DUTY ELECTRIC CO. (U.S.P. 1,802,892, 28.4.31. Appl., 18.4.29).—Oxidation of the terminal connected with a heating coil is prevented by a stream of gas, e.g., H<sub>2</sub>, flowing in a ceramic refractory tube surrounding the terminal and passing through the porous wall of the furnace chamber.

J. S. G. THOMAS.

**[Three-phase] electric furnaces.** BRIT. HARTFORD-FAIRMONT SYND., LTD., ASSRS. of C. E. CORNELIUS (B.P. 358,656, 19.8.30. Swed., 3.7.30).—Melting of glass, cement, etc. is carried out in 3 recesses separated by angles of 120°, and each phase is provided with two sets of electrodes respectively opposing one of those of the next phase; the surfaces of opposed electrodes are substantially parallel.

J. S. G. THOMAS.

**High-temperature electric heating furnace.** J. D. MORGAN, ASSR. to DOWERTY RES. CO. (U.S.P. 1,803,282, 28.4.31. Appl., 15.10.28).—An inner, refractory, conducting layer containing ground SiO<sub>2</sub> and ZrO<sub>2</sub>, e.g., Zr silicate, is surrounded by a porous, refractory, insulating layer, e.g., of "Silocel" brick or bonded diatomaceous earth.

J. S. G. THOMAS.

**Induction furnace. Induction furnace protective circuits.** J. V. BREISKY and T. H. LONG, ASSRS. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,801,790—1, 21.4.31. Appl., [A, B] 5.3.30).—(A) An earthed shield of electrically-conducting laminated or subdivided material is arranged in the insulating material placed between the crucible or lining and the surrounding inductor coil. Means are provided to interrupt the supply of current to the inductor coil and to actuate an alarm in case of breakdown of the insulator and the escape of metal through the crucible. (B) A non-magnetic conducting shield connected with a source of current and comprising two parts, e.g., an inner and an outer cage, insulated from each other, is arranged between and insulated from a crucible and a surrounding inductor coil, and current supply to the coil is interrupted should a short circuit occur between parts of the shield.

J. S. G. THOMAS.

**Induction furnace.** P. H. BRACE, ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,802,701, 28.4.31. Appl., 1.11.29).—A relatively shallow crucible, provided with fluid-cooled, superposed energising coils arranged axially, is surrounded by laminations extending axially and radially. Heavy metal plates, of substantially the same contour as the laminations, interfit

with and support refractory roof members, and relatively thin, fluid-cooled, conducting metal plates interfit with one edge of the laminations. J. S. G. THOMAS.

**Regenerative treatment of electrolyte.** T. LEWIN (U.S.P. 1,800,457, 14.4.31. Appl., 9.9.27).—In a process for recovering Cu, Sn, and Pb from brass or bronze (cf. U.S.P. 1,574,043; B., 1926, 369), the electrolyte is removed from the bath and heated at 66–100° to accelerate the hydrolysis of its Sn and Sb content. J. S. G. THOMAS.

**Electrical precipitation [of dust from gases].** G. H. HORNE, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,800,529, 14.4.31. Appl., 27.2.29).—Gas flows, preferably vertically, through a series of precipitators, each comprising a number of elements arranged in parallel, and means are provided for reversing or varying the sequence of gas flow through these elements and for cleaning the electrodes of the respective elements between successive changes in the sequence of gas flow, when the element in question is not the last in the sequence and the gas flows therethrough preferably in a downward direction. J. S. G. THOMAS.

**Apparatus for electrical treatment of gases containing corrosive material and mercury.** K. I. MARSHALL, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,801,515, 21.4.31. Appl., 13.3.29).—Amalgamated electrodes are claimed. J. S. G. THOMAS.

**Gaseous electric [unidirectional] discharge devices [for use as polarity indicator etc.].** BRIT. THOMSON-HOUSTON CO., LTD., Assees of T. E. FOULKE (B.P. 358,614, 21.7.30. U.S., 27.7.29).—One electrode of a cathode glow-discharge tube is concealed and the surface of the other, *e.g.*, of Ni or Fe, is coated, at least partly, with a material, *e.g.*, Ba, K, or compounds thereof, having a lower work function than that of the material of the concealed electrode. J. S. G. THOMAS.

**Electrode [for negative-discharge glow lamps].** W. R. WALKER, Assr. to GEN. ELECTRIC VAPOR LAMP CO. (U.S.P. 1,803,985, 5.5.31. Appl., 20.7.25).—A Ni-plated Fe electrode is impregnated with  $\text{H}_2$ . J. S. G. THOMAS.

**Manufacture of luminescent tubes.** P. F. J. LEBRUN (U.S.P. 1,802,473—4, 28.4.31. Appl., [A] 12.12.27, [B] 13.3.29).—(A) Residual gases and impurities are removed from the tubes by passing a high-tension discharge through a conducting gas, *e.g.*,  $\text{N}_2$ , introduced into the tubes, and exhausting the tubes when lines in the region 5880 Å. appear in the spectrum of the discharge. (B) Electrodes are heated in the presence of  $\text{NH}_3$ , and tubes are flushed with  $\text{NH}_3$  through which a high-tension discharge is passed. J. S. G. THOMAS.

**Photoelectric cells.** TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 358,539, 6.6.30. Ger., 6.6.29).—An alternating voltage is applied to a semi-conducting layer coating the photoelectrically active surface of the cell. J. S. G. THOMAS.

**Treating [lead] storage-battery plates.** W. S. HUTCHINSON, Assr. to LEKTOPHONE CORP. (U.S.P. 1,802,818, 28.4.31. Appl., 27.10.25).—The composition of the paste used in the plates and the  $d$  ( $>1.2$ ) of the  $\text{H}_2\text{SO}_4$  in which the plates are "formed" are adjusted

so that the acid, at the end of the "forming" process, is of normal operating concentration and replacement of acid is not necessary. J. S. G. THOMAS.

**[Separator for] storage battery.** E. W. SMITH (U.S.P. 1,800,364, 14.4.31. Appl., 30.1.22. Renewed 23.10.28).—A sheet of inert, finely-porous material, *e.g.*, asbestos, is arranged in contact with and between the positive plate and a layer of perforated hard rubber forming part of a rubber-wood separator. J. S. G. THOMAS.

**[Combining separators for] electric batteries.** M. and P. WILDERMAN (B.P. 358,466, 3.4.30).—Porous or non-porous strips, tubes, etc., *e.g.*, of vulcanised rubber and/or a material, *e.g.*, a solution of celluloid in  $\text{COMe}_2$ , are employed. J. S. G. THOMAS.

**Preparation of depolarising mixtures.** G. W. HEISE, Assr. to NAT. CARBON CO., INC. (U.S.P. 1,801,743, 21.4.31. Appl., 10.10.27).— $\text{MnO}_2$  is added in stages to a mixture containing approx. equal proportions of  $\text{MnO}_2$  and C which has been milled in stages, until the  $\text{MnO}_2$ :C ratio is about 3:1, and the final mixture is milled. J. S. G. THOMAS.

**Electric, liquid [mercury] flow switch.** W. R. WALKER, Assr. to GEN. ELECTRIC VAPOR LAMP CO. (U.S.P. 1,803,986, 5.5.31. Appl., 18.10.29).—Hg containing 0.1% Ni, or Fe, Co, Pt, Pd, Os, or other metal of group VIII, is employed in the switch. J. S. G. THOMAS.

**Dynamo brush.** N. R. HAAS, Assr. to DELCO-REMY CORP. (U.S.P. 1,804,052, 5.5.31. Appl., 10.10.25).—The brush is composed of a sintered mixture of graphite (81%), coked bakelite (10%), and uncoked bakelite (9%). J. S. G. THOMAS.

**[Cathodes for] electric discharge devices.** BRIT. TALKING PICTURES, LTD., Assees. of L. DE FOREST (B.P. 358,571, 10.7.30. U.S., 13.7.29).

**Pure gases by diffusion.**—See I. C from flames.—See II.  $\text{MeCHO}$ ,  $\text{Ac}_2\text{O}$ .—See III. Nitrated cellulose.—See V.  $\text{O}_3$ .—See VII. Rubber goods.—See XIV.

## XII.—FATS; OILS; WAXES.

**Oxyns. VII. Drying process of free unsaturated fatty acids from oils.** A. EIBNER and V. JUNG (Chem. Umschau, 1931, 38, 267—273, 279—285).—Thin layers of linoleic, linolenic (prepared from the insol. bromides), and  $\alpha$ -clæostearic acids dried slowly to more or less tenacious colourless films; under certain conditions of warmth and moisture the dried films might yellow (the strong discoloration and re-softening observed in earlier experiments did not occur in this set). Clupanodonic acid films were exceptional in becoming brown while drying (at 100°). All films sintered or melted between 110° and 170°, and were much more hydrophile than films of drying oils; the dried films of clupanodonic, linolenic, and elæostearic acids were sol., difficultly sol., and partly sol., respectively, in warm  $\text{EtOH}$ . The  $\alpha$ -clæostearic film approached nearest to the character of a glyceride film. The Ac vals. were high (276—310; presence of true hydroxy-acids?). Microscopic crystals were visible in drying linoleic acid and linseed oil films, but could not be isolated for identification. The colourless, oxidised (filmed) acids seem to be a labile form

(cf. Mulder's "white linolic acid") analogous to that in the dried glyceride films, for, on alkaline saponification and reacidification of the acid films, the usual dark red-brown soaps and "oxidised" acids were obtained, those from  $\alpha$ -elavostearic acid film alone being completely insol. in  $\text{Et}_2\text{O}$ . E. LEWKOWITSCH.

**Tonka-bean oil.** C. D. V. GEORGI and G. L. TEIK (J.S.C.I., 1931, 50, 318 T).—Seeds (beans) of *Dipteryx odorata* (N.O. *Papilionaceae*) contain a little coumarin and 24.8% (calc. on fresh seed) of fixed oil extractable by light petroleum and having:  $d_{4}^{20}$ , 0.878, m.p.  $7.2$ – $11.8^\circ$ , sap. val. 198.5 (cf. vals. in lit.), I val. (Wijs) 72.6,  $n_D^{27}$  1.4680, acid val. 1.0, unsaponifiable matter 0.5%; the titer, mean mol. wt., and I val. (Wijs) of the fatty acids are 42.0°, 302.9, and 76.9, respectively. It is unlikely that the oil could compete commercially with, e.g., arachis oil or synthetic coumarin.

E. LEWKOWITSCH.

**Monel metal [vats].**—See X. **Fat in buttermilk.**—See XIX.

See also A., Nov., 1269, **Monoglycerides of lower fatty acids.** 1271, **Alcoholysis.** Prep. of **linoleic acid.** Polymerisation of **Me linolenate and linoleate.** 1272, **Lignoceric acid and its alcohol from sulphate soap.** 1321, **Insect waxes.** 1334, **Synthesis of fats from carbohydrates.** 1341, **Tea-seed oil.**

## PATENTS.

**[Solvent] extraction of [fats and] oils.** E. E. REID, Assr. to COLUMBIA ENG. & MANAGEMENT CORP. (U.S.P. 1,802,533, 28.4.31. Appl., 13.9.28).—A solvent, e.g., *n*- or *iso*-butane, which is gaseous at room temp. and pressures is employed, and sufficient pressure is applied (e.g., at  $16$ – $27^\circ$ ) to maintain it in the liquid state while in contact with the material to be extracted.

E. LEWKOWITSCH.

**Extracting fat from cacao bean.** I. S. HOCKER, Assr. to HOCKER CORP. (U.S.P. 1,803,615, 5.5.31. Appl., 24.8.28).—Ground cacao bean (chocolate liquor, 1000 lb.) is warmed (at  $93^\circ$ ) with  $\text{H}_2\text{O}$  (10 gals.) while stirring slowly about a horizontal axis in order to gelatinise the starchy matter, which is then saccharified by the addition of  $\text{H}_2\text{O}$  (140–240 gals.) and diastatic malt extract (25 lb.) while stirring about a vertical axis at  $65^\circ$ ; on adding  $\text{Na}_2\text{CO}_3$  (3 lb.) the liberated fat rises to the surface with the  $\text{CO}_2$  bubbles generated.

E. LEWKOWITSCH.

**[Lard] rendering tank.** H. E. DUDYCHA, Assr. to SWIFT & CO. (U.S.P. 1,803,604, 5.5.31. Appl., 26.2.27).—A flexible tube provided with a float draws off the rendered fat from just below the surface; the mouth of the float is protected with wire screens

E. LEWKOWITSCH.

**Catalytic hydrogenation of fatty acids.** HENKEL & CIE. G.M.B.H. (B.P. 358,328, 10.11.30. Ger., 23.12.29).—Fatty acids from soya-bean oil are purified by steam-distillation in vac. before hydrogenation, and the hardened fatty acids are removed from the Ni catalyst by steam-distillation; these will furnish stable white soaps. [Stat. ref.] E. LEWKOWITSCH.

**Manufacture of oxidation products of derivatives of higher fatty acids containing sulphur.** A. CARPMEL. From I. G. FARBEIND. A.-G. (B.P. 353,736,

25.8.30. Addn. to B.P. 340,012; B., 1931, 355).—The products of the prior patent are oxidised, e.g., with  $\text{KMnO}_4$ ,  $\text{H}_2\text{O}_2$ , Caro's acid, hypochlorites, to give disulphides, and by further oxidation sulphonic acids. The alkali salts are  $\text{H}_2\text{O}$ -sol. viscous oils and are used for cleansing; the disulphides are also disinfectants.

C. HOLLINS.

**Purification and concentration of emulsions of sulphurised fatty-oil products.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, and R. B. F. F. CLARKE (B.P. 358,037, 2.5.30. Addn. to B.P. 343,533; B., 1931, 501).—The emulsion prepared according to the prior patent is diluted and settled to allow scum, sediment, etc. to settle out, decanted, and flocculated with, e.g.,  $\text{AcOH}$ ; the flocculated mass is re-emulsified by trituration with conc. aq.  $\text{NH}_3$ . E. LEWKOWITSCH.

**Removal of oil from olives and other fruits.** H. BERNIER, Assr. to CALIFORNIA PACKING CORP. (U.S.P. 1,800,336, 14.4.31. Appl., 1.6.21).—The flesh is separated from the pits and pulped by forcing through wire screens, and the oil is removed by centrifuging without further addition of  $\text{H}_2\text{O}$ . A second-grade oil is recovered from the aq. liquor by heating and breaking the emulsion with acid. E. LEWKOWITSCH.

**Dispersions. Oxidation of org. compounds.**—See III. **Synthetic cream. Edible emulsions.**—See XIX. **Antiseptic etc.**—See XXIII.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Varnish pitches and their investigation.** F. EICHSTÄDT (Farben-Chem., 1931, 2, 466–467).—The types of pitches used in black varnishes are indicated and methods of evaluating them by determining their softening and drip points, solubility (in  $\text{CS}_2$ ,  $\text{CCl}_4$ , and light petroleum), ductility, adhesion, viscosity, ash and wax contents are summarised. Some characteristic tests for individual pitches are also detailed.

S. S. WOOLF.

**Comparison of physical characteristics of French and American commercial pine gum, rosin, and turpentine.** A. R. HITCH (Ind. Eng. Chem., 1931, 23, 1135–1136).—The composition and properties of representative French pine gums, and rosin and turpentine obtained therefrom, were compared with those of the appropriate American grades. The French gums are freer from foreign suspended matter and  $\text{H}_2\text{O}$ , and give a slightly higher turpentine yield than the American. French turpentines have the lower initial b.p., but contain more of the higher-boiling components than the American. French rosins are clearer and freer from suspended matter than American, but have a greater tendency to crystallise, although this tendency is less than in the case of wood rosins. S. S. WOOLF.

**Shellac industry.** W. GARVIE (J. Oil Col. Chem. Assoc., 1931, 14, 324–339).

See A., Nov., 1227,  $\text{Al}_2\text{O}_3$  as ionising adsorbent. 1230, **Osmometer for lyophilic colloids.** 1231, **Solutions of cellulose derivatives.** 1263, **Structure [of paint films on metals].** 1297, **Pinabietic acid in Canada balsam.** 1299,  $\beta$ -Amyrin from Manila elemi resin.

## PATENTS.

**Coating compositions.** BAKELITE CORP. (B.P. 358,120, 5.7.30. U.S., 6.7.29).—Cellulose in a gelatinisable form, *e.g.*, cotton flock,  $\alpha$ -cellulose, is kneaded with a resin, preferably of the  $\text{PhOH-CH}_2\text{O}$  type, plasticised by the reaction product of a phenol and a fatty oil, *e.g.*, tung oil, in the presence of a promoter, *e.g.*,  $\text{H}_3\text{PO}_4$ , until the fibrous structure of the cellulose is destroyed. The activity of the resinoid may be retarded by incorporating cellulose acetate etc. The resulting product may be applied as a sheet veneer, *i.e.*, not in the form of a solution. S. S. WOOLF.

**[Coating] compositions containing organic esters [acetate] of cellulose.** CELLULOID CORP. (B.P. 358,428, 6.6.30. U.S., 8.6.29).—Flexible non-blooming coatings which do not become sticky at high temp. are obtained by incorporating 100% or more (on the wt. of the cellulose ester) of a relatively  $\text{H}_2\text{O}$ -insol. non-volatile tartaric ester, *e.g.*,  $\text{Bu}_2$  or  $\text{Ph}_2$  tartrate; triaryl ( $\text{Ph}_3$ ) phosphates are suitable plasticisers. E. LEWKOWITSCH.

**Manufacture of [resinous] condensation products from polybasic acids and polyhydric alcohols.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,491, 7.7.30).—The formation of insol. masses is restrained by effecting the condensation in the presence of 1–10% of  $\text{H}_3\text{BO}_3$  (its anhydride or esters, or borax). Drying oil fatty acids may be present. E. LEWKOWITSCH.

**Manufacture of [resinous] condensation products [from polyhydric alcohols and polybasic acids].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 352,981, 12.2.30).—In the condensation of glycerol etc. with phthalic anhydride etc., with or without fatty acids or oils etc., a  $\text{H}_2\text{O}$ -insol. org. liquid of b.p. between  $100^\circ$  and  $200^\circ$ , capable of forming a pseudo-azeotropic mixture with steam, is added and the mixture of org. liquid and  $\text{H}_2\text{O}$  is continuously distilled off. Suitable liquids are xylene, cyclohexanone,  $\text{BuOAc}$  and  $\text{Bu}_2\text{O}$ . C. HOLLINS.

**[Water-soluble resinous] derivatives of polyhydric alcohol-polybasic acid condensation products.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. BAIRD (B.P. 358,095, 3.7.30).—The resin component described in B.P. 356,738 (B., 1931, 1062) is treated with an alkali hydroxide or salt of a weak acid, *e.g.*,  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{S}$ , and dissolved in  $\text{H}_2\text{O}$ . The films obtained by drying at  $100^\circ$  are  $\text{H}_2\text{O}$ -sol. and may be rendered insol. by treatment with acids. E. LEWKOWITSCH.

**Non-crystalline highly chlorinated diaryl resins.** FEDERAL PHOSPHORUS CO., ASSEES. of R. L. JENKINS (B.P. 358,704, 24.9.30. U.S., 28.9.29).—Non-cryst. resins of high softening pt. are obtained by chlorinating mixtures of  $\text{Ph}_2$  with the high-boiling fraction obtained as a by-product in its synthesis. E. LEWKOWITSCH.

**Moulding of objects from synthetic resins.** M. M. EATON, and POLLOPAS, LTD. (B.P. 358,075, 4.6.30).—About 3% of a mixture of various plasticisers, *e.g.*, Plastol C 11, Mollite A, are added to a rather highly condensed urea- or thiourea- $\text{CH}_2\text{O}$  resin (powder) to assist the flow in moulding (*e.g.*, at 170 atm.). E. LEWKOWITSCH.

**Compositions for plastic moulding.** DR. THOMAS & Co. G.M.B.H. (B.P. 358,089, 1.7.30. Ger., 2.7.29).—Pentamethylenediaminedisulphine (obtained from  $\text{NH}_4\text{HS}$  and  $\text{CH}_2\text{O}$ ), enriched if desired with further S, is used as a binder for fillers, *e.g.*, albuminous substances, condensation products of the  $\text{PhOH-}$  or urea- $\text{CH}_2\text{O}$  types, wood pulp, asbestos, or as a plasticiser in conjunction with other binders, or as an accelerator in rubber vulcanisation. S. S. WOOLF.

**Atomiser.**—See I. Dispersions. Naphthazarin [pigment].—See III. Azo pigments.—See IV. Treatment of felt. Nitrated cellulose. Cellulose derivatives and fireproofing.—See V. Dynamo brush.—See XI.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Accelerated ageing of rubber with the aid of the mercury-vapour lamp.** L. GRAFFE (Caoutchouc et Gutta-Percha, 1931, 28, 15,714–15,718).—Four thin rubber samples of different qualities when exposed to the light from a Hg lamp showed an initial small loss in wt., attributed to removal of natural moisture, followed by an increase in wt. which was accompanied by a progressive increase in the proportion extractable by  $\text{COMe}_2$ . The rate of alteration induced by continued exposure to the radiation gradually decreased; in the early stages the accelerated change induced by the ultra-violet radiation continued unabated even when the illumination was discontinued. A screen of Wood's glass, which permits only ultra-violet radiation to pass, effectively protected the samples; visible rays near the ultra-violet region are probably involved in the ageing influence. D. F. TWISS.

## PATENTS.

**Processing of [rubber] latex.** R. F. ELDER and R. B. HILL, ASSRS. to BROWN CO. (U.S.P. 1,804,157, 5.5.31. Appl., 5.8.29).—Latex to which a creaming agent, *e.g.*, gum tragacanth, and vulcanising agents, such as S, ZnO, and Zn piperidinocarbothionolate, have been added, is heated, *e.g.*, to  $75^\circ$ , so as to induce concurrent creaming and vulcanisation. The vulcanised concentrate is preferably removed after about 24 hr. D. F. TWISS.

**Increasing the stability and filterability of [rubber] latex.** S. M. CADWELL and E. HAZELL, ASSRS. to NAUGATUCK CHEM. CO. (U.S.P. 1,802,753, 28.4.31. Appl., 22.4.27).—Increased mechanical stability and rate of filtration are obtained by the addition of an aldehyde (above  $\text{C}_3$ ), *e.g.*, 1% of cinnamaldehyde, preferably with previous introduction of a soap, *e.g.*, 1% of Na undecenoate. The effect develops in a few days and may be accelerated by heating at  $100^\circ$ . D. F. TWISS.

**Preservation of [rubber] latex.** A. A. NIKITIN, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,801,754, 21.4.31. Appl., 10.7.29).—Latex is preserved by the addition of a small proportion of a phenol and an aliphatic hydrocarbon such as kerosene. D. F. TWISS.

(A) Adhesive aqueous dispersions of matter [containing rubber]. (B) Treatment of colloidal substances. (C) Manufacture of aqueous dispersion

of matter [containing rubber]. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. (U.S.P. 1,801,621, 1,802,866—7, [A] 21.4.31, [B, C] 28.4.31. Appl., [A] 21.7.27, [B] 9.11.26, [C] 10.5.27).—(A) An adhesive of improved resistance to  $H_2O$  and to bacterial action is obtained by adding to rubber latex an alkaline-earth hydroxide, *e.g.*,  $Ca(OH)_2$ , a  $H_2O$ -sol. chloride, *e.g.*,  $NaCl$ , and a hydrophilic colloid, *e.g.*, casein, with other ingredients if desired. (B) Glue and rubber latex are mixed together with an agent such as  $K_2Cr_2O_7$  to render the glue insol.; the product, which may also include other ingredients, has adhesive properties. (C) Latex (100 pts.) of approx. 35% rubber content is mixed with casein (20 pts.),  $CaO$  (4 pts.),  $Na_2SO_3$  (4 pts.), and  $H_2O$  (70 pts.). D. F. TWISS.

**Manufacture of goods of or containing rubber or similar material.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., H. C. YOUNG, and C. HEMM (B.P. 358,011—2, 1.7.30. Cf. B.P. 353,656; B., 1931, 1020).—Goods of rubber or similar material provided with perforations are made (A) by applying an aq. dispersion to a surface provided with a number of suitable projections and thereafter removing the projecting portions of the applied layer, or (B) by electrodepositing the material from an aq. dispersion on to an electro-conducting base, *e.g.*, of Zn, provided with holes plugged with ebonite or other suitable non-conductor.

D. F. TWISS.

**Manufacture of masses resembling hard rubber.** I. G. FARBERIND. A.-G. (B.P. 358,115, 5.7.30. Ger., 5.7.29).—A mixture of natural rubber and artificial rubber-like material, *e.g.*, the Na polymerisation product of  $\alpha\gamma$ -butadiene, with other compounding ingredients as desired, is vulcanised. [Stat. ref.] D. F. TWISS.

**Compounding and vulcanising rubber.** W. A. STEINLE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,802,740, 28.4.31. Appl., 19.10.27).—A rubber composition containing an incomplete vulcanising combination, *e.g.*,  $ZnO$ , S, and  $NH(CH_2Ph)_2$ , is immersed in an aq. dispersion of the complementary vulcanising ingredient, *e.g.*,  $CS_2$ . The last-named diffuses into the mass, which then becomes readily vulcanisable. Tank linings of rubber may be vulcanised in this way. Colours, antioxidants, etc. dissolved in suitable rubber solvents and emulsified in  $H_2O$  may be introduced in an analogous manner.

D. F. TWISS.

**Making articles of rubber [bonded to metals etc.].** H. GRAY, Assr. to B. F. GOODRICH CO. (U.S.P. 1,801,667, 21.4.31. Appl., 22.4.27).—Rubber compositions are attached to metals and other relatively impervious materials by applying an intermediate coherent coating of an org. material, such as rubber solution, thermoplastic rubber derivatives, linseed oil, or asphalt, which when heated in a thin film harden by oxidation. The rubber compound is then applied, after previously coating the oxidised film, if desired, with a substance adsorbable by the film and by the rubber. The assembled article may then be vulcanised.

D. F. TWISS.

**Treatment of rubber [for preservation].** S. M. CADWELL and L. MEUSER, Assrs. to NAUGATUCK CHEM. CO. (U.S.P. 1,802,752, 28.4.31. Appl., 29.1.27).—Tri- and/or di-ethanolamine, or a derivative containing the

grouping  $N\cdot C\cdot C\cdot O\cdot$  is used as an "age-improver" for rubber. They do not lead to discoloration of the material or of paper wrapping.

D. F. TWISS.

**Treatment of rubber.** W. A. GIBBONS, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,802,761, 28.4.31. Appl., 9.12.26).—The deficient plasticity of rubber from alkali-preserved conc. latex can be prevented and a satisfactory product ensured by adding to the latex, already compounded if desired, an acidic substance such as  $H_3PO_4$  so as to bring the  $p_H$  to 7 or a little below without substantial coagulation, and evaporating until a dry mixture is obtained.

D. F. TWISS.

**Antioxidant [for rubber].** L. J. CHRISTMANN, Assr. to AMER. CYANAMID CO. (U.S.P. 1,800,435, 14.4.31. Appl., 21.8.28).—Tetra-alkyl-substituted benzidines, particularly the  $Me_4$  compound, assist the preservation of oxidisable org. materials such as rubber.

D. F. TWISS.

**Rubber-paper pulp.**—See V. Plastic moulding.—See XIII.

## XV.—LEATHER; GLUE.

**Pickling [of skins].** E. R. THEIS and A. W. GOETZ (J. Amer. Leather Chem. Assoc., 1931, 26, 505—545).—The amount of  $HCl$  which combined with bated pelt increased as the concentration of the  $NaCl$  in the pickle was increased, the  $NaCl$  effect being greatest when 0.125 mol. of  $HCl$  per 100 g. of bated pelt was used. The  $HCl$  was found to be combined with the pelt in accordance with the Donnan membrane equilibrium theory. The swelling of the pelt was retarded by the addition of  $NaCl$  and was in accordance with the second law of thermodynamics. Swelling was observed when the amount of diffusible ions in the external solution was less than that absorbed by the pelt; the pelt was depleted under the reverse conditions. At low acid concentrations in a pickle, the pelt hydration effect was that due to the  $NaCl$  in the pickle. Above 2.5%  $HCl$  the hydration effect was affected by both the  $HCl$  and the  $NaCl$  in the pickle, and less hydration was observed. At low acid concentrations the whole of the acid was combined by the pelt, and the latter was hydrated. Hydrolysis of the pelt was retarded by the addition of  $NaCl$ , but any coagulable proteins in the pelt were thereby dissolved.

D. WOODROFFE.

**Hydrolysis of chestnut- and quebracho-tanned leathers by sulphuric acid.** E. L. WALLACE (J. Amer. Leather Chem. Assoc., 1931, 16, 545—557; cf. B., 1931, 1021).—The hide substance is hydrolysed by the  $H_2SO_4$  in a leather and can be extracted as sol. N compounds. Leather tanned with chestnut extract was hydrolysed by  $H_2SO_4$  to a greater extent than that tanned with quebracho extract. The rate of hydrolysis of freshly tanned leather decreased as the degree of fannage decreased.

D. WOODROFFE.

**Jelly strength of skin, chrome leather, and bone glue and its relation to the viscosity and chromium content of the materials.** O. GERNGROSS and H. MENDEL (Kunstdünger u. Leim, 1931, 28, 109—116; Chem. Zentr., 1931, 1, 3081).—Chrome leather and bone glue possessed relatively higher jelly strength than skin glue of equal viscosity.

A. A. ELDRIDGE.

**Monel metal [vats].**—See X.

## PATENTS.

**Manufacture of tanning materials and preparations containing them.** L. POLLAK (B.P. 353,872, 23.4.30. Czechoslov., 23.4.29).—An aliphatic aldehyde is condensed with one or more polyhydric phenols (with or without vegetable tanning material) and urea or a urea derivative below 50°, preferably in presence of acid, and then heated to 90–100°, if desired. Products from urea with resorcinol and  $\text{CH}_2\text{O}$ , with pyrogallol and  $\text{CH}_2\text{O}$ , with resorcinol and  $\text{MeCHO}$ , and with resorcinol, quebracho extract, and  $\text{MeCHO}$ , are described.

C. HOLLINS.

**Tanning [agents].** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 353,046, 9.4.30).—Compounds of the formula  $\text{R}(\text{NH}\cdot\text{SO}_2\text{R}')_2$ , where R is an arylene or diarylene residue and R' is the C-radical of a di- or poly-nuclear arylsulphonamide or acylamino-compound, are used as tanning agents. Examples are:  $\text{NN}'$ -bis-(*m*-3:4-dichlorobenzenesulphonamidobenzenesulphonyl)-benzidine-2:2'-disulphonic acid; the corresponding derivative of 4:4'-diaminostilbene-2:2'-disulphonic acid;  $\text{NN}'$ -bis-(3:4-dichlorobenzenesulphon-*m*-amidobenzenesulphon-*m*-amidobenzenesulphonyl)benzidine-2:2'-disulphonic acid;  $\text{NN}'$ -bis-(*m*-4-hydroxy-3-carboxybenzenesulphonamidobenzenesulphonyl)benzidine-2:2'-disulphonic acid;  $\text{NN}'$ -bis-(*m*-naphthalene- $\alpha$ - or - $\beta$ -sulphonamidobenzenesulphonyl)benzidine-2:2'-disulphonic acid; etc. The two R' radicals may be the same or different.

C. HOLLINS.

**Hardening of gelatin films.** F. C. ARWOOD, Assr. to TECHNICOLOR MOTION PICTURE CORP. (U.S.P. 1,801,478, 21.4.31. Appl., 22.4.27).—Alkali (or  $\text{NH}_4$ ) dichromate is incorporated in the gelatin, which is hardened by treatment with a reducing gas, e.g.,  $\text{SO}_2$ . The cellulosic film backing may be treated with the gas before coating with the gelatin, development being effected by warming at 27–49°.

J. LEWKOWITSCH.

**Briquettes for boilers etc.**—See I. Dispersions.—See III. Stiffening [of leather].—See V. Adhesives containing rubber.—See XIV.

## XVI.—AGRICULTURE.

**Soil profile studies. III. Process of podsolisation.** J. S. JOFFE (Soil Sci., 1931, 32, 303–323).—Theories of podsol formation are critically examined and their relation to pan, gley, and brown earth production is discussed. The rate of decomp. and mineralisation of org. matter and the relation of the humates in anion exchange reactions (cf. Mattson, B., 1931, 173, 556) have an important bearing on the formation of podsols.

**Effects of vegetation and climate on soil profiles in N. and N.-W. Wyoming.** J. THORP (Soil Sci., 1931, 32, 283–301).—The classification of these soils according to the nature of their profiles is associated with areas of different average rainfall and characteristic vegetation. The presence of  $\text{CaCO}_3$  depends on rainfall and mean temp. The depth of the zone of accumulation of  $\text{CaCO}_3$  is that of the average moisture penetration and increases with rainfall up to 20 in. With greater rainfall the definite accumulation of  $\text{CaCO}_3$  disappears. Gypsum and salt accumulations disappear sooner than

that of  $\text{CaCO}_3$ . The depth of the heavy subsoil (upper B) horizon increases with increasing rainfall. This is the horizon of max. illuviation. The colour of the soils becomes darker with increasing proportions of grass in the vegetation, and this, in turn, depends on rainfall. Under similar climatic conditions forest vegetation produces an entirely different soil from that under prairie conditions. Where rainfall is <20 in. soils have  $p_H$  6.5 approx., and  $p_H$  >7.0 where rainfall is >20 in. Forested soils have a lower  $p_H$  than prairie soils with similar rainfall, max. vals. in both cases occurring in the C-horizons.

A. G. POLLARD.

**Intensive management of permanent pasture in dairy farming.** R. M. SALTER and R. E. YODER (Ohio Agric. Exp. Sta. Bimonthly Bull., 1931, No. 152, 155–163).—The increased production of protein and dry matter obtained by the use of fertilisers in an intensive grazing system is produced more economically than by supplementing cattle feeds with concentrates.

A. G. POLLARD.

**Humus as a source of energy for soil micro-organisms and the energy liberated by their activity.** F. H. VAN SUCHTELEN (J. Bact., 1931, 21, 33–34).—The heat of combustion of humus varied from 4.7 to 5.4 kg.-cal. per g. The heat liberated during the bacterial decomp. of humus is a measure of the availability of the humus. In laboratory experiments the heat liberation reached a const. val. after 300–400 hr. The conception of humus as a source of energy for soil micro-organisms is discussed.

A. G. POLLARD.

**Molasses as fertiliser.** ANON. (S. Afric. Sugar J., 1931, 15, 521–523).—In order to establish the stimulating value of the sugar present in molasses, a series of plots were cultivated in Java with cane after having received, besides the usual amount (250 kg. per acre) of  $(\text{NH}_4)_2\text{SO}_4$ , the following additions: (1) molasses, 10 tons per acre; (2) sugar, 1920 kg. per acre; (3) N (as  $(\text{NH}_4)_2\text{SO}_4$ , equiv. to that present in the molasses); and (4) as in the previous series, but also potash equiv. to that in the molasses. Two years' results showed the molasses plots to yield 11–12 tons of cane per acre more than any other, and also proved the sugar to be the constituent of the molasses to which the superior yield was due.

J. P. OGILVIE.

**Absorption of ammonium and nitrate by the root of *Zea mais* seedlings in relation to the concentration and acidity of the culture solution.** T. L. LOO (J. Fac. Agric. Hokkaido, 1931, 30, I, 1–118).—At high concentrations only  $\text{NH}_4^+$  is at first absorbed,  $\text{NO}_3^-$  being absorbed later; at low concentrations both ions are absorbed. If the concentration of  $\text{NH}_4\text{NO}_3$  is much less than that of other salts,  $\text{NO}_3^-$  is absorbed more readily than  $\text{NH}_4^+$ .  $\text{NH}_4^+$  is absorbed more readily at low acidity and  $\text{NO}_3^-$  in slightly acid solution; depressions in the absorption curve are observed at  $p_H$  5.1–5.4, 6.1–6.4, 7.0–7.2, and 7.8–7.9.

CHEMICAL ABSTRACTS.

**Fixation of nitrogen by leguminous plants under bacteriologically controlled conditions.** P. W. WILSON, E. W. HOPKINS, and E. B. FRED (Soil Sci., 1931, 32, 251–269).—Nodule organisms of clover and lucerne

fixed N under sterile conditions. Atm. contamination did not affect the process. The amount of N fixed depended on the general growth of the plant. In plants from unsterilised seed or inoculated with unsterilised nodule cultures N fixation was greater than under sterile conditions. Fixation in various strains of clover was examined. A. G. POLLARD.

**Causes of reduced yields and starch contents of potatoes produced by different potash fertilisers. II. Chemical investigations.** S. GOV and RUDOLPH (Z. Pflanz. Düng., 1931, 10B, 457—496).—The intake of K<sup>+</sup> and Cl<sup>-</sup> by potatoes is very high in comparison with that of the other constituents of common potash fertilisers (Mg<sup>++</sup> and SO<sub>4</sub><sup>==</sup>). High Cl<sup>-</sup> intake is associated with high H<sub>2</sub>O intake. The greater yield of potatoes obtained from K fertilisers rich, as compared with those poor, in Cl<sup>-</sup> is attributed to the higher H<sub>2</sub>O content of the tubers. K<sub>2</sub>SO<sub>4</sub>, but not KCl, is physiologically acid in respect of potato crops. A. G. POLLARD.

**Changes in composition of soya beans towards maturity as related to their use as green manure.** W. A. ALBRECHT and W. H. ALLISON (Soil Sci., 1931, 32, 271—282).—The efficiency of soya bean as a green manuring crop depends to a large extent on the stage of growth attained. The % of total N and easily nitrifiable and H<sub>2</sub>O-sol. constituents decreased with age. The proportion of alkali-sol. constituents (slowly decomposable) increased rapidly in the later stages of growth. The N content of this fraction, especially of the alkali-sol. lignin, decreased with age. Reducing sugars, cellulose, and pentosans increased with maturity. The % of total lignin increased rapidly in both roots and tops. The narrow pentosan : lignin ratio indicated low decomposability, especially in the roots. The C : N ratio of the crop is sufficiently wide to involve the risk of a N deficiency in crops following green manuring with soya bean. A. G. POLLARD.

**Control of "damping-off" of flower seedlings.** P. E. TILFORD (Ohio Agric. Exp. Sta. Bimonthly Bull., 1931, No. 152, 167—175).—Admixture of "formalin dust" (15 pts. of formalin + 85 pts. of adsorptive carrier) with soil prior to sowing seed prevents "damping off" and does not injure seeds. A. G. POLLARD.

**Effect of time and rate of application of seed disinfectants on oats and wheat.** B. KOEHLER (Phytopath., 1931, 21, 127).—Storage of seed oats after treatment with "formalin dusts" reduced grain yields as compared with seed sown 24 hr. after treatment. A. G. POLLARD.

**Use of oxides of unsaturated hydrocarbons for the eradication of barberries and other pests.** R. B. HARVEY (Phytopath., 1931, 21, 126).—Ethylene oxide diffuses rapidly in soil and into the plant roots, causing death in 1—2 weeks. Methods of injection are described, and the use of propylene oxide is examined. A. G. POLLARD.

**Organic mercury compounds for the control of scab and *Rhizoctonia* in potatoes.** B. A. BROWN (Connecticut [Storrs] Agric. Exp. Sta. Bull., 1930, No. 164, 87—106).—The average yield of potatoes receiving standard treatments with HgCl<sub>2</sub> or CH<sub>2</sub>O were slightly higher than when org. Hg compounds were

used. For *Rhizoctonia* control the efficiency of the treatments was in the order HgCl<sub>2</sub> > hot CH<sub>2</sub>O > org. Hg compounds. Cold CH<sub>2</sub>O treatment was ineffective for scab. A. G. POLLARD.

**Control of powdery mildew and red spider on greenhouse cucumbers.** L. J. ALEXANDER and H. C. YOUNG (Science, 1931, 74, 314—315).—Hydrophilic colloidal S spray (2 lb. per 100 gals. of H<sub>2</sub>O) is an effective and safe control. L. S. THEOBALD.

**Esthonian phosphorite.**—See VII. Milk for cheesemaking.—See XIX.

See also A., Nov., 1258, Determination of H<sub>3</sub>PO<sub>4</sub> by filtration. 1266, Forest types in the south-west. Chernozem-like soils of N. Dakota. Arctic and Norwegian soils. 1339, Influence of soil moisture on plant growth. 1340, Effect of Shive's nutrients on rice seedlings. Effect of S and P on plant nutrition. Tobacco plant. Production of alkaloids in plants.

#### PATENTS.

**Production of phosphate fertilisers.** W. W. TRIGGS. From G. OBER & SONS Co. (B.P. 358,529, 7.4.30).—In the process described in B.P. 357,508 (B., 1931, 1114), the rate of flow of the mixture to the autoclave is regulated according to the reactivity and temp. of the acid and the dust, to the fineness of the dust, and to the concentration of the acid. L. A. COLES.

**Plant stimulant.** G. WESENBERG and E. BISCHOPFF, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,803,157, 28.4.31. Appl., 4.2.27. Ger., 27.3.26).—A thionic acid salt, preferably Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, is applied in solution or mixed with, e.g., kieselsguhr to seed potatoes, Jerusalem artichokes, etc. L. A. COLES.

**NH<sub>4</sub> sulphate nitrate.** Colloidal suspensions containing Cu.—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Composition of beets and juice of abnormally high purities.** J. BRZEK (Z. Zuckerind. Czechoslov., 1931, 56, 73—77).—Working with the very pure juice of the Semcicer beet, CaO was found to exert a relatively greater purifying effect than with the ordinary juice corresponding to the average of the past 10 years. Thus 5.9% more non-sugars, 9.9% more total N, and 21.06% more melassigenic N were removed. J. P. OGILVIE.

**Colloid content of cane-mill juices.** M. R. MONSOUUD (Philippine Agriculturist, 1931, 20, 53—74).—Dye-val. tests made on the crusher juice gave an average val. of 762 with means of 331 and 1191. In the 2nd—4th mill juices the colloids increased in proportion to the amount of maceration H<sub>2</sub>O added. Using the ordinary defecation process of clarification, about 15% of the colloids were removed. J. P. OGILVIE.

**Increase of the polarisation of beet pulp during its preparation due to evaporation.** O. KOPECKÝ (Z. Zuckerind. Czechoslov., 1931, 56, 81—91).—As the result of the evaporation of H<sub>2</sub>O from the sample of beet during its pulping and mixing, the polarisation, as determined according to the official method of the Czechoslovakian beet factories, is increased 0.1—0.2%, and sometimes higher. J. P. OGILVIE.



**Hot-liming [of cane juice] in Java.** A. BUSSEMAKER (Arch. Suikerind. Nederl.-Indië, 1931, 39, 69—81).—Trouble was experienced in turning out an acceptable white sugar, the product having a greenish tinge; this difficulty disappeared when a procedure involving hot-liming was followed. Raw juice was heated to the b.p. and limed with saccharate in such a manner that admixture of the two liquids was almost instantaneously effected. All the advantages of a particularly well-clarified juice were then observed with cleaner evaporator tubes, easier boiling, and better centrifugal work. No glucose decomp. had taken place, the molasses sugar having a golden-yellow colour with no tinge of green.

J. P. OGILVIE.

**Decomposition of alkaline sugar solutions at high temperatures.** II. O. SPENGLER, E. LANDT, and J. OST (Z. Ver. deut. Zucker-Ind., 1931, 81, 487—500; cf. B., 1931, 509).—Numerous experiments similar to those of the previous study were made, except that the solutions were rendered alkaline with  $\text{Na}_2\text{CO}_3$  instead of with  $\text{CaO}$ , and only two alkalinities were employed corresponding to 0.1 and 0.2%  $\text{CaO}$ , as determined by titration hot in presence of phenolphthalein. The conclusions drawn previously are confirmed in general, but not in all respects. The increase in colour with duration of heating was progressive even when the solutions became acid, no max. of colour near the neutral point and subsequent lightening being observed. Coloration was more rapid in the more alkaline than in the less alkaline solutions, and with other factors const., but for short periods of heating and at low temp. (110° and 120°) the differences were very slight. The fall in polarisation showed some anomalies, being in some cases greater for the less alkaline than for the more alkaline solutions, and in some cases greater for 30% than for 50% sugar solutions at temp. of 110° and 120°. The previous conclusion that a rise of temp. of 10° trebles the rates of deteriorative changes was confirmed in general, but with more exceptions than in the earlier study.

J. H. LANE.

**Juice purification by Teatini's process.** J. VON DRÁK (Z. Zuckerind. Czechoslov., 1931, 56, 57—66).—Laboratory experiments showed juice defecated with 1% of  $\text{CaO}$  added in one dose to filter badly, whereas the same juice heated to 85° and then limed in two stages, first with 0.1% and a few min. after with 0.9%, filtered much better. Pre-defecation in the cold (i.e., at 20°) did not give good filtration results, and juice so treated re-coloured notably in subsequent working. Only in one instance did the Teatini process lead to a more favourable filtration than did simple fractional liming, using the same amount of  $\text{CaO}$ . Sometimes it gave juice of worse filtering quality, but results appear to depend on the type of juice worked.

J. P. OGILVIE.

**Filterability of raw cane sugars.** I. Effect of various factors prior to pan-boiling. II. Effect of pan-boiling operations. J. C. KEANE and H. G. HILL (Ind. Eng. Chem., 1931, 23, 421—427).—I. Filterability was measured by the rate of filtration of solutions of 50° Brix containing Hyflo Super Cel, through a Dawson laboratory pressure filter fitted with twill filter-cloth, at 85° and under 20 lb./sq. in. pressure. The filterability of raw cane sugars could not be generally

correlated with any other single easily measurable value, although in many cases, especially for samples from the same factory, it appeared definitely related to the colloid content as determined by the dye test. Pulverisation of 6 representative samples of raw sugar increased the average filterability by 54%. That suspended matters are to some extent responsible for poor filtration was shown by prefiltration of defecated juices, which doubled the rate of filtration of the resulting syrups. By adding  $\text{H}_3\text{PO}_4$  to raise the  $\text{P}_2\text{O}_5$  content of raw juice from 0.016 to 0.027%, and liming always to  $\text{pH}$  8.5, the filterability of the resulting syrups and sugars was much improved. In spite of a general relation between the filterability of syrups from factories in different districts and the  $\text{P}_2\text{O}_5$  content of the raw juices, there are exceptions due probably to a part of the  $\text{P}_2\text{O}_5$  not being available for combination with  $\text{CaO}$  in defecation.

II. Well-filtering syrups are essential to the production of sugars of good filterability, but they may yield poorly-filtering sugars as the result of faulty design or operation of vac. pans. Syrups from different factories, boiled in the same laboratory pan, yielded sugars corresponding fairly with their respective syrups in filterability; yet the sugars produced in the factories themselves did not show this general correspondence with their parent syrups. The coil pans for which data were obtained yielded better-filtering sugars than the calandria pans, probably owing to better circulation of the syrup, but this difference may not apply generally.

J. H. LANE.

**[Sugar juice] carbonatation studies.** III. Parallel filtration. Influence of  $\text{CO}_2$  pressure and of temperature on their filterability. L. DOSTAL (Z. Zuckerind. Czechoslov., 1931, 56, 25—31; cf. B., 1931, 900).—Two beet juices, one filtered at 70° and the other at 95°, were saturated at 95° to the optimum alkalinity, boiled up, and refiltered. Both were found to have almost the same purity, the former being (at the same  $\text{CaO}$  content) only 0.08% higher than the other. A high  $\text{CO}_2$  gas pressure was found to act favourably on the rate of filtration.

J. P. OGILVIE.

**Sulphuring middle thick-juice [in beet sugar manufacture].** K. SOLON (Centr. Zuckerind., 1931, 39, Festausg., 493—496).—Immediately after sulphuring the thick-juice, 91.1% of the total amount of S used was found to be present as sulphite and 4.9% as sulphate. Of the total amount of sulphite in the thick-juice, analysis of the end-products accounted for 9.21% as sulphite, 6.98% as sulphate, and 30.60% in the form of other S compounds—a total of 46.79%. The remainder must have gone into the incrustations.

J. P. OGILVIE.

**Prospects and advantages of rotary filters in sulphitation [cane sugar] factories.** W. H. FOSTER (Proc. S. Afr. Sugar Tech. Assoc., 1930, 71—76).—Owing to the character of the Uba cane grown in S. Africa about 2% of the total sugar is commonly lost in the filter-press cakes. A second filtration of the cakes in presses will recover most of this sugar, but at a purity so low as to retard boiling and curing of the lower product sugars. A recovery of over 1% of sugar, at a considerably higher purity, was attained by employing a Mauss continuous rotary vac. filter for the

second filtration after the cakes had been made into a sludge containing 15–19% of solids. The rotary filter is not so well adapted for the direct filtration of defecation settlings, as these contain much more liquid, but results obtained so far are promising. J. H. LANE.

**Determination of the sugar content of exhausted beet slices.** A. DOLINEK (Z. Zuckerind. Czechoslov., 1931, 56, 47–48).—A comparison was made of the rapid-pressure method, using the "Kosmos" apparatus, and of the hot-and-cold digestion methods at present in general use. In 159 determinations an average difference of only 0.01% was recorded. The author recommends that this rapid method be included among the official methods for use in Czechoslovakian sugar factories. J. P. OGILVIE.

**Sucrose determination in sugar factory products by double-polarisation methods.** B. B. BHATTACHARJEE and J. H. HALDANE (Internat. Sugar J., 1931, 33, 441–446).—Especially with low-purity products, as waste cane molasses, the Clerget-Herzfeld method and that of Jackson and Gillis (B., 1920, 634 A) gave results with a high positive divergence from the standard invertase method. On the other hand, Deerr's method (B., 1915, 503) with modifications by Coates and Shen (B., 1928 423) gave results only slightly below those determined by means of invertase, which latter method, however, is too long for ordinary control purposes. Deerr's method is therefore recommended for factory routine work. J. P. OGILVIE.

**Molasses as fertilisers.**—See XVI. **Raspberry syrup.**—See XIX. **Sugar cane mill effluents.**—See XXIII.

See also A., Nov., 1242, **Inversion of sucrose by tartaric acid.** 1274, **Determination of reducing sugars.** **Hydrazones and osazones of sugars.** 1275, **Effect of concentration of dextrose on its rotation.** 1277, **Polyamyloses in starch.** **Iodometric determination of starch.** 1341, **Determining starch in plants.**

#### PATENTS.

**Purification of the juices of sugar works and of refineries.** D. TRATINI (B.P. 351,160, 1.5.30. Belg., 15.2.30).—The colloids are flocculated by treating raw juice at 80–90° with CaO sufficient to produce an alkalinity in excess of that corresponding to the  $p_H$  of the isoelectric point, and then reducing the alkalinity to that corresponding to the isoelectric point by introduction of gaseous or liquid  $SO_2$ . Whether or not the flocculate is removed at this stage, the juice may be further limed and carbonated, but the total consumption of CaO is much less than in the ordinary carbonation process. Juice which has passed through the process and been limed prior to carbonation may be used for the initial alkalisation of raw juice. J. H. LANE.

**Affining raw sugar.** J. WIESNER (B.P. 352,619, 9.5.30).—Raw sugar, loosened, warmed, and moistened, is distributed evenly over the walls of a rotating centrifuge, and there treated with successive syrup washes, e.g., with 50% of a syrup of purity 66–73, 25% of syrup of purity 78–88, and finally 10% of a syrup of purity 99. The sugar is introduced into the centrifuge through an open-ended vertical tube supported by a fixed bracket

in which it is movable up and down by rack and pinion. The sugar is delivered to the enlarged upper end of the tube from a supply chamber above, and ejected horizontally from the lower end inside the centrifuge, whilst the tube is moved up and down to distribute the charge evenly from top to bottom of the centrifuge walls. The ejection of the sugar from the tube may be effected by a mechanical device operated by a rotating shaft within the tube, or by means of jets of compressed air. J. H. LANE.

#### XVIII.—FERMENTATION INDUSTRIES.

**Souring of sliced potatoes.** G. RUSCHMANN and G. GRÄF (Z. Spiritusind., 1931, 54, 263).—Surplus potatoes are converted into silo-fodder by the lactic acidification process. Sliced potatoes are filled into silos and inoculated with lactic acid bacteria. "Foaming" fermentation, which is due to other micro-organisms and prevents the full use of the silo space, is checked by a vigorous lactic fermentation. A high sugar content of the potatoes favours the lactic fermentation, and if the sugar content is deficient molasses is added to the potatoes. The addition of 2.5–3% of turf to the potatoes favours the acidification by excluding the air and by absorbing the molasses and the sap from the potatoes. Straw is used only as bottoming, as it activates a "foaming" fermentation which is not checked by vigorous acidification. Pre-draining of the potatoes neither inhibits the "foaming" fermentation nor activates the lactic fermentation. C. RANKEN.

**Freezing of wine.** H. MARCELET and S. BESPALOFF (Ann. Falsif., 1931, 24, 353–355).—By keeping wine at –12° to –14° and removing the ice each evening and morning until half the original vol. had been withdrawn, the EtOH content was raised from 9.9° to 14° (at 15°). The frozen fraction had 5.5° EtOH. When a centrifuge was used to drain the liquid from the ice crystals, the vol. being reduced by 50% as before, the EtOH contents of the unfrozen and frozen fractions were 18.05° and 0.9°, respectively. A fraction of "strength" 22.9° was obtained by withdrawing four fifths of the original vol. as ice. Wine so treated is mellowed and its bouquet improved. W. J. BOYD.

**Raisin wine. II.** L. CHAUVEAU and A. VASSEUR (Ann. Falsif., 1931, 24, 341–345; cf. B., 1929, 793).—Liquors from raisins may be distinguished from wines made from fresh grapes by their characteristic blue fluorescence in ultra-violet light and by Gautier's reaction as carried out by Moredod (B., 1930, 212). Liqueurs cannot be distinguished thus from raisin piquettes, but are otherwise readily distinguishable. Wines made from the fresh grapes of diseased vines or from grapes partly dried on the vine by the sirocco may give faintly positive results with the Gautier test. W. J. BOYD.

**Glycerin content of Mascara wines.** C. BERTIN (Ann. Falsif., 1931, 24, 350–352).—In comparison with other wines, many genuine Mascara (Algiers) wines have abnormally high glycerol content and glycerol/EtOH ratio. This bears no relation to the age or the  $SO_2$  content of the wine. W. J. BOYD.

**Detection of whortleberry juice by a form of Plahl's reaction which is also suitable for sweet**

**wines.** R. OFNER (Chem.-Ztg., 1931, **55**, 666).—Plahl's test (A., 1907, ii, 204) is applicable only to fully fermented wines. In the case of sweet (or sweetened) wines, 100 c.c. are decolorised by boiling with activated charcoal, the evaporated  $H_2O$  being replaced. The C is recovered, washed with hot  $H_2O$ , and the colouring matter redissolved by boiling with dil. alkali (5 c.c. of 2*N*-NaOH in 100 c.c. of  $H_2O$ ) for 2 min. After filtering, 40 c.c. of the cold solution are treated with 2 c.c. of *N*-HCl and 2 c.c. of aq.  $Pb(OAc)_2$  and filtered. Then 10 c.c. of the filtrate (which should be colourless) are mixed with 1 c.c. of conc. HCl and boiled. The appearance of a blue coloration after  $\frac{1}{2}$  min. indicates the presence of whortleberry juice. H. J. DOWDEN.

**Detection of fruit wines in grape wines by Werder's sorbitol method.** J. SCHINDLER and J. KOZÁK (Chem. Listy, 1931, **25**, 369—373).—The above method yields good results when the dibenzylidenesorbitol is transformed by Jahr's method (B., 1930, 787) into the hexa-acetyl derivative. R. TRUSZKOWSKI.

**Effect of sulphur dioxide on the determination of volatile acidity of wine.** J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1931, **24**, 345—349).—The presence of  $SO_2$  in wines in quantities within the permitted limit gives high results for the volatile acidity as determined by the Mathieu and the Denigès-Cazenave methods, respectively. If *n* c.c. of 0.1*N*-NaOH are equiv. to the acidity of the distillate from 10 c.c. of wine and *n'* c.c. of 0.1*N*-I are equiv. to the  $SO_2$  present in the distillate, the volatile acidity of the wine after correction for that due to  $SO_2$  is  $0.49(1.1n - n')$ , expressed as g. of  $H_2SO_4$  per litre of wine. Lactic acid present normally in many wines has only a negligible disturbing effect. Mathieu's process is more adaptable to serial determinations than the Denigès-Cazenave method. W. J. BOYD.

See also A., Nov., **Determination of  $H_2O$  content by distillation.** 1277, **Prep. of glycogen from yeast.** 1331, **Amylases of malt and potato.** **Determination of enzyme concentration.** Amylosynthase. 1333, **Formation of citric acid by moulds.** 1334, **Bacteria producing trimethylene glycol.** **Lactic-acetic fermentation.** 1341, **Enzymes of *Gibberella saubinetii*.**

## PATENT.

**Apparatus for pasteurising beer or other liquids.** R. F. GRAHAM (B.P. 358,220, 5.9.30).—A platform or carrier for the liquid containers is furnished with a short cam (extending at an angle to the edge of the carrier) which engages and pushes the containers across the rotating carrier a distance equal to the diam. of the container. Sprays of pasteurising liquid are directed on to the containers during their passage across the carrier. C. RANKEN.

## XIX.—FOODS.

**Hardening of "moti" (a Japanese rice-food).** T. MAEDA and R. SYÔZI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, **16**, 261—275).—Rice paste, prepared by grinding boiled rice, hardens on keeping, due to evaporation of  $H_2O$  from the surface and to binding of free  $H_2O$  in the interior of the mass by starch particles. In

this respect the hardening of "moti" is analogous to the setting of cement. R. TRUSZKOWSKI.

**Employment of milk for production of cheese.** K. STÖREN (Tidsskr. Kjemi Berg., 1931, **11**, 81—84).—The importance for the cheese industry of determining the relationship between the fat and albumin content of the milk used is emphasised. The formula  $p = 0.395f + 1.642$  ( $f$  = fat content) has been deduced to express this, but affords only approx. results. This relationship enables the optimum proportions in which whole and skimmed milk must be blended so as to obtain a cheese of adequate fat content to be ascertained. The most valuable milk for cheese production is one rich in fat and relatively poor in albumin, and attention is directed to the possibility of developing a breed of cows to give milk exclusively of the above type. H. F. HARWOOD.

**Determination of chloride in milk.** A. BECKEL (Chem.-Ztg., 1931, **55**, 818).—The milk (11 c.c.) is treated with 10 c.c. of  $AgNO_3$  solution (15.81 g. of  $AgNO_3$ , 350 c.c. of conc.  $HNO_3$ , and 20 g. of Fe alum in 2 litres) and titrated with aq.  $NH_4CNS$  (2.3613 g./litre), using a burette graduated in c.c. in which the divisions 0, 1, 2, 3, etc. are marked 150, 140, 130, etc. The reading of the burette then gives directly mg. of Cl' per 100 c.c. of milk. A. R. POWELL.

**Fat losses in buttermilk.** W. B. COMBS and S. T. COULTER (Minnesota Agric. Exp. Sta. Bull., 1930, No. 273, 30 pp.).—Fat determinations in buttermilk by the Babcock method are low, and by the Röse-Gottlieb method high, in comparison with those by the Minnesota method (B., 1930, 1128). In creamery practice, fat losses in buttermilk are high in spring and early summer and are attributed to seasonal conditions, temp. of churning, and temp. of storage prior to churning. A. G. POLLARD.

**Determination of caffeine in "decaffeinated" coffee.** K. BORGMANN (Süddeut. Apoth.-Ztg., 1931, **71**, 56; Chem. Zentr., 1931, i, 2692—2693).—The roasted coffee (20 g.) is moistened, mixed with sand, and extracted (Sohxhlet) with  $CCl_4$ , the extract being evaporated to dryness after addition of solid paraffin (1 g.). The residue is treated in portions with 125 c.c. of hot  $H_2O$ , and the filtrate, after cooling to 20°, is treated with 30 c.c. of 1%  $KMnO_4$ . After 15 min. 3%  $H_2O_2$  acidified with AcOH is added; the mixture is heated on the water-bath for 15 min., filtered, and evaporated. The residue is dried at 100° and extracted with  $CHCl_3$ , the residue left on evaporation of the  $CHCl_3$  being weighed. "HAG" coffee contains 0.086% of caffeine. A. A. ELDRIDGE.

**Soya-bean cake as protein supplement of poultry feed.** K. SUZUKI and T. HATANO (Proc. IV World's Poultry Cong., 1930, 288—291).—The substitution of soya-bean cake for fish meal had no effect on the growth of chicks, number of eggs, or hatchability. CHEMICAL ABSTRACTS.

**Fodder conservation.** A. KROCZEK (Z. Zuckerind. Czechoslov., 1931, **56**, 95—102).—A comparative study has been made of the formation of AcOH, lactic and butyric acids in trenches and cement silos in the cases of beet leaves, clover, lucerne, maize, and the results are tabulated. The effect of these 3 acids on animal organisms is discussed. J. P. OGILVIE.

**Production of non-crystallising raspberry syrup, and rapid determination of its invert sugar content.** V. STANĚK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1931, 56, 41—46; cf. B., 1930, 76).—On boiling fermented raspberry syrup with sugar both taste and aroma are improved, sharp-tasting aromatic substances being thus volatilised. Most of the invert sugar formed during the production of the syrup is formed during cooling in the vats. A non-crystallising product, *i.e.*, one containing about 20% of invert sugar, is obtained by first cooling the syrup to about 50° before filling it into the vats. A method for determining the approx. content of invert sugars is outlined; it depends on the reduction of  $K_3Fe(CN)_6$  and methylene-blue in an alkaline medium. J. P. OGILVIE.

**Corrosion of tinfoil as used for making containers for conserves.** G. GIRE (Ann. Falsif., 1931, 24, 355—362).—The actions of 0.1*N*-AcOH, and of 0.04*N*, 0.02*N*, and 0.01*N* solutions of oxalic, tartaric, citric, malic, and lactic acids on tinfoil, under similar conditions to those obtaining in conserve containers, have been studied with reference to regularity of corrosion and the quantities of Sn and Fe passing into solution. W. J. BOYD.

**Souring of potatoes.**—See XVIII.

See also A., Nov., 1938, **Determination of vitamin-B.** 1341, Tea-seed oil.

#### PATENTS.

**Manufacture of substances of dietary and therapeutic value.** F. F. TISDALL (B.P. 357,223, 18.6.30. Can., 24.1.30).—A foodstuff containing vitamin-A, -B, -B<sub>2</sub>, -D, and -E in addition to Cu and P is prepared from a mixture of whole wheat, wheat germ, yeast, bone meal, and raising agents, the biscuit being cooked at 150° for 15—20 min. The effects of heat, alkalis, and acids on the vitamins are described. E. B. HUGHES.

**Disinfection of cereals, fruits, foodstuffs, textiles, etc.** R. M. CABRERA (B.P. 357,127 and Addn. B.P. 357,200, 16.5.30).—The vapour of (A) CS<sub>2</sub>, or (B) CCl<sub>4</sub>, HCN, or H<sub>2</sub>S, is circulated in a closed system over the materials to be disinfected. E. B. HUGHES.

**Production of synthetic cream rich in fat and having high whippable qualities.** H. C. HEIDE. From INTERNAT. GRADIN A.-G. (B.P. 357,246, 20.6.30).—Groundnut oil hardened to have m.p. 32° is sprayed at about 70° into milk containing an emulsifying agent such as egg yolk. Additional heated milk is sprayed into the bulk at the same time as the oil. The final product is claimed to contain 35—43% of fat. E. B. HUGHES.

**Pasteurisation of dairy products.** J. H. BAUMGARTNER, Assr. to C. and H. DOERING (U.S.P. 1,802,455, 28.4.31. Appl., 10.12.28).—Plastic dairy products, such as butter and cheese, are forced through constricted spaces. The friction develops heat sufficient to effect pasteurisation. Syrupy flavouring materials may be added during the process. Apparatus is described. E. B. HUGHES.

**Preparing edible emulsions of solid in fat.** L. B. ESOMOND and W. W. DUECKER (U.S.P. 1,800,985—7, 14.4.31. Appl., 6.3.29).—Granulation and blooming of, *e.g.*, chocolate coatings are reduced by dispersing

gelatin in the emulsion by (A) milling a portion of fat, *e.g.*, cacao butter, with dry, powdered gelatin and sufficient H<sub>2</sub>O to swell it, before adding the bulk of the chocolate liquor, and heating at 60° to drive off H<sub>2</sub>O; (B) grinding an aq. gelatin solution into a portion of heated chocolate liquor, and heating to 104° to remove H<sub>2</sub>O, before milling in the rest of the chocolate (or fat); and (C) boiling the cacao nibs to dryness with aq. gelatin (with or without sugar) before working up.

E. LEWKOWITSCH.

**[Spray-]drying of fruit juices.** C. B. VON BÖRNEGG, Assr. to CALIFORNIA FRUIT GROWERS' EXCHANGE (U.S.P. 1,800,501, 14.4.31. Appl., 27.2.25. Ger., 24.3.24).—The addition of a small proportion of gum arabic, gum tragacanth, or agar agar to fruit juices prior to spray-drying results in the dried product being non-hygroscopic. E. B. HUGHES.

**Conservation or preservation of meat.** L. SENTCHENKO (B.P. 358,158, 16.7.30. Fr., 26.8.29).—A canula, with conical ferrule of adjustable diam., is described for introducing conserving solution into the aorta of a slaughtered animal. E. B. HUGHES.

**Production of protein products.** W. B. WESCOTT, Assr. to AMER. PROTEIN CORP. (U.S.P. 1,804,042, 5.5.31. Appl., 5.12.22. Renewed 6.2.30).—Unlaked blood is secured by slaughtering beef animals with a special butchering knife. The plasma is obtained free from the corpuscles and haemoglobin, using a modified centrifuge, and may be used as such or in a dried form as general food base. E. B. HUGHES.

(A) [Apparatus for] chilling, freezing, and defrosting of foodstuffs. (B) Preserving, packing, and heating foodstuffs [retail packages of preserved raw flesh or meat]. M. T. ZAROTSCHENZEFF, and LIVERPOOL REFRIGERATION CO., LTD. (B.P. [A] 358,596 and [B] 359,005, [A] 15.7.30, [B] 9.7.30).

**Drying of fresh and salted fish [*in vacuo*].** N. ANTHONISEN and H. C. M. INGEBERG (B.P. 359,306, 24.12.30. Norw., 28.12.29).

**Grinding chocolate.**—See I. Fat from cacao bean. Oil from fruits.—See XII. Pasteuriser.—See XVIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Examination of tinctures.** L. W. WINKLER (Pharm. Zentr., 1931, 72, 641—644).—The vol. of tincture which must be added at 20° to 1 c.c. of CHCl<sub>3</sub> to obtain a clear solution is sp. for tinctures made up from EtOH of the same concentration. Tables of CHCl<sub>3</sub> vals. are given. S. I. LEVY.

**Chemical examination of pyrethrum.** J. RIPERT (Ann. Falsif., 1931, 24, 325—341).—Determination of chrysanthemic acids in pyrethrum by saponification with alkali, acidification, and extraction of the acid volatile in steam and the other non-volatile acid sol. in light petroleum, these fractions being titrated with 0.02*N*-NaOH, is subject to large errors owing to the presence of fatty acids. Methods based on determination of the N content of the isolated semicarbazide of the pyrethrolone and those based on the reducing properties of the pyrethrolone are reviewed. An objection to the use of reduction methods is the possibility of adulteration of

the product with reducing substances. Physiological methods are useful but inaccurate. The plant contains 0.1—0.4% of methylpyrethrolone, which although non-toxic is determined along with the pyrethrins by the methods mentioned.

W. J. BOYD.

**Caffeine in coffee.**—See XIX.

See also A., Nov., 1259, Determination of Ag in colloids and org. compounds. 1287, *p*-Acetamidophenylurethanes as antipyretic. 1289, Monoethers of resorcinol and *sym.* org. sulphides as bactericides. 1290, Condensation of olefinic acids and C<sub>6</sub>H<sub>6</sub> rings. 1311, Synthetic corydine. 1312, Synthetic sinactine. *Strychnos* alkaloids. Test for morphine. 1313, Alkaloids of *Cocculus trilobus*. Alkaloids of pereiro bark. 1314, Precipitation of alkaloids in gels. 1314—15, Arsinic acids. 1316, Phenarsazine derivatives. Org. compounds of Au. 1318, Colour reaction of adrenaline. Determination of compounds precipitable by K<sub>2</sub>HgI<sub>3</sub>. 1319, Detection of morphine. 1329, Determination of traces of strychnine. 1333, Alkaloids in *Claviceps purpurea*. 1335, Prep. of human tubercle bacillus polysaccharide MB-200. 1336, Org. peroxide bactericides. Effect of ultra-violet rays on adrenaline. 1338, Determination of vitamin-B. 1340, Production of alkaloids in plants. Tobacco plant.

#### PATENTS.

**Preparing a therapeutic compound.** SHARIT CHEM. CO., INC., ASSEES. OF G. S. SHANOVSKOY (B.P. 358,610, 17.7.30. U.S., 17.7.29).—Aq. solutions of adrenaline and FeCl<sub>3</sub> are heated, preferably at or below 70°, until a reddish-brown coloration is formed. The FeCl<sub>3</sub> content of the final solution may vary from 0.5 to 3.0% and the adrenaline content from 0.01 to 0.10%.

E. H. SHARPLES.

**Manufacture of [therapeutically valuable] bis-muth salts.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 353,539, 30.4.30).—Neutral Bi salts sol. in fatty acids are obtained by the action of Bi<sub>2</sub>O<sub>3</sub> or Bi(OH)<sub>3</sub> on acids CHIR'·CO<sub>2</sub>H, in which R and R' are aliphatic or carbocyclic radicals (e.g.,  $\alpha$ -phenylbutyric,  $\alpha$ -phenylhexoic,  $\alpha$ -ethylbutyric, and  $\alpha$ -cyclohexenylbutyric acids), or by double decomp. of suitable salts. C. HOLLINS.

**Manufacture of complex salts of tervalent antimony yielding neutral solutions.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 352,928, 14.1.30).—During or after the prep. of complex salts from aliphatic polyhydroxy-acids of C<sub>5</sub> or C<sub>6</sub>, or from alicyclic polyhydroxy-acids, and Sb<sub>2</sub>O<sub>3</sub> or Sb(OH)<sub>3</sub> sufficient alkali or org. base is added to give a neutral solution. The Sb(OH)<sub>3</sub> may be produced *in situ* from SbCl<sub>3</sub> etc. Thus dil. NaOH is added to a mixture of SbCl<sub>3</sub> in MeOH and 50% aq. gluconic acid until neutral, and the whole, diluted with H<sub>2</sub>O, is heated for 30 min. at 100°, filtered, and the complex salt precipitated by MeOH.

C. HOLLINS.

**Preparation and purification of preparations of an active substance which retards the development of sex.** S. LOEWE (B.P. 353,635, 16.6.30. Ger., 19.6.29).—Animal or human fluids (blood, lymph, serum, urine, etc.) other than substances from the

hypophysis and corpus luteum are centrifuged, conc., fractionally precipitated by EtOH etc., and dialysed or otherwise fractionated, the fractionation being followed by physiological tests so as to separate the desired active constituents.

C. HOLLINS.

**Therapeutic substances.**—See XIX. Antiseptic etc.—See XXIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Precautions in practical sensitometry.** G. S. MOORE (Phot. J., 1931, 71, 403—412).—A crit. discussion of reciprocity failure is given. Standardisation of processing is detailed, and the deviations caused by variation of the colour temp. of the light source, the temp. of development, and rate of drying of the print are recorded.

J. LEWKOWITSCH.

See A., Nov., 1250, Highly disperse emulsions. Experiments with polarised light. Photodichroism and photoanisotropy. Action of peroxides on latent images. 1251, Light-sensitivity and development. 1252, Photographic action of  $\alpha$ - and of cathode rays. 1263, Neutral ultra-violet lighting. 1264, Applications of the Schlieren method of photography.

#### PATENTS.

**Manufacture of photographic sensitisers.** IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and E. H. RODD (B.P. 354,898, 31.5.30).—Unsymmetrical polymethine dyes, obtained by condensing diphenylformamidine (etc.) successively with 1 mol. each of 2 different mols. of heterocyclic N compounds containing external reactive CH<sub>2</sub> groups, are applied to photographic emulsions. Examples are compounds derived from: quinaldine ethiodide and 2-methylene-1 : 3 : 3-trimethylindoline; 2-methylbenzthiazole ethiodide and 2 : 3 : 3-trimethylindolenine methiodide; 2 : 3 : 3-trimethylindolenine, or 2-methylbenzthiazole ethiodide methiodide, or 2-methylbenzthiazole ethiodide and 2-methylbenzoxazole ethiodide.

C. HOLLINS.

**Increasing the resistability of photographic emulsion layers.** J. J. F. STOCK (U.S.P. 1,801,375, 21.4.31. Appl., 5.4.30).—The film, after the photograph has been finished, is swollen by treatment with conc. AcOH solution containing NH<sub>3</sub>; a cellulose ester is incorporated in the swollen film. The ester may be added to the swelling solution. The dry film has increased resistance to wear, the process being suitable for kinema film.

J. LEWKOWITSCH.

**Fixing of images obtained by a negative diazo-type process.** N.V. CHEM. FABR. L. VAN DER GRINTEN (B.P. 358,814, 20.12.30. Holl., 6.1.30).—The images are fixed by treatment with agents which prevent further dye-formation by acidification of the medium, or by reduction of the diazo compounds. The treatment may be exposure to vapours of a volatile acid (e.g., HCl) or application of a thin layer of solution (e.g., SnCl<sub>2</sub>, FeSO<sub>4</sub>, etc., according to the diazo compounds used).

J. LEWKOWITSCH.

**Iodomethanesulphonic acid etc.**—See III. Cellulose derivatives [for films].—See V. Hardening gelatin films.—See XV.

## XXII.—EXPLOSIVES; MATCHES.

**Influence of the moisture content of a [nitro-cellulose] powder on the calorimetrically measured heat of explosion. Effect of oxygen in the explosion bomb.** HAID, GOETZE, SELLE, KOINEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 129—131; Chem. Zentr., 1931, i, 2712).—A 0.4% excess of  $H_2O$  afforded vals. const. within the limits of error. A  $H_2O$  content of 1% in cellulose nitrate powder reduces the heat of explosion, calc. for 1 g. of moist powder, by about 0.8%. Decomp. of the powder in air instead of  $N_2$  gave a val. for the heat of explosion about 150 g.-cal. above normal. When technical  $N_2$  (3—4%  $O_2$ ) is used, vals. 25—30 g.-cal. too high are obtained. Experiments in Wolff's bomb gave more exact results than those in the Berthelot-Mahler bomb.

A. A. ELDRIDGE.

See A., Nov., 1268, Ethylidene peroxide.

## PATENTS.

**Explosive and its manufacture.** ITAL. SVILUPPO INVENZ. SOC. ANON., and V. GALLARATI (B.P. 358,116, 5.7.30).—Material containing proteins, e.g., glue, is treated with a mixture of  $H_2SO_4$  and  $HNO_3$ , the oily product then being separated and nitrated with a mixture of these acids containing more  $HNO_3$  than the original one. A solution of nitrocellulose in  $COMe_2$  together with castor oil or  $C_{10}H_7NO_2$  may be incorporated with this product to give a gelatinous or granular explosive.

W. J. WRIGHT.

**Manufacture of explosives and blasting charges.** DYNAMIT A.-G. (B.P. 358,157, 15.7.30. Addn. to B.P. 350,293; B., 1931, 783).—The ethanalamine derivatives of the prior patent may be wholly or partly replaced by nitrates of other saturated aliphatic amines containing one or more OH groups, or by *N*-alkyl derivatives of these amines.

W. J. WRIGHT.

Nitrated cellulose.—See V.

## XXIII.—SANITATION; WATER PURIFICATION.

**Sewage plant operation by  $p_H$  control.** H. HEUKLEKIAN (Sewage Works' J., 1931, 3, 428—437).—Laboratory experiments show that by the addition of  $Ca(OH)_2$  to unseeded fresh sewage solids the rate of digestion may be accelerated by 25% and the gas yield increased by 16%. Properly seeded digesting mixtures need no  $Ca(OH)_2$  provided the raw solids are fresh and strictly domestic, but the addition of  $Ca(OH)_2$  increases the possible daily load of fresh solids from 2.0 to 3.5%.

S

C. JEPSON.

**Sludge ripeness studies.** E. L. PEARSON and A. M. BUSWELL (Ind. Eng. Chem. [Anal.], 1931, 3, 359—360).—The drainability of a digested sludge is inversely related to its ripeness and to the ease with which non-draining moisture is removed. The ash and solids contents are not universally applicable as an indication of ripeness, though they may be useful in particular cases. In a "continuous-feed" tank the volatile acids indicate the course of digestion better than does its degree of completion. The details of the methods used in making the various tests are given. C. JEPSON.

**Treatment of [sugar-cane] mill effluents.** E. P. BEDLEY (Proc. S. Afr. Sugar Tech. Assoc., 1930, 77—

84).—In a plant described, up to 3000 gal./hr. of mill- and floor-washings and wash waters from filter-cloths are effectively clarified by liming to 0.06—0.1%, treating with "alumina ferric," straining from coarse matters, and flowing through four 3000-gal. hopper-shaped tanks. The clear yellow liquid then contains about 2% of solids including 0.5—1.5% of sugar, and is mixed with 100 pts. of condenser water before being discharged into a river, but some pollution occurs due to fermentable matter.

J. H. LANE.

See also A., Nov., 1259, Determination of Cs and Rb in mineral waters, and of Ca with picrolonic acid. 1263, Resistivity of mineral waters.

## PATENTS.

**Garbage incinerator.** E. M. OWENS (U.S.P. 1,803,206, 28.4.31. Appl., 20.1.28).—A form of destructor is claimed in which wet garbage is dried on a sloping  $H_2O$ -cooled grating placed in the upper part of the furnace and from whence it passes for complete combustion on to the lower main grate, which also deals with the dry garbage delivered to it by a  $H_2O$ -cooled chute.

C. JEPSON.

**Manufacture of neutral or substantially neutral compositions for antiseptic, germicidal, disinfectant, and deodorant purposes.** F. H. FAULDING & Co., LTD., Assees. of L. R. SCAMMELL (B.P. 358,865, 24.4.31. Austral., 25.7.30).—A Hg or Ag salt ( $HgI_2$ ) and a sol. iodide (KI) are added to an emulsion prepared from eucalyptus and/or Ti-tree oil and an EtOH solution of a fat (butter fat) and/or fatty acid (oleic acid) saponified by heating with KOH and/or NaOH. The product is toxic, e.g., to *Staphylococcus aureus*, and can be used for injection into the blood.

L. A. COLES.

**Filter [for main water].** A. C. MENGE (U.S.P. 1,800,093, 7.4.31. Appl., 13.11.29).—Through a filter-chamber filled, e.g., with alternate layers of sponges and charcoal, the water rises slowly and overflows into an outer (closed) casing having an air vent at the top which is closed by a float valve when the apparatus becomes full of water. The filtered water is withdrawn from the lower part of the casing, and the rate of flow may be greater than the rate of filtration. B. M. VENABLES.

**Cleaning of trickling filters used in purifying industrial wastes and other sewages.** M. LEVINE, Assr. to IOWA STATE COLL. OF AGRIC. & MECHANICAL ARTS (U.S.P. 1,802,726, 28.4.31. Appl., 28.10.29).—The filtering medium of flooded beds is agitated by compressed air delivered through pipes laid on the bed floor, and the detached humus is carried away by a "back-washing" stream of water and passes from the filter through a trough located above the surface of the filter.

C. JEPSON.

**Purification of water.** J. KRÜGER (B.P. 358,604, 15.7.30. Denm., 14.9.29).—After softening with the usual chemical reagents, the water is passed through a bed of irregular stone bodies coated with insol. carbonates which exert a "catalysing" effect and accelerate the precipitation of the  $CaCO_3$  and  $MgCO_3$ , which can be rapidly removed by mechanical filtration. C. JEPSON.

**Clarification of alkaline liquors.**—See VII. Fatty acid derivatives.—See XII. Disinfection of textiles etc.—See XIX.